

PROJECT LIGNITE: PREMIUM FUELS FROM
NORTHERN GREAT PLAINS LIGNITE

R & D Report No. 106 - Interim Report No. 2

PROCESS DEVELOPMENT FOR SOLVENT REFINED LIGNITE
LABORATORY AUTOCLAVE STUDIES

Donald E. Severson, Principal Investigator
Engineering Experiment Station
University of North Dakota
Grand Forks, North Dakota 58202

February 12, 1976

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

PREPARED FOR
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. E(49-18)-1224
UND Account No. 4567

EP

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

TABLE OF CONTENTS

I. Summary	
II. Introduction	4
A. General	4
B. Bench Scale Program	6
III. Batch Autoclave Tests on Lignite Liquefaction	8
A. General	8
B. Catalytic Effect of Native and Added Cations	10
C. Evaluation of Starting Solvents	12
D. Recycle of Solvent	17
E. Effect of Lignite Storage	19
F. Effect of Type of Reducing Gas	20
G. Effect of Prior Carbonization	21
H. Effect of Lignite Dehydration	23
I. Miscellaneous Studies	24
IV. Appendices	30
A. Laboratory Autoclave Tests	31
B. Analytical Methods	45
C. Distillation Methods	50
D. Computerized Data Reductions	51
E. Complete Autoclave Run Data	55

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States ERDA, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."

Printed in the United States of America

Available from

National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

Price: Printed Copy \$6.50; Microfiche \$3.00

I. SUMMARY

Batch autoclave tests are described which were made in a continuing investigation of solution-hydrogenation of lignite. Information was sought that will be of help in the design and operation of a continuous process development unit to produce an upgraded solvent refined lignite (SRL). Under a variety of test conditions conversion of over 90 percent of the moisture-ash-free lignite was achieved and a high heating value, low-ash, and low-sulfur product was made in addition to some light oils and gases. (Equipment and test procedures employed are presented.)

Lignite with its natural moisture can be solution-hydrogenated at high yields under pressure at elevated temperature in atmospheres of hydrogen, carbon monoxide, or carbon monoxide plus hydrogen (synthesis gas). Since the gas atmosphere for commercial plants to produce SRL would probably have to be produced from lignite, the process would be simpler if the synthesis gas could be used directly rather than be converted to pure hydrogen or carbon monoxide. Tests in which naturally occurring cations present in lignite were removed, and individual cations added, indicated that the naturally occurring concentration of cations seemed to provide the necessary catalytic effect for the solution-hydrogenation reactions.

Tests with several coal and petroleum-derived solvents showed that a petroleum-derived carbon-black feedstock should be suitable as an initial solvent. Simulation of continuous operation using recovered solvent in successive tests indicated that replacement of the carbon-black feedstock by lignite-derived solvent improved processing.

Prior carbonization or drying of the lignite was shown to be detrimental to the liquefaction processes. No major differences were noted in solution-hydrogenation of lignites from North Dakota, the Denver Basin, or Thailand. A half hour at reaction temperature was sufficient for completion of the reaction. Lignite sized to $-1\frac{1}{2}$ inch could be as readily processed as -100 mesh particles. Pretreatment of lignite with phenol was beneficial when the treated lignite was liquefied without drying. Storage as long as 36 weeks under various conditions did not significantly effect the solution hydrogenation.

II. INTRODUCTION

A. GENERAL

Production of upgraded fuels from coal provides one route to decreasing the present dependence of the United States on foreign sources for a significant portion of the nation's energy needs. Requirements for providing environmentally safe, clean, and convenient fuels have placed additional restrictions on traditional sources, resulting in shortages in some areas. Projections for future energy needs indicate a worsening situation in regard to supplies, and thus it will be necessary to convert and upgrade available fuels to clean and convenient fuels while meeting environmental restrictions. It is generally conceded that coal will be a major source in the near to middle term in helping achieve self sufficiency in the energy sector despite problems in mining and utilization.

Coal can be upgraded into clean burning solid, liquid, or gaseous fuels by several processes, many of which require demonstration of both economic and technical feasibility. Basically, coal conversion is a process of thermal or chemical depolymerization of coal "molecules" followed by addition of hydrogen for stabilization. Depending on extent and manner of hydrogen addition, the product can be solid, liquid or gaseous. Two general methods of accomplishing this conversion are direct hydrogenation to liquids, and gasification to synthesis gas followed by Fischer-Tropsch synthesis to hydrocarbons. In either case, the desired product is a low-ash, low-sulfur fuel.

The processing method and the coal used determine product quality and costs. Solid, liquid, and gaseous fuels all have their place in our present patterns of use, and the proper combination of "new" and "old" processes is required to optimize the product mix.

Of primary interest in the present project is a two-stage hydrogenation of lignite; first the production of an upgraded solid fuel and then conversion of this material to a light crude oil type product by catalytic hydrogenation. Usually some modification of the Pott-Broche process is employed in the first stage in which the coal is hydrogenated in the presence of an organic solvent. Slurrying with the solvent carrier facilitates handling and feeding of coal into the high pressure reactors, and the solvent may also act as a hydrogen donor to the coal. Low-rank, high-moisture coals may be hydrogenated using carbon monoxide rather than hydrogen. Synthesis gas (carbon monoxide and hydrogen) is also effective. If processing conditions are severe, liquids can be obtained as well as by-product fuel gases. Under milder conditions, the major product is liquid at process conditions but is solid at room temperature.

Several investigations are underway supported by the Energy Research and Development Administration and private industry on solution-hydrogenation. Most of these have placed primary emphasis on bituminous coals. Project Lignite is concerned essentially with low-rank coals, and particularly lignite.

The economics of fuel upgrading could be improved significantly by optimization of processing technology in an integrated refinery complex producing several upgraded fuel products. A major objective of the project is to investigate the feasibility of the refinery concept as applied to lignite. Work specific to lignite is justified because the properties of lignite are quite different from those of high rank coal. Lignite is more chemically reactive, does not agglomerate and its organic content is less aromatic, less condensed, has longer side chains and more cross linkages, and is considerably higher in non-moisture oxygen. Sulfur concentrations in lignites are generally lower than in most bituminous coals but the higher moisture and oxygen contents result in a lower calorific value. A 1974 Department of the Interior report (1) estimates that about 24 billion tons of lignite are recoverable by surface mining in the Great Plains area at relatively low cost. In terms of costs, reserves and properties, lignite is capable of making a significant contribution to the nation's energy requirements.

Bench scale laboratory research at the University of North Dakota under previous sponsorship and continued under Project Lignite was concerned with batch autoclave experiments to determine optimum operating parameters, suitable solvent and hydrogenating atmospheres for production of solvent refined lignite (SRL), and other data necessary for the design and construction of a continuous Process Development Unit (PDU). For reporting purposes, the status of the project is covered in two interim reports. The first deals with the design and construction of the PDU and the second is concerned with laboratory investigations.

This report is the second of the two and deals with the bench scale, batch autoclave test program which was conducted in the Project Lignite Laboratory. Over 400 experiments were conducted using the batch autoclave over the several years before the initiation of Project Lignite. In the first 2½ years of the Project 159 additional runs were conducted (Runs 407-565, inclusive) and are discussed herein. The test program developed initial hydrogenation process parameters such as reaction time, temperature, pressure, reactant ratios and type

(1) Demonstrated Coal Reserve Base of the United States on January 1, 1974. Mineral Industry Surveys, U.S. Department of the Interior, Bureau of Mines.

of solvent. Information from these batch autoclave runs has been used to design Project Lignite's Process Development Unit (PDU). The autoclave test program has been a vital link in the evaluation of Project Lignite. It has provided much of the preliminary data necessary for design, start-up and initial operation. However, only continuous operation of the process using the PDU will provide definitive data to permit optimizing operating conditions and for subsequent scale up of the process.

B. BENCH SCALE PROGRAM

Solvent hydrogenation of low-rank coals in bench scale, batch autoclave tests are summarized in the following sections. The reactor was a one-gallon stainless-steel stirred autoclave in which coal slurried with an organic solvent was processed in a hydrogen, hydrogen plus carbon monoxide, or carbon monoxide atmosphere at pressures to about 5000 psi and at temperatures as high as 900°F.

Briefly, organic solvent and pulverized lignite were charged to the autoclave in a 2:1 ratio of solvent to moisture-ash-free (MAF) lignite. Approximately 320 grams of lignite (200 grams MAF) was used for each batch. Water was added in some instances to replace moisture lost from the lignite during pulverization to maintain a constant water content within the autoclave. The autoclave was pressurized with gases to give the atmosphere desired, brought to reaction temperature, and kept at temperature for the desired reaction time. After completion of a test, the autoclave was cooled to about 400°F, and the gases discharged through a series of cold traps and collected in a balloon receiver. The collected gases were mixed, analyzed and metered. After discharge of the gases, the autoclave was repressurized with nitrogen, and the slurry, still at 400°F, was discharged through a dip tube onto a heated Buchner funnel where the solvent and coal-derived liquids were separated from the unreacted lignite by filtration. Detailed equipment arrangement, test procedures, analytical procedures, product work-up, and calculation methods are given in Appendices A, B, C, and D.

The apparatus, its operation, and some results have been presented previously (2) (3) (4) (5) (6). The present report gives results obtained in investigation of the effects of variables in the batch autoclave tests on product parameters including added cations, prior carbonization, different solvents, solvent recycle, prior drying, conditions of coal storage and others.

(2) Skidmore, Duane R., David S. Gleason and D.E. Severson. Low-Ash Carbon from Lignite. Technology and Use of Lignite, Proceedings: Bureau of Mines - University of North Dakota Symposium, Grand Forks, North Dakota, April 27-28, 1967. Bu. Mines Inf. Circ. 8376, May 1968, pp 137-143.

(3) Severson, D.E., D.R. Skidmore, and D.S. Gleason. Solution Hydrogenation of Lignite in Coal-Derived Solvents. AIME Trans., v 247, June 1970, pp 133-136.

(4) Wright, C.H. and Severson, D.E., Experimental Evidence for Catalyst Activity in Coal Minerals. 163rd National Meeting of the American Chemical Society, Boston, Mass Fuels Division Preprint, V.16, No. 2, 1972, pp 68-92.

(5) Severson, D.E., A. Max Soubey, and Wayne R. Kube. Laboratory Studies on Solution-hydrogenation of Lignite. Paper in Technology and Use of Lignite, Proceedings: Bureau of Mines - University of North Dakota Symposium, Grand Forks, ND, May 9-10, 1973. Bu Mines In. Circ. 8650, 1974, pp 236-246.

(6) Severson, D.E., A. Max Soubey and Wayne R. Kube. Project Lignite: Convenience Fuels from Northern Great Plains Province Lignite. Presented at the North Dakota Academy of Sci. Annual Meeting Fargo, N.D., May 1974, to be published in proceedings.

III. BATCH AUTOCLAVE TESTS ON LIGNITE LIQUEFACTION

A. GENERAL

Coal is a heterogeneous mixture of a complex organic material, associated extraneous minerals (ash) and moisture. The organic matter consists chiefly of carbon, hydrogen, and oxygen with some heterocyclic structures containing nitrogen and sulfur. Some cations are attached to the organic material, particularly in low rank coals. Models of the coal "molecule" have been proposed, but none have satisfactorily explained the properties of various coals. Apparently, the coal substance is not a definite compound but is a dispersed micelle consisting of solid humic substances of generally large molecular weights, differing somewhat depending upon extent of coalification (rank). Generally, the young coals (lignite and sub-bituminous) are not as aromatic as the older coals (bituminous and anthracite) and have more cross linkage, bridges and peripheral groups.

Much work has been done on the effect of solvents on coal since the mid-1800's. However, most of this has been directed towards separation and identification of organic compounds, determination of structure of a coal "molecule", or investigation of and elucidation of the coking properties of coking coals. The high molecular weight organic matter can be broken down into lighter fragments by a solubilization-hydrogenation process. Depending upon the severity of conditions and resulting extent of conversion, the product may be solid, liquid or gaseous. The addition of hydrogen to the unsaturated aromatic structure by various hydrogenolysis schemes helps to fragment the larger molecules and prevent polymerization of the fragments. A high hydrogen partial pressure and elevated temperature increases decomposition of the coal substance. In some cases, hydrogenation catalysts such as cobalt, nickel, iron or tin may promote the hydrogenation reactions.

Many modifications of hydrogenolysis have been proposed. The need for clean burning, low-polluting solid fuels and for petroleum-type products has generated interest in commercialization of a coal liquefaction process, and several projects, both private and governmental funded, are underway. Most of these are in the development stage with some projects being relatively large pilot plants. However, none of these have progressed sufficiently to establish commercial viability of the processes. The majority of the work is being done with bituminous coals.

Lignitic coals respond differently to hydrogenolysis than do bituminous coals. Extensive solubilization can be obtained in a hydrogen plus carbon monoxide or a carbon monoxide atmosphere provided the moisture of the lignite has not been removed by drying. Synthesis gas (H_2+CO) from gasification has been proposed for commercial application to avoid chemical conversion and purification to obtain pure hydrogen or carbon

monoxide. It appears that it is not necessary to have molecular hydrogen present initially since the reducing agent may be carbon monoxide. A simplistic explanation is that the water-gas shift reaction occurs releasing atomic hydrogen near reaction sites on the coal substance. The actual mechanism may involve free radicals with formate intermediates.(7)

In variable evaluation, yield data were obtained with an arbitrarily chosen "standard" lignite from a large bulk sample of known analysis. A standard set of operating conditions was established, and variable evaluation carried out by changing these conditions one or two at a time. In a standard test, a minus 100-mesh lignite sample containing 200 grams of MAF lignite plus its inherent moisture and ash was charged to the autoclave. Water was added to give a total lignite moisture plus added water content of 100 grams in the autoclave, corresponding to about 31 percent moisture in the added lignite. The sample was slurried with 400 grams of process solvent (such as anthracene oil), and pressurized with carbon monoxide and hydrogen in a 1:1 ratio to an initial pressure of 1000 psig. The reaction batch was heated at a constant rate of 3°C/minute to 400°C (752°F), held there for 30 minutes reaction time, cooled and removed from the autoclave at 205°C (400°F), gas being collected separately and liquids and solids separated by filtration at 205°C. Variations from these base conditions are indicated in the discussion of individual experiments. Over 500 batch autoclave experiments have been completed. Under a variety of test conditions and with various ranks of coal, conversions of MAF coal to liquids, light oils and gases of 90 percent or better were obtained. Recovery of solvent in the batch experiments has varied, but with some solvents was 100 percent or more, indicating that with a continuous process and adequate distillation and solvent recovery procedure the process would be self sustaining with regard to solvent.

Details of equipment and of test procedures are given in Appendix A. Analytical procedures are discussed in Appendix B, and included in Appendix C are the procedures used in distillation of solvents and of the liquefaction products. Information on computerized data reduction is included in Appendix D. Data on the autoclave tests are summarized in Appendix E.

Run yields are reported in terms of net conversion of MAF lignite to products, assigning changes in water, gas and liquids during the run to the MAF lignite alone. Thus the reported yield structure gives SRL produced, net liquids produced

(7) Appell, Herbert K., Irving Wender, and Ronald D. Miller, "Liquefaction of Lignite with Carbon Monoxide and Water," Technology and Use of Lignite. Proceedings: Bureau of Mines - University of North Dakota Symposium, Bismarck, North Dakota, May 12-13, 1971, BU Mines Inf. Circ. 8543, 1972, pp 32-39.

(lighter than SRL and including change in solvent inventory), net gas produced and net water. Changes in mineral matter accounted for, reported as ash, which are relatively minor, are lumped with the change in water as a "net water plus ash" item. The water production figure is usually negative, since water from the lignite moisture is consumed during the reaction and shows up as a negative amount produced from the MAF lignite. In the tables, light oil (liquids below SRL boiling range) and SRL yields are lumped under "net liquids", but are also shown separately. The results are normalized to a basis of 100 percent recovery of total input.

In the following sections, test results are reported categorized according to the objective of each series of experiments.

B. CATALYTIC EFFECT OF NATIVE AND ADDED CATIONS

The naturally occurring cations in the lignitic material and its accompanying mineral matter are believed to have some catalytic influence on the solution-hydrogenation process. To investigate the effect of cations on conversion and yields, a series of tests were made in which various cations - Na, K, Fe, Ca, Co, Ni, Mo - and a Ni-Mo catalyst (HDS-3A) - were added to the lignite prior to liquefaction. Prior extraction of naturally occurring cations in the lignite allowed the effect of individual cations to be investigated.

The ability of lignite to act as an ion-exchanger was used to control the concentration of the added cations. A sample of lignite, sufficiently large to provide material for the entire test series, was extracted with 1 N HCl which reduced ash content on a dry basis from about 10 to about 3 weight percent. Over 95 percent of the ash remaining after extraction consisted of alumina, silica, and iron oxide. Reverse ion exchange was used to add the desired cations to the extracted lignite. In cases where the cation formed a water-soluble acetate, this salt was used to avoid addition of "foreign" elements to the lignite. By adjustments of contact time and concentration of cation in solution, concentration of individual cations associated in the lignite could be controlled. It was possible to achieve individual cation concentrations above those occurring naturally in lignite. In two of the tests, one with nickel addition and another adding cobalt, a hydrogen atmosphere was used instead of a syngas (CO+H₂) mixture. Data for the cation exchange tests are given in Tables 1 through 3, parts 1 and 2, Appendix E. Cation additions were from 107 to 447 millimoles per Kg of raw lignite.

Although there was a great deal of scatter in these run data, some general conclusions can be drawn as to catalyst effect. For this discussion a table of average yields and conversions for runs shown in Tables 1, 2, 3, and 13 is

included below:

Effect of Cations

Cations Present Yields, Wt% of MAF Lignite:	Raw Lignite Natural	Extracted Lignite			Transition Metals
		None	Alkali Metals	Alkaline Earths	
Net Gas	38	26	41	30	25
Net Liquid (Light Oil) (SRL)	68 (15) (53)	64 (13) (51)	67 (17) (49)	62 (19) (43)	68 (20) (48)
Net Water	-13	-1	-14	0	-1
Unconverted	7	11	6	8	8
Solvent Recovery, %	93	91	93	89	90

Most tests with added alkali metal or transition metal cations gave liquid yields between 67 and 71 percent. The highest yields of SRL were obtained with the raw lignite, along with relatively high gas yields and water consumption. The catalyst ions generally produced more conversion to lighter products, i.e., light oil and gas.

Sodium and potassium, which are water gas shift catalysts, gave high consumption of water during the reaction, and the untreated lignite had high water consumption due to its alkali metal content. Water consumption with calcium and with the hydrogenation catalysts cobalt, nickel, and iron was much lower.

The effect of a given cation was quite insensitive to the amount added in the ranges studied, which for the alkali metals were of the same order of magnitude as the amounts naturally present in the coal.

Solvent recovery was less than 100 percent of solvent charged for all of these experiments. The natural cations and the shift catalyst additions gave closest to self-sufficiency of solvent together with good liquid yields.

The average conversions of lignite to gases and liquids was about 89 percent for extracted lignite, and 92 to 94 percent with cations present. Addition of cations increased the production of light oil at the expense of heavy fuel components.

Apparently, the naturally occurring cations present in the as-mined lignite provide sufficient catalytic action to give high yields of liquid products in a relatively short reaction time. This is a most favorable result since the expense and operational difficulty encountered from catalyst addition are avoided. Ash and unreacted coal can be separated from the polymerization products which removes some of usual hydrogenation catalysts. Additionally, some sulfur and nitrogen are removed. If lower molecular weight liquids are desired, the

depolymerized product can be hydrogenated in a second step with a specific catalyst designed for the particular process with reduced possibilities for deactivation and the advantages of liquid processing.

C. EVALUATION OF STARTING SOLVENTS

Anthracene oil had generally been used as solvent for the batch autoclave tests. Availability of sufficient anthracene oil even for start-up of a demonstration scale unit is doubtful, and several proposed units may compete for the limited supply. Consequently, other solvents were studied in the batch-autoclave for use in start-up and initial operation of the PDU until process-derived solvent is available.

Coal-derived and petroleum-derived solvents were used in otherwise standard procedure for lignite-liquefaction runs. Solvent performance was evaluated relative to:

1. Product yields
2. Characteristics of the solvent-refined lignite (SRL)
3. Characteristics of recycle-solvent recovered
4. Ease of handling
5. Percentage of "standard" cut in as-received solvent.

The standard cut in solvent distillation is the fraction obtained in the temperature range of 100 to 230°C at a pressure of 1.6 mm Hg. Light oil is the fraction distilled from IBP to 100°C, and heavy ends is that distilling between 230 to 255°C. For some tests, the raw or "as received" solvent was used directly. In others, the standard solvent cut was used, and for some, the solvent was hydrogenated before use. The solvents evaluated and the corresponding run numbers are shown on the following page. The "heavy ends cut" includes both the standard cut and the higher boiling material in the as-received solvent.

Coal-Derived Solvents

Raw creosote oil, heavy ends cut; Run 463
Raw creosote oil, standard solvent cut; Runs 447 and 491
Hydrogenated raw creosote oil, heavy ends cut; Run 464
Hydrogenated raw creosote oil, standard solvent cut; Run 479
Chilled creosote oil, heavy ends cut; Run 462
Chilled creosote oil, standard solvent cut; Run 475
Hydrogenated chilled creosote oil, standard cut; Run 474
Raw anthracene oil, heavy ends cut; Run 511
Raw anthracene oil, standard solvent cut; Run 455
Hydrogenated raw anthracene oil, standard solvent cut; Run 468
Chilled anthracene oil, heavy ends cut; Run 510
Chilled anthracene oil, standard solvent cut; Runs 465, 483, and 484
Hydrogenated chilled anthracene oil, standard solvent cut; Runs 471 and 480
Light creosote oil, as received; Run 503
Middle-heavy creosote oil, as received; Runs 507 and 508

Petroleum-Derived Solvents

Heavy aromatic naptha, as received, Run 519
#6 fuel oil, standard solvent cut; Run 473 and 478
#5 fuel oil, as received; Run 513
#5 fuel oil, standard solvent cut; Run 486
Aromatic tar, as received; Runs 496 and 509
Aromatic tar, standard solvent cut; Runs 495 and 499
Carbon black feedstock, as received; Run 514
Carbon black feedstock, standard solvent cut; Runs 485 and 497
Aromatic concentrate, as received; Runs 500 and 506
Aromatic concentrate, standard solvent cut; Run 494 and 501

Test conditions and results for the solvent evaluation series are summarized in Tables 4 through 7, parts 1 and 2, Appendix E. In Table 19, Appendix E, distillation data are given for the initial solvents.

The ranges for unconverted lignite, yields of gas, and the water consumption in weight percentage on an MAF basis of lignite charged for the coal derived solvents were:

<u>Item</u>	<u>Weight Percent</u>
Unconverted lignite:	5.7 to 14.6
Gas yield:	35.0 to 46.5
Water consumption:	5.0 to 17.3

Yields of light oil when the standard distillation cut was used were higher than when the heavy ends or as-received solvents were used. Hydrogenating creosote oil gave enhanced light oil yields; however, hydrogenation of anthracene oil had no apparent effect on light oil yields.

Filtration characteristics of the product slurries, rated from best to worst, for various solvents, follows: creosote oil, light creosote oil, anthracene oils, middle heavy creosote oils. Ease of handling of as-received solvents, from best to worst, are: anthracene oil, light creosote oil, creosote oils, middle-heavy creosote oil.

The approximate weight percentage of standard cut solvent recoverable from the as-received solvents was:

Creosote oils	70
Anthracene oils	85
Light creosote oils	30

The product SRL had H:C atomic ratios greater than 0.9 only when hydrogenated creosote oil was the solvent. Use of middle-heavy creosote oil resulted in low H:C ratios < 0.7, and a high sulfur content (~ 0.4 weight percent) in the SRL.

Product that was recovered in the solvent boiling range exhibited infra-red-ratios (IRR)* lower than that of the charged solvent indicated hydrogenation of the solvent during liquefaction. Sulfur content of the recovered solvent was also lower except when hydrogenated creosote oil was used. Standard cut solvent recovery for possible recycle was nearly sufficient to meet recycle requirements for the anthracene, but was low for the creosote solvents.

Petroleum-derived solvents exhibit similarities in yield and conversion as follows:

* See Appendix B.2.a.

<u>Item</u>	<u>Weight Percent</u>
Unconverted (maf basis)	8.3 to 12.8
Gas yield (maf basis)	~ 40
Water consumption	7.4 to 14.0

Exceptions were #5 fuel oil which resulted in lower overall conversion and the aromatic concentrate which gave higher gas yields.

Low light oil yields which may be associated with more stable solvents were obtained with carbon black feedstock, aromatic concentrate, and #5 fuel oil when distilled to the standard cut. Some as-received solvents also gave low light oil yields. These were heavy aromatic naphtha, carbon black feedstock, and aromatic concentrate. Yield of standard cut solvent obtained from the as-received solvents ranged from 42.2 to 55.4 weight percent except for the carbon black feedstock where the yield was 65.9 weight percent. Ease of solvent handling from best to worst was:

1. Carbon black feedstock and heavy aromatic naphtha
2. Aromatic concentrate and the fuel oils
3. Aromatic tar

Separation of the unconverted material from SRL by filtration was superior with carbon black feedstock, aromatic tar, and aromatic concentrate as solvents to that obtained using heavy aromatic naphtha and the fuel oils as solvents.

SRL with the highest H:C atomic ratio of about 0.9 was produced using the fuel oils as solvent. However, the initial H:C ratio of solvent was also the highest with the fuel oils, about 1.5. The petroleum-derived solvents, except heavy aromatic naphtha and aromatic tar, resulted in relatively higher sulfur content of the SRL (~ 0.9 weight percent) when used as-received. When the solvent was distilled to the standard cut, low sulfur content SRL (~ 0.35 - 0.40 weight percent) was produced using aromatic tar and the carbon black feedstock.

The IRR's of initial and recovered solvents were not appreciably different, although the IRR of the recovered solvent was slightly lower than that of the initial solvent in the case of aromatic tar. During the liquefaction process, sulfur content of the solvent fraction was reduced. All solvents used as-received showed a net increase in standard solvent boiling range after one pass through the liquefaction step. Recovery of solvent was sufficiently high for the standard cut solvents to indicate that the process might be self-sustaining, except when using aromatic tar and aromatic concentrate.

Of the various solvents investigated, carbon black feedstock, anthracene oil and creosote oil appear the most promising

for liquefaction of lignite. Because of availability and low cost, carbon black feedstock is the present candidate for use as the start-up solvent in the Process Development Unit.

Other solvents tested exhibited several undesirable characteristics:

Light creosote oil gave a low fraction of suitable solvent cut in as-received solvent.

Middle-heavy creosote oil was difficult to handle because it was nearly solid at room temperature, and gave poor filtration characteristics. The SRL product had high sulfur content and reduced H:C ratio.

Heavy aromatic naphtha gave a high fraction of light oil in as-received solvent and poor filtration characteristics of product slurry.

Fuel oils gave poor filtration characteristics, and there was excessive yield of gas with #5 fuel oil.

Aromatic tar gave reduced solvent recovery and a relatively high gas yield for the standard cut fraction. The initial solvent was difficult to process.

Several general conclusions regarding the various solvents are:

1. On the as-received basis, the fraction of standard cut solvent present was higher for the coal-derived than for the petroleum-derived solvents.

2. Hydrogenation of coal-derived solvents prior to use significantly changed the properties of creosote oils but not of anthracene oils.

3. During processing, coal-derived solvents were more highly hydrogenated than were the petroleum-derived solvents, as indicated by IRR.

4. Some hydrodesulfurization of all solvents, except the aromatic tar, was evident during processing.

5. A net increase in standard cut solvent was noted when processing as-received or heavy ends solvents indicating that some heavy ends distillable from the SRL should be included as recycle solvent.

6. Gas yields, water consumption, and the fraction of unconverted material were nearly the same for most solvents although standard solvent cuts from coal-derived solvents gave higher light oil yields than did the petroleum-derived solvents.

7. SRL prepared using petroleum-derived solvent had a higher sulfur content than did product obtained using coal-derived solvent. The higher sulfur content of the petroleum-type solvents apparently retards desulfurization of the SRL.

D. RECYCLE OF SOLVENT

Petroleum-derived carbon black feedstock appeared to be a viable alternate to anthracene oil as a solvent for hydrogenation-liquefaction of lignite. To indicate the effect of changing from the petroleum based solvent to lignite derived solvent, a series of tests was made in which recovered solvent from a previous run was used in a subsequent test. Such a sequence of batch experiments simulates a continuous process, and the change in solvent characteristics with successive passes should approach those obtainable in a continuous unit.

Recycle solvent was recovered by distillation from the solvent-slurry mixture and used for the next pass. Because of losses and requirements for sampling as well as the inability quantitatively to recover solvent from the filter cake, several tests at a given pass level were required to supply sufficient solvent for the next pass. For example, eight first-pass runs were required to supply recovered solvent for five second-pass runs which provided solvent for three third-pass runs, etc. The recycle tests were taken through five passes. The following runs are associated with the various pass levels:

1st pass - Runs 527, 528, 533, 536, 537, 546-548, 557, and 560.

2nd pass - Runs 529, 534, 543, 551, 552, and 558.

3rd pass - Runs 535, 544, 553, and 559.

4th pass - Runs 545, and 554.

5th pass - Run 565.

Standard test conditions were used for the entire series. A summary of test conditions and results are contained in Tables 8 through 10, parts 1 and 2, Appendix E.

Yields were similar for all tests regardless of the number of passes. Filtration improved as the pass number increased although viscosities of the reaction slurry measured at room temperature tended to increase as follows:

<u>Pass No.</u>	Brookfield <u>Slurry Viscosity, cp</u>
1	2580-4170
2	3570-4080
3	3770-3890
4	3575-4500
5	5864

However, improved filtration is indicated by a tendency towards reduction in pyridine solubilities in the filter cake:

<u>Pass No.</u>	<u>Pyridine Solubility, wt-pct</u>
1	69.9-80.3
2	67.6-77.3
3	65.1-69.1
4	63.6-72.1
5	64.1

In addition, the sulfur content of the SRL was somewhat lower as the number of passes increased.

<u>Pass No.</u>	<u>Sulfur Content, wt-pct</u>
1	0.35-0.44
2	0.33-0.40
3	0.31-0.38
4	0.31
5	0.33

No indication of unfavorable changes were noted. Generally, liquefaction characteristics were not significantly changed. Reduction in sulfur content of SRL and of solvent are encouraging as is the improved filterability as lignite-derived solvent replaces the initial petroleum-derived solvent. To illustrate this, the following tabulation gives average basic yield data as a function of number of passes:

Pass	Yields, Wt% of MAF Lignite				Solvent Recovery, %
	Net Gas	Net Liquid	Unconverted	Net H ₂ O	
1	35.6	63.9	11.5	-11.1	100.9
2	35.1	64.1	10.8	-10.0	102.1
3	34.4	65.1	11.0	-10.4	101.3
4	34.7	64.6	10.4	-9.8	99.5
5	34.2	64.4	10.7	-9.3	101.9

No significant trend in results is evident considering the variability in replication and that the number of tests averaged ranged from 10 in pass 1 to one in pass 5. The recycling of solvent, with consequent replacement of some of the starting solvent with material derived from coal, had no effect on conversion or yields, and had a favorable effect on handling quality, as indicated by improved filterability. This, together with the production of more than 100 percent of the solvent charged, indicates that continuous operating with solvent recycle will be feasible.

E. EFFECT OF LIGNITE STORAGE

Storage is known to alter some properties of coals such as caking, heating value, and moisture content. Consequently, a series of tests was designed to evaluate the effect of various storage conditions on liquefaction of lignite. In most processing applications, particularly development work, some delay between mining and utilization is unavoidable.

A bulk sample of lignite was subdivided, and the representative fractions stored under the following conditions:

1. Lump lignite under distilled water at ambient conditions. After storage and just prior to liquefaction, the sample was pulverized in a single pass through the mill.
2. Lignite, pulverized by one pass through the mill, in air at 50 pct relative humidity, ambient conditions.
3. Lignite, pulverized by one pass through the mill, in nitrogen at 50 pct relative humidity, ambient conditions.

After predetermined times in storage, samples from each of the three storage conditions were liquefied using the standard procedures. A duplicate test was made on one of three samples in each set. The following sequence of tests were completed:

<u>Run No.</u>	<u>Storage Conditions</u>	<u>Time in Storage, Week</u>
504, 505	Base conditions	0
515	Water immersion	6
516	Air	6
517, 518	Nitrogen	6
521, 524	Air	12
522	Nitrogen	12
523	Water immersion	12
539	Nitrogen	24
540, 542	Water immersion	24
541	Air	24
561	Nitrogen	36
564	Water immersion	36
562, 563	Air	36

Complete data for these tests are presented in Tables 11 and 12, parts 1 and 2, Appendix E.

Little difference in yield was noted for the various storage conditions. Ranges for yields of gases were:

<u>Storage Conditions</u>	<u>Gas Yield, wt. pct, MAF</u>
Water immersion	35.1 - 41.7
Air	30.6 - 41.6
Nitrogen	29.8 - 39.6

Liquid yield given as ranges were:

<u>Storage Conditions</u>	<u>Liquid Yield, wt. pct, MAF</u>
Water immersion	64.6 - 72.4
Air	61.7 - 70.3
Nitrogen	61.5 - 72.2

For up to 36 weeks storage under the conditions specified, no significant changes were apparent in the liquefaction results. Variability in test results would obscure small changes in the yields of products. Operationally, filtration characteristics of the resulting liquid-solid slurry for the lignite that had been stored were less favorable than for the baseline conditions. Storage will continue for an extended period to indicate if there are long term adverse effects.

F. EFFECT OF TYPE OF REDUCING GAS

Three different atmospheres were used in otherwise standard batch-liquefaction tests, carbon monoxide, hydrogen, and synthesis gas (syngas). Syngas was 50-50 mol percent mixture of CO+H₂. Initial pressure was 1000 psig in each case. Both as-received and acid-extracted lignites were used. The complete results of the runs are presented in Table 13, parts 1 and 2, Appendix E.

Liquefaction of As-Received Lignite

Run	410	422	412
Atmosphere	CO	Syngas	H ₂
<i>Yields, wt% of MAF Coal</i>			
Net Gas	66.8	35.2	13.6
Net Light Oil	9.3	13.1	14.5
Net SRL	33.3	55.3	43.2
Unconverted	17.2	10.0	17.8

Liquefaction of Acid-Extracted Lignite

Run	411	414	413
Atmosphere	CO	Syngas	H ₂
<i>Yields, wt% of MAF Coal</i>			
Net Gas	24.9	27.1	13.6
Net Light Oil	11.3	12.4	14.4
Net SRL	48.7	47.2	43.4
Unconverted	22.6	12.1	15.8

Conversion of lignite was generally higher with the syngas atmosphere, 88 to 90 percent, as compared with 77 to 84 percent for the other tests.

Yields of liquid products were appreciably higher using the syngas atmosphere and the as-received lignite with its natural moisture content, 68 percent as compared with 43 to 60 percent in all the other tests. With acid-extracted lignite the liquid yields were not appreciably different regardless of the atmosphere.

For both extracted and unextracted lignite, yields of gaseous components were higher with more carbon monoxide in the charge gas because of production of carbon dioxide by the water gas shift reaction. Highest gas yields were obtained with carbon monoxide and as-received lignite, because of the catalytic effect of the mineral matter which was largely removed by acid extraction.

Thus, for the batch liquefaction procedures, a syngas atmosphere with as-received lignite gave the best liquefaction as determined by high conversion and high yield of liquid products.

G. EFFECT OF PRIOR CARBONIZATION

Carbonization or mild heat-treating of lignite prior to liquefaction has been suggested as a means for upgrading lignite, reducing carbon dioxide generated during liquefaction, concentrating methane production, and improving liquefaction characteristics.

A test series was designed to explore the effects of carbonization under the following conditions:

<u>Run</u>	<u>Carbonization conditions</u>
430	Carbonized at 260°C for 1/2 hr with anthracene oil in batch autoclave; cooled and gas removed; CO and H ₂ added and standard liquefaction conducted.
440	Same as 430, except carbonized at 315°C.
451	Same as 430, except carbonized at 370°C.
450, 452	Carbonized at 370°C for 1/2 hr in batch autoclave; cooled and gas removed; solvent, CO, and H ₂ added and standard liquefaction conducted.
457, 493	Carbonized at 370°C for 1/2 hr in external carbonizer; cooled, products collected; and char liquefied under standard conditions.
421, 422, 455	Baseline liquefaction tests.

Carbonization and liquefaction data for this test series are given in Tables 14 and 15, parts 1 and 2, Appendix E, and the baseline tests are given in Tables 3 and 13.

Some carbon dioxide was removed during carbonization which would reduce the load on the gas purification system in a liquefaction process. The amounts of carbon dioxide evolved during carbonization and subsequent liquefaction are shown:

Run	<u>Carbon dioxide, moles CO₂/100 lb. dry lignite</u>		
	<u>Carbonization</u>	<u>Liquefaction</u>	<u>Total</u>
451	0.261	0.955	1.216
452	0.216	0.882	1.098
457	0.170	1.008	1.178
455	None	1.459	1.459

Prior carbonization of lignite apparently reduced the total CO₂ generation compared to the baseline test. Approximately 20 percent of the total CO₂ which was evolved during the carbonization-liquefaction sequence was released during carbonization.

Comparative methane yields for those runs, where the lignite was carbonized prior to liquefaction, are shown below:

Run	<u>Methane yield, moles/100 lb. dry lignite</u>		
	<u>Carbonization</u>	<u>Liquefaction</u>	<u>Total</u>
451	0.025	0.064	0.089
452	0.078	0.049	0.127
457	0.022	0.092	0.114
455	None	0.096	0.096

Appreciable methane production occurred during carbonization. Perhaps slightly more total methane is formed during combined carbonization-liquefaction but would not be significant or important enough to warrant a carbonization step. The concentration was higher in the carbonization gas than in the gases from subsequent liquefaction because there was a greater total gas production in liquefaction. For Run 457, methane in the product gases was 9.3 percent in the carbonization step and 3.6 percent in the liquefaction step.

Overall, the results from the mild carbonization or heat-treatment of the raw lignite in the presence of the liquefaction solvent show slight deteriorous effects on the overall yields and conversion, as indicated in the table following:

Runs	Base	430	440	451
Pretreatment, °C	None	260	315	370
Yield, Wt% MAF Lignite				
Net Gas	38	44	40	33
Net Liquid	68	64	61	57
(Light Oil)	(15)	(25)	(10)	(12)
(SRL)	(53)	(39)	(51)	(45)
Net Water	-13	-15	-10	-8
Unconverted	7	7	9	18

The effect of carbonization without solvent prior to liquefaction, whether in the autoclave with only gaseous products removed before liquefaction or in a separate carbonizer with only the char charged to liquefaction, is to reduce the liquid yields appreciably:

Runs	Base	450, 452	C4, 457	C5, 493
Carbonization, °C	None	370	375	395
Charge to Liquefaction	Lignite	Char & Liq.	← Char only →	
Yields, Wt% MAF Lignite				
Net Gas	38	52	28	24
Net Liquid	68	35	37	39
(Light Oil)	(15)	(13)	(4)	(10)
(SRL)	(53)	(22)	(33)	(29)
Net Water	-13	-15	23	23
Unconverted	7	28	12	14

Although somewhat lower yields of carbon dioxide were achieved and small (< 20 percent) quantities of methane were produced by carbonization, the undesirable effects of prior carbonization on the liquefaction step make the combined process unattractive.

H. EFFECT OF LIGNITE DEHYDRATION

Several advantages have been suggested for drying lignite before liquefaction. Reducing the moisture content would result in lower transportation costs if the liquefaction plant was not at the mine mouth. A lower moisture content of the charged lignite may decrease the generation of carbon dioxide resulting in a reduced load on the gas purification system. Also a lower operating pressure would be possible at the same level of hydrogen-carbon monoxide partial pressure.

The effect of prior dehydration on liquefaction was measured in a test series in which the solvent and lignite were heated at 215°C in the autoclave to remove the moisture. The lignite-solvent mixture was cooled, and standard liquefaction run made. The following tabulation shows other conditions for the various tests:

Run	Test Conditions
530	Chilled anthracene oil and CO + H ₂ atmos.
531	Carbon black feedstock (FS-120) and CO + H ₂ atmos.
532	Chilled anthracene oil and hydrogen.
484 (Table 4)	Baseline test with chilled anthracene oil.
497 (Table 6)	Baseline test with carbon black feedstock.

Test conditions and results for these tests are detailed in Table 16, parts 1 and 2, Appendix E.

Reducing the moisture content adversely influenced the extent of liquefaction especially in the synthesis gas atmosphere. Percentage of unconverted lignite for the dehydrated slurry ranged from 23.9 to 41.6 weight percent on an MAF basis compared with 8.0 to 9.8 weight percent for the baseline tests. Conversion was higher in a hydrogen atmosphere than in the synthesis gas atmosphere. Yields of liquids were low, ranging from 36 to 54.2 weight percent of the MAF lignite charged, compared to base test liquid yields from 61.0 to 63.4 weight percent. Filtration rates for the product slurry were reduced significantly starting with dehydrated lignite.

However, yields of carbon dioxide were drastically reduced as shown in the following tabulation:

<u>Run</u>	<u>Test Condition</u>	<u>Gas Yield, Wt %</u>	<u>Mol % CO₂ in Gas</u>
484	anthracene oil-baseline	43.9	32.0
530	anthracene oil-syn gas	12.5	12.3
532	anthracene oil-hydrogen	13.1	0.1
497	carbon black feedstock-baseline	38.2	35.1
531	carbon black feedstock-syn gas	20.5	13.5

Reduction in moisture content reduced availability of water for the water-gas shift reaction which produces CO₂. Loss of the hydrogen from the shift reaction may be partly responsible for the reduced liquefaction especially when using the CO + H₂ atmosphere.

When liquefaction is the primary concern, the gain resulting from reduced carbon dioxide production does not offset the loss in reactivity from dehydration which results in low yields.

I. MISCELLANEOUS STUDIES

Several miscellaneous process variations were investigated in limited experimental programs to determine their influence on the liquefaction process. Factors studied were:

1. Filtration temperature
2. Reaction time
3. Particle size of lignite
4. Moisture content of lignite
5. Lignites from various sources
6. Pretreatment of lignite with phenol
7. Product work-up by distillation only

Test conditions and experimental results for some of these runs are contained in Tables 17 and 18, parts 1 and 2, Appendix E. For other runs, complete results are not reported, but a limited amount of data is summarized in the discussion.

1. Filtration Temperature

The liquid-solid slurry remaining in the autoclave after discharge of product gases is highly viscous at ambient temperatures. At higher temperatures, the viscosity is reduced sufficiently to allow separation of the solids by filtration. A limited number of tests using anthracene oil solvent were made under standard liquefaction conditions except that the temperature at which the liquid-solid products were discharged from the autoclave was varied. Experimental conditions and results follow:

<u>Run</u>	<u>Filtration Temperature</u>	<u>Filtration Characteristics</u>
417	500°F	Excessive volatilization of solvent; filter paper charred. Filtration difficult.
419, 420	450°F	Volatileization of solvent, Filtration difficult.
421	425°F	Good filtration
422	400°F	Good filtration. No noticeable solvent volatilization.

These data indicate that 400°F is a reasonable temperature for the filtration step when anthracene oil is used as solvent. Consequently, 400°F was selected as the standard temperature for filtration. The upper temperature limit for filtration is established as the boiling point of the solvent at discharge conditions. Thus, the best filtration temperature may be determined by solvent characteristics and may be different for different solvents.

2. Reaction Time

In operation of the autoclave, the fastest that the batch operation can be cycled includes a minimum of 0.5 hr at reaction temperature because of operational considerations. To determine if 0.5 hr was sufficient time for the hydrogenation-solution reactions, a comparison was made with runs otherwise identical except for the time the reactor was at reaction temperature.

<u>Run</u>	422	418
<u>Time, hr</u>	0.5	2.0
<u>Yields, wt% of MAF Coal</u>		
Net gas	35.2	49.3
Net liquids	68.7	56.8

These data indicate that longer time at reaction temperature results in more decomposition of the lignite which increases gas yield and reduces liquid yield. Decomposition of water allows total yield of gases and liquids to exceed 100 percent on an MAF lignite basis for both reaction times. The hydrogenation-solution process is apparently rapid with high liquid yields

occurring at the shortest reaction time practical with the equipment used. Thus the 0.5 hr at reaction temperature was used as the standard test time at reaction temperature.

3. Particle Size of Lignite

Standard testing procedure required that the lignite be pulverized to -100 U.S. mesh before liquefaction. Previously, it had been postulated that such a small particle size would be necessary to insure rapid reaction. To check this assumption, Run 487 (Table 17) was made with a larger top size, 0.25 inches, under the otherwise standard liquefaction procedure. The results of this run are compared with those of three standard runs (Table 4) in the summary following:

<u>Run</u>	465, 483, 484	487
<u>Lignite size</u>	-100 mesh	-0.25 in.
<u>Yields, wt% of MAF Coal</u>		
Net Gas	43	45
Net Liquid	62	61
(Light Oil)	(14)	(17)
(SRL)	(48)	(44)
Net Water	-13	-11
Unconverted	8	5

These data showed that liquefaction is essentially the same under the employed test conditions either with an initial particle size of -100 mesh or -1/4 inch. In pilot plant operation, it should not be necessary to pulverize the lignite completely to -100 mesh to have satisfactory liquefaction.

4. Moisture Content of Lignite

As reported in a previous section, dehydration of lignite and solvent in the autoclave before liquefaction gave unfavorable results. To see if this was actually because of change in reactivity of the lignite or because of reduced availability of moisture, a test was designed in which the lignite was dried externally to the autoclave, the dried lignite slurried with the solvent and water added so as to have the same total water present as when full moisture lignite was liquefied under the standard procedures. The results of these runs are compared with those of three standard runs (Table 4) in the summary following:

<u>Run</u>	465, 483, 483	442	445
<u>Lignite</u>	Full moisture	dried	dried
<u>Yields, wt% of MAF Coal</u>			
Net Gas	43	39	40
Net Light Oil	14	8	7
Net SRL	48	59	60
Unconverted	8	8	10
Net Water	-13	-14	-17

Little difference exists between gas make and conversion as reported above. However, yield of light oil was much lower when using dried lignite. In addition, the liquid-solid product slurry was more viscous and difficult to filter indicating reduced conversion to the lower molecular weight liquids.

5. Lignites from Various Sources

To determine if different lignites responded differently to liquefaction, two lignites from outside the Fort Union Formation in North Dakota were treated by standard liquefaction procedures in the batch autoclave (Table 17). One lignite was from Thailand (Run 512) and the other from the Denver Basin in Colorado (Runs 469 and 476). These are compared to Run 465, in which the standard North Dakota lignite was used, in the following tabulation:

Run	465	469	476	512
Lignite	North Dakota	Denver Basin	Thailand	
<u>Yields, wt% of MAF Coal</u>				
Net gas	39.0	32.6	35.7	31.9
Net liquid	67.5	69.0	61.8	67.0
Unconverted	8.4	5.9	7.0	2.1

The reported yield parameters did not indicate major differences in the various lignites although water consumption and the fraction unconverted were low for the Thailand lignite. It was also noted that filtration was better for the Denver Basin and Thailand lignites than for the North Dakota lignite indicating somewhat lower molecular weights of liquid products. Additional work would be required for more definite comparisons.

6. Pretreatment of Lignite with Phenol

When treated with phenol, coals are known to swell indicating partial dissolution. To see if this partial dissolution is beneficial to the solution-hydrogenation liquefaction process some phenol treated lignite was tested (Table 17).

Crushed lignite (-0.25 in.) was immersed in phenol at room temperature and the phenol was removed from the lignite by one of two procedures. Lignite prepared for liquefaction Run 489 had the phenol removed by water washing, followed by acetone washing and vacuum drying. For Run 492, the phenol was removed by only water washing. These runs are compared with a baseline test, Run 487, in the following:

Run	487	492	489
Lignite	As received	Phenol treated Dehydrated	Phenol treated Wet
<u>Yields, wt% of MAF Coal</u>			
Net gas	45.6	44.4	37.1
Net liquid	61.0	63.8	73.9
Unconverted	4.7	4.2	2.9
Light Oil	16.7	18.0	25.3

Yield data for the baseline test and for the dehydrated phenol treated lignite were similar. However, previous data has indicated a loss in reactivity when lignite was dehydrated. Wet phenol-treated lignite gave lower gas yields, higher net liquid and light oil yields, and had less unconverted lignite. This indicates that phenol pretreatment can change the reactivity of lignite towards a more useful distribution of liquid-type products and increased conversion. Additional work would be required to indicate if the increased process costs for the phenol treatment could be justified.

7. Product Work-up by Distillation Only

In several cases the slurry-product from an otherwise standard liquefaction test was removed from the liquefaction reactor without filtration and was subjected directly to the vacuum distillation step to recover light oil and solvent. The amount of SRL was determined analytically by pyridine extraction of the vacuum bottoms. The complete results of these runs are presented in Table 18, parts 1 and 2, Appendix E. A summary comparison with conventional runs is shown below:

Product Work-up by Distillation Only

Run No.	481	482	488	Avg.
Yields, wt% of MAF Coal				
Net Gas	46.8	42.6	39.5	43.0
Net Light Oil	17.0	17.8	14.2	16.3
Net SRL	33.6	38.2	40.3	37.4
Unconverted	14.4	14.0	19.3	15.9
Solvent Recovery, wt%	94.9	95.6	97.7	96.1
Overall Recovery, wt%	100.1	97.9	98.5	98.8

Product Work-up by Filtration and Distillation

Run No.	465	483	484	Avg.
Yields, wt% of MAF Coal				
Net Gas	39.0	46.5	43.9	43.1
Net Light Oil	13.1	13.0	15.1	13.7
Net SRL	54.4	43.9	45.9	48.1
Unconverted	8.4	8.9	8.0	8.4
Solvent Recovery, wt%	96.4	92.4	95.5	94.8
Overall Recovery, wt%	94.4	95.4	97.4	95.7

The significant differences between the two methods of product work-up are in the yields of SRL and in the conversion, though the light oil yield, solvent recovery, and overall recovery are slightly higher for the distillation only work-up, indicating there may be some losses in the filtration step. However, the lower indicated SRL yield and lignite conversion indicates there to be some material in the unfiltered vacuum bottoms that is less soluble in the pyridine extraction analysis than when liquefaction solvent is also present. It may be that the material is degraded or polymerized by the high temperatures in the still pot during distillation, or it may be that

the liquefaction solvent has a solubilizing effect on the material during pyridine extractions. At any rate, it may be well to remember that higher yields of SRL may be realizable by a process in which the reactor slurry is deashed prior to solvent recovery.

IV. APPENDICES

A. LABORATORY AUTOCLAVE TESTS	31
1. Equipment	31
2. Calibration	31
a. Charge Gas Calibration	31
b. Pressure Gage Calibration	32
c. Thermocouple Calibration	32
3. Input Material Preparation	32
4. Test Operation	32
5. Product Workup	34
a. Product Gas	34
b. Reactor Liquid-Solid Products	34
c. Processed Products	36
B. ANALYTICAL METHODS	45
1. Standard (ASTM) Procedures	45
a. Moisture	45
b. Ash	45
c. Volatile Matter	45
d. Carbon-Hydrogen	45
e. Sulfur	45
f. Nitrogen	46
g. Specific Gravity (liquids)	46
h. Extractable Hydrocarbons	46
i. Calorific Value	46
2. Specific Test Procedures	46
a. IR Ratio	46
b. Blackness	47
c. Melting Point	47
d. Brookfield Viscosity	47
e. Hydrogen Sulfide	48
f. Specific Gravity (gases)	48
g. Gas Analysis (GC)	48
C. DISTILLATION METHODS	50
1. Atmospheric Distillation	50
2. Vacuum Distillation	50
D. COMPUTERIZED DATA REDUCTION	51
E. COMPLETE AUTOCLAVE RUN DATA	55

APPENDIX A

LABORATORY AUTOCLAVE TESTS

1. Equipment

All experiments were conducted in a one-gallon Magne-Drive type 316 stainless steel autoclave manufactured by Autoclave Engineers, Inc., Erie, Pennsylvania. The autoclave unit is equipped with a magnetic stirrer assembly, a thermocouple in a well for measuring the reaction mixture temperature, a reaction mixture withdrawal tube, gas ports, and a four-element nine-KW jacket heater. High pressure cylinders of nitrogen, carbon monoxide and hydrogen used in the experiments are manifolded to the autoclave with 3/8 inch o.d. high-pressure stainless steel tubing. Electric power to the heater is controlled by a Honeywell Electr-O-Volt Controller connected to two Electronik 15 strip chart recorders; one of the recorders is also connected to a pressure transducer on the autoclave to permit monitoring of the internal pressure. The temperature controller can be used for either manual or automatic control of heating rate; the recorders enable the operator to monitor both the external and internal temperature. See Figures A-1 and A-2 for schematic diagrams of the autoclave and the accessory equipment.

2. Calibration

a. Charge Gas Calibration

To obtain a complete material balance for the batch liquefaction test, the weight of synthesis gas components (CO and H₂) charged to the autoclave must be known. In order to calculate the weights of carbon monoxide and of hydrogen in any mixture of CO and H₂ charged under pressure to the autoclave containing the reaction mixture (lignite solvent slurry), calibration runs are made with pure CO, pure H₂, and a mixture of CO and H₂. The CO-H₂ gas mixture is of known composition.

Calibration curves (psig vs. moles) are determined for pure CO, pure H₂, and a 1:1 molar CO and H₂ mixture with the autoclave charged with a typical liquid-solid reaction mixture, i.e., a lignite-solvent slurry weighing approximately 715 grams of about 2:1 weight ratio of solvent to MAF lignite. For the CO calibration curve, the void space above the reaction mixture is first purged with CO to remove air, followed by pressurization to 1000 psig with pure CO. The autoclave stirrer is turned on to mix the gas with the slurry and to stabilize pressure. A wet test meter attached to the gas exit port on the autoclave head assembly measured the CO which is released slowly from the autoclave through the wet test meter at approximately 0.30 cf/min. Barometric pressure and wet test meter temperature readings are recorded; the cumulative volume of CO gas released from the reactor is measured at each 100 psig increment from 1000 psig to 0 psig. From these data, the number of moles of gas released at each gage reading could be calculated assuming an ideal gas at atmospheric pressure and ambient temperature. Identical procedures are followed for pure hydrogen and for approximately

a 1:1 molar mixture of CO and H₂ whose actual composition is determined accurately using gas chromatography.

The calibration data are plotted as pressure in psig per mole of total gas versus mole percent H₂ using three points, one for pure H₂, one for CO and one for an intermediate composition. A straight line relation is assumed between points. A sample calibration curve is shown in Figure A-3.

b. Pressure Gage Calibration

Pressure gages used in autoclave work (0 to 6000 psig) are calibrated periodically, using a Model 1305 Ashcroft portable dead-weight tester for pressures to 5000 psi. A plot of actual gage readings (in psig) vs dead-weight standards (in psi) is made so that the corrected gage reading (in psig) for any actual gage reading can be obtained. The corrected gage readings are then calibrated against the pressure transducer signal response (read as temperature units on the Electronik 15 strip chart recorder). From these data, reactor pressure versus time plots as well as temperature versus time charts are prepared for each batch autoclave test.

c. Thermocouple Calibration

Chromel-alumel thermocouples used in the autoclave work are calibrated against an NBS platinum-rhodium standard from ambient temperature to 800°F.

3. Input Material Preparation

Lignite is pulverized and sieved to -100 mesh and analyzed for moisture, volatile matter, fixed carbon, and ash content. The weight of as-pulverized lignite equivalent to 200 grams of moisture and ash free lignite, sufficient water to make a total of 100 grams of moisture, and 400 grams of solvent are thoroughly mixed to form a slurry at room temperature.

4. Test Operation

Immediately following preparation, the lignite-solvent slurry is charged to the autoclave by pouring from a preweighed stainless steel container into the autoclave and transferring adhering slurry with a preweighed rubber spatula. The empty slurry container and spatula are reweighed to determine the amount of material transferred into the autoclave. The relative amounts of solvent and coal transferred are assumed to be proportional to the amounts of lignite and solvent in the originally prepared slurry, so that the actual grams of lignite and solvent charged to the autoclave can be determined. It is also assumed that none of the added water is lost during the transfer of slurry to the autoclave.

Following the charging of slurry, the autoclave head is attached and the input gas line, stirrer assembly, water jacket

cooling line, stirrer drive belt, and both inside and outside thermocouples connected. The autoclave assembly is checked for leaks by pressure testing to approximately 1000 psig with nitrogen for several minutes. The nitrogen is then bled from the autoclave, and the void space above the reaction slurry purged three times by pressuring to 100 psig with CO and then releasing the pressure to remove air and nitrogen from the reactor chamber. The magnetic stirrer is then turned on and the autoclave pressurized to 500 psig with CO. Stirring proceeds for several minutes to enable the CO to mix completely in the reaction slurry; once mixing is completed, as indicated by a stable pressure reading, a small amount of additional CO is added to adjust the pressure to 550 psig. Hydrogen gas is then added to the autoclave to raise the pressure to 1100 psig. The pressure is again stabilized and the autoclave stirrer is shut off. The autoclave containing the reaction mixture is allowed to stand overnight at room temperature.

Prior to the start of an actual autoclave test, the stirrer is turned on, and about 50 ml of the gas in the autoclave is sampled. The autoclave pressure is decreased to 1000 psig and the autoclave jacket heater turned on. The temperature control system is adjusted to maintain a heating rate of approximately 5°F/min until the desired reaction temperature, usually 752°F, is attained. Once at 752°F, the reaction slurry in the autoclave is maintained at this temperature for 30 minutes. The jacket heater is shut off and lowered from the reactor, allowing the autoclave contents to cool to 400°F. Next, the reactor jacket heater is raised and turned on to control the temperature at 400°F during product recovery operations. See Figure A-4 for a typical pressure-temperature-time profile of a batch autoclave test.

Product recovery is accomplished in two steps; first, removal of product gas followed by removal of the reactor product slurry. The product gas recovery system consists of a series of six cold traps and a 200-liter rubber gas bag. The cold traps range in temperature from ambient to -70°C, and collect moisture and light and heavy oils from the product gas before the rubber gas bag. The first trap in the system is a 1000 ml Erlenmeyer flask maintained at room temperature; next are four 250 ml glass tube traps mounted in Dewar flasks, two maintained at 0°C, and the last two at -70°C. The sixth trap, a glass-wool packed drying tube maintained at room temperature, removes entrained oil and tar-like substances not previously trapped.

Following the traps, the dry, oil-free product gas is collected in the rubber gas bag. Prior to collection of gases, a vacuum pump is attached to a side arm valve on the gas bag, and used to evacuate the entire trap system. After several minutes under vacuum, the pump is turned off and disconnected from the trap system. (See Figure A-5). The product gas is then slowly admitted through the trap system at a rate of about 0.30 cf/min. After product gas is removed from the auto-

clave, the trap system and the gas bag are disconnected and sealed for later analysis.

The autoclave is then prepared for slurry removal and filtration by connecting a 3/8 inch stainless steel transfer tube to the slurry withdrawal tube port. A stainless steel disk attached to the end of the transfer tube spreads the reacted slurry symmetrically onto a Whatman No. 5 filter paper mounted in a 10-1/2 inch Buchner funnel which is maintained at 400°F. A 10 inch glass pie plate covering the Buchner funnel has two 1/4-inch holes through which the slurry transfer tube and a nitrogen gas line are inserted to maintain an inert atmosphere on the filter cake. A 2000 ml heavy walled filter flask attached to the discharge of Buchner funnel collects the filtrate; connected to the outlet arm of the filter flask is a cold trap maintained at 0°F followed by a glass wool-packed drying tube to trap light fractions of the filtrate not condensed in the cold trap during filtration.

To remove the slurry from the autoclave, the autoclave reactor is pressurized to 100 psig with CO and the vacuum pump turned on. When the slurry temperature stabilizes at 400°F, the autoclave stirrer is shut off, the time noted, and the ball valve on the slurry transfer tube carefully opened to transfer the hot slurry to the Buchner funnel at a slow and uniform rate. After all of the slurry is removed from the autoclave, filtration is allowed to proceed until the flow of filtrate stops.

5. Product Workup

a. Product Gas

Product gas collected from the autoclave is analyzed immediately for H₂S content and non-condensable gas components. The specific gravity and the volume of the product gas are also measured. Approximately 1500 ml of gas is required for analytical purposes and this volume is included in the total gas measurement. In addition, a correction for water vapor is made for the measured gas, since the dry gas in the gas bag is measured through a wet test meter.

b. Reactor Liquid-Solid Products

In order to obtain quantitative yield data for liquid and solids products, all glassware, funnels, traps, sample bottles, etc. are preweighed for each test; losses due to transfer of the products to permanent sample containers are included as actual yields for material balance calculations.

Water and light oil traps from the product gas removal step are weighed individually and the contents mixed in a separatory funnel. Oil and water are separated and weighed separately. Other than determining weight, no analytical work is performed on the water sample; the separated light oil

fraction is analyzed for carbon, hydrogen, and sulfur. Light and/or heavier oil retained in the glass-wool trap just upstream of the vacuum pump in the gas recovery system is weighed and the result is added to the weight of light oil collected in the trap system. The light oil obtained from the product gas trap system is reported as "light oil in condensate" in autoclave data reduction calculations.

Filtrate collected during the coal liquid recovery step (filtration) is weighed in the collection flask. Additionally, any vapor from the filtrate condensed and trapped immediately downstream from the filter flask is weighed and added to the original filtrate collected in the Erlenmeyer flask; filtrate residue collected in the glass-wool packed trap immediately upstream of the suction filtration system vacuum pump is weighed and the weight included in the total weight of filtrate. Since the filtrate is quite viscous at room temperature, it is heated to approximately 100°F in an Erlenmeyer flask using a heating mantle before being transferred to a sample bottle. A filtrate sample is analyzed for carbon, hydrogen, sulfur and ash contents, and specific gravity and apparent viscosity determined. Depending on the nature of the autoclave test, infrared analysis may be employed to characterize the filtrate.

For evaluation of the filtrate, approximately 250 grams are vacuum distilled at 1.6 mm Hg and fractions in the following boiling ranges collected and weighed: IBP - 100°C (light oil), 100°-230°C (recycle solvent), 230°-255°C (heavy oil), and 255°C plus (vacuum bottoms containing primarily solvent-refined lignite). The heavy oil is also considered a part of the solvent refined lignite. Weight yields obtained for each fraction are prorated to the total filtrate for material balance and yield calculations. The weight of light oil obtained from the distillation of filtrate is added to the weight of light oil condensed from the product gas for calculating the total yield of light oil for the autoclave test.

Filter cake produced by suction filtration of the reaction slurry is carefully removed from the Buchner funnel on its filter paper and weighed. The cake is scraped from the filter paper and placed in a sample bottle for subsequent chemical analysis. Samples of the filter cake are analyzed for carbon, hydrogen, sulfur and ash contents and for pyridine solubility.

Pot residue is the slurry that adheres to the walls and bottom of the autoclave reactor chamber after the slurry has been transferred to the suction filtration assembly. The pot residue is removed from the autoclave reactor wall using a rubber spatula, transferred to a sample jar and weighed.

Residue is the slurry adhering to the autoclave stirrer, the Buchner funnel interior and wall, the pie-plate cover for the funnel, the filter paper hold-down ring, the spreader, the reactor head assembly, the coal liquid transfer tube, the

nitrogen gas purge line, and the clean-up tools. See Table A-1 for autoclave raw product distributions. Most of the residue is removed using a rubber spatula and weighed; residue not recovered by spatula is collected on pre-weighed Kimwipes and the resultant weight increase of the Kimwipes is added to the weight yield of residue. Both pot residue and residue samples are combined and analyzed as residue; analyses of the composite residue sample includes carbon, hydrogen, sulfur, and ash contents, and pyridine solubility. See Figure A-7 for a schematic of analytical tests performed on both the input coal and solvent and on the products from an autoclave test.

The calculated yields include those of light oils, reclaimed solvent, SRL, and mineral residues. The distribution of these calculated products is obtained in the following manner: The light oils are included in the filtrate. The pot residue and residue are extracted with pyridine and the soluble material is included in the filtrate and the insoluble material included in the mineral residue. The pyridine-soluble portion of the cake is included in the filtrate and the remainder of the cake in the mineral residues. The mineral residues are further distributed between ash (inorganic mineral matter) and unconverted lignite. The vacuum distillation of a portion of the filtrate is the basis for distributing all the filtrate into light oil, reclaimed solvent, and SRL.

c. Processed Products

Reclaimed solvent is collected as the 100°-230°C @ 1.6 mm Hg liquid fraction from the vacuum distillation of the filtrate, and is analyzed for carbon, hydrogen, sulfur, and ash contents, and IR ratio (ratio of aromatic to aliphatic hydrogen). In addition, specific gravity, and viscosity measurements are made on the reclaimed solvent.

Vacuum bottoms solid is obtained from the vacuum distillation of the filtrate at 1.6 mm Hg; it consists of heavy oil (boiling range 230 to 255°C @ 1.6 mm Hg) and solvent refined lignite boiling at 255°C and higher at 1.6 mm Hg. The vacuum bottoms solid is chipped from the flat bottom distillation flask at room temperature, pulverized, and analyzed for carbon, hydrogen, sulfur, and ash contents, heating value, and melting point.

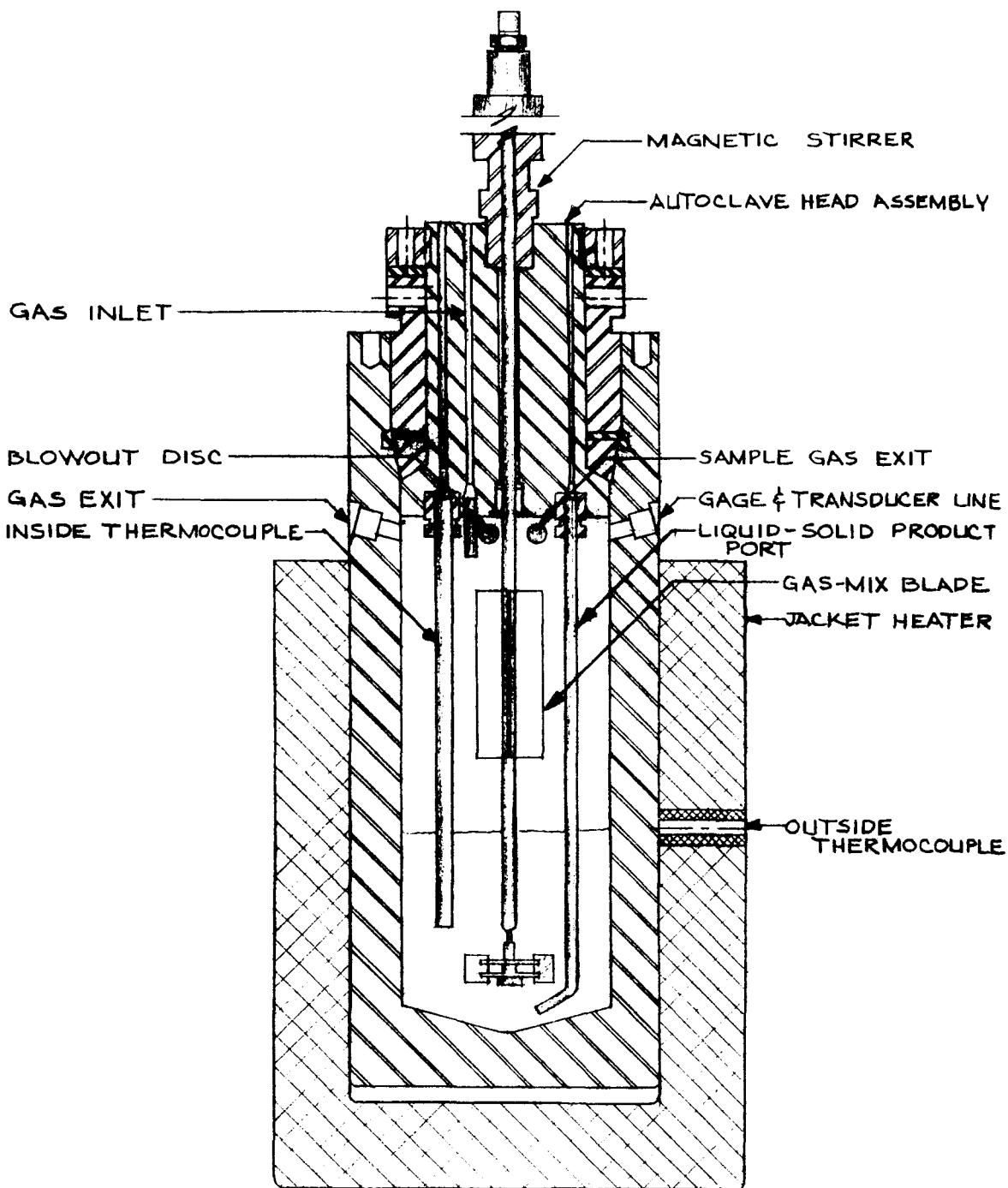
APPENDIX TABLE A-1
LABORATORY AUTOCLAVE COAL LIQUEFACTION TESTS: PRODUCT DISTRIBUTION

<u>Autoclave Products from:</u>	<u>Filter Cake</u>	<u>Filtrate</u>	<u>Pot Residue</u>	<u>Residue</u>	<u>Water</u>	<u>Light Oil</u>	<u>Gas</u>
1. Autoclave wall				X			
2. Autoclave stirrer					X		
3. Filter paper		X					
4. Buchner funnel wall and pie-plate cover						X	
5. Filter paper hold-down ring	X(1)				X(1)		
6. Spreader	X(1)				X(1)		
7. Erlenmeyer filtration flask		X					
8. Filtration trap (0°C)		X					
9. Filtration tar trap (RT)		X					
10. Pre-weighed Kimwipes (R)					X(2)		
11. Gas recovery Erlenmeyer flask trap (RT)						X	X
12. Gas recovery 0°C trap						X	X
13. Gas recovery -70°C							X
14. Gas recovery tar trap (RT)							X
15. 200-liter gas bag (RT)							X

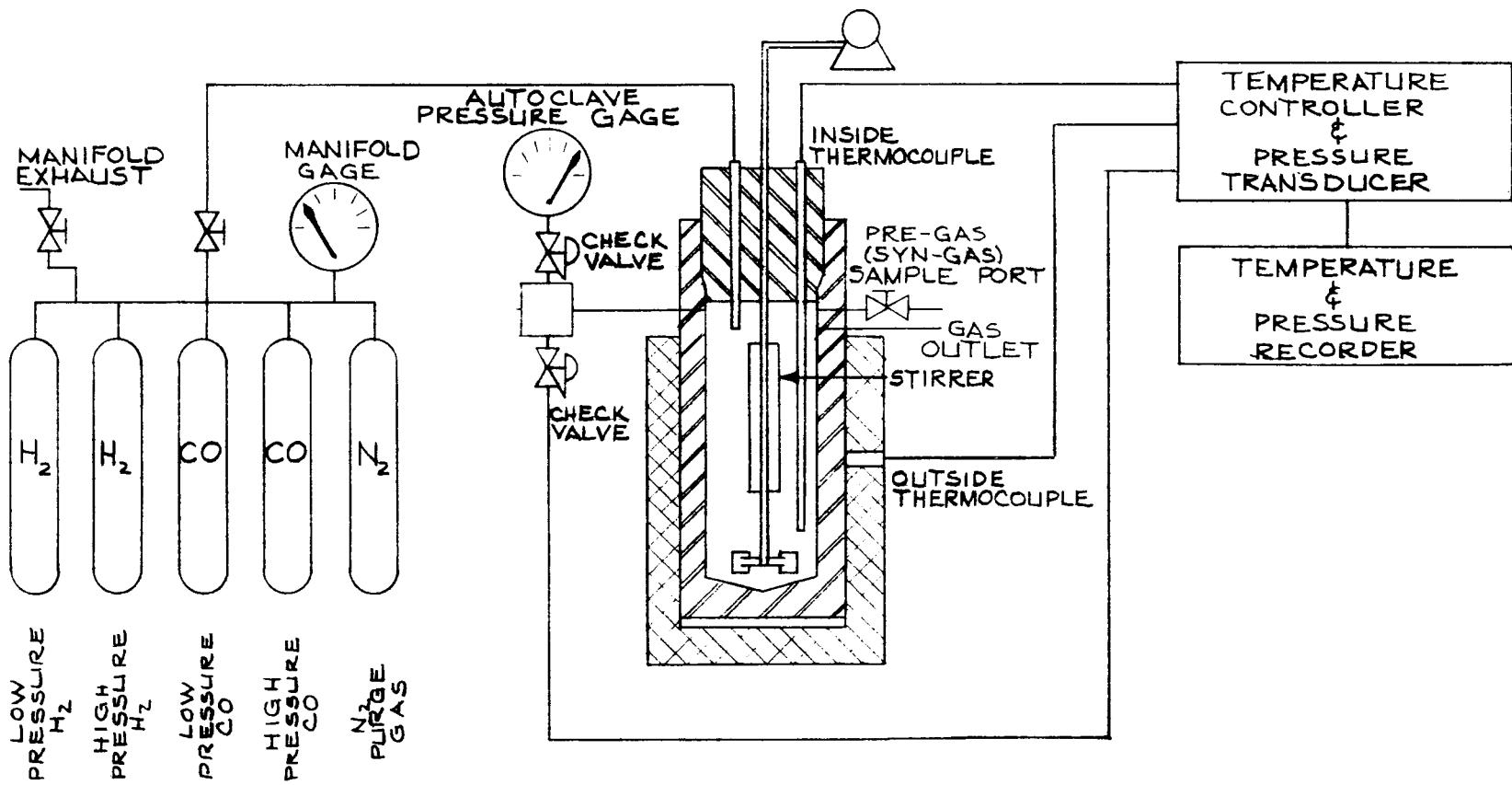
(1) Material adhering to the ring or spreader is considered to be filter cake or residue depending on its visual appearance; i.e., if it is oily substance not resembling the filter cake, it is considered residue; however, if it closely resembles the filter cake, it is considered filter cake.

(2) Pre-weighed Kimwipes are wetted with thinner and used to remove adhering residue from the reactor head assembly, discharge tube, nitrogen purge line, coal-liquid transfer line, and clean up tools; Kimwipes are then forced air-dried to evaporate thinner and weighed to obtain residue weight.

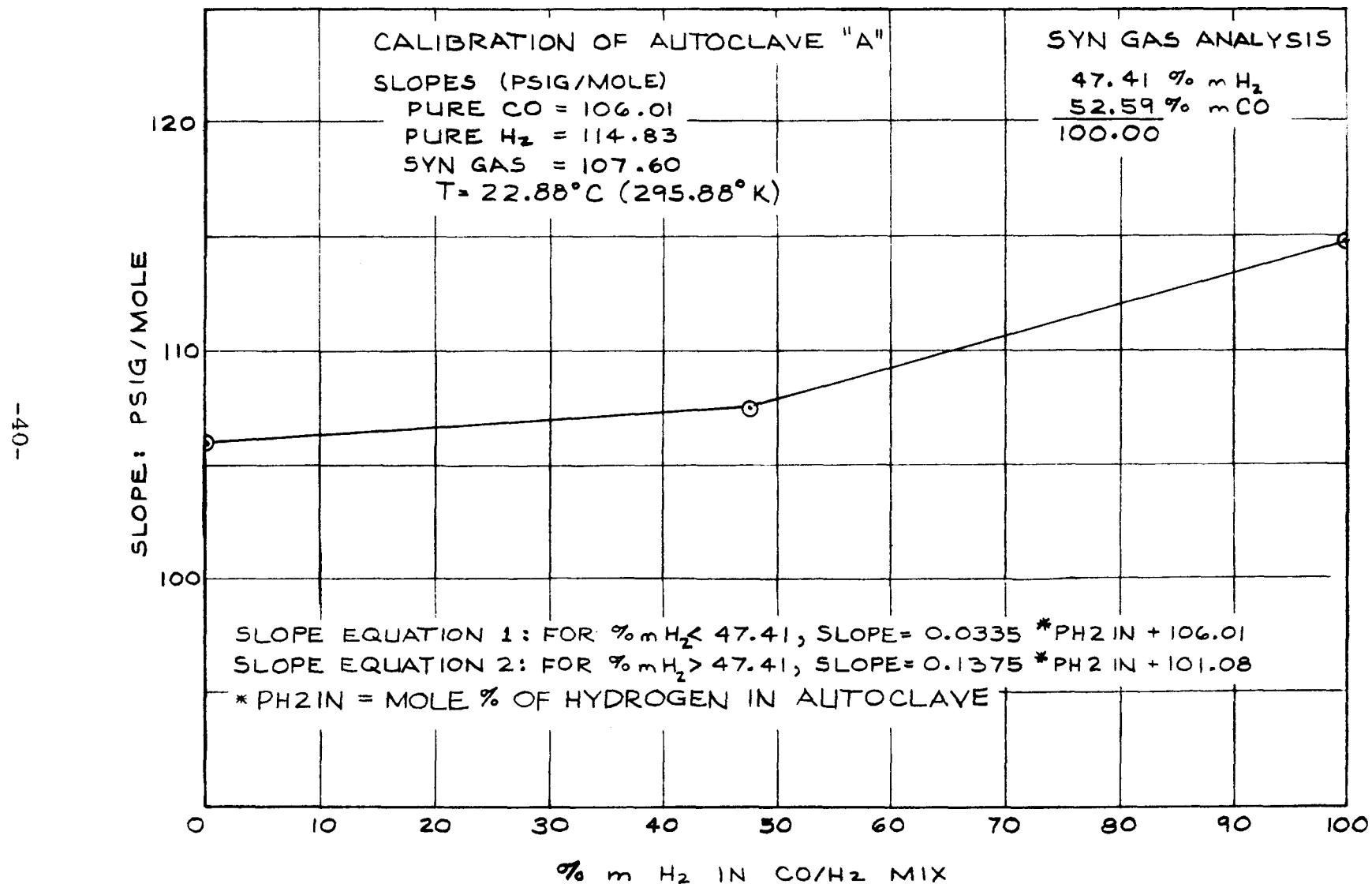
APPENDIX FIGURE A-1
BATCH AUTOCLAVE APPARATUS



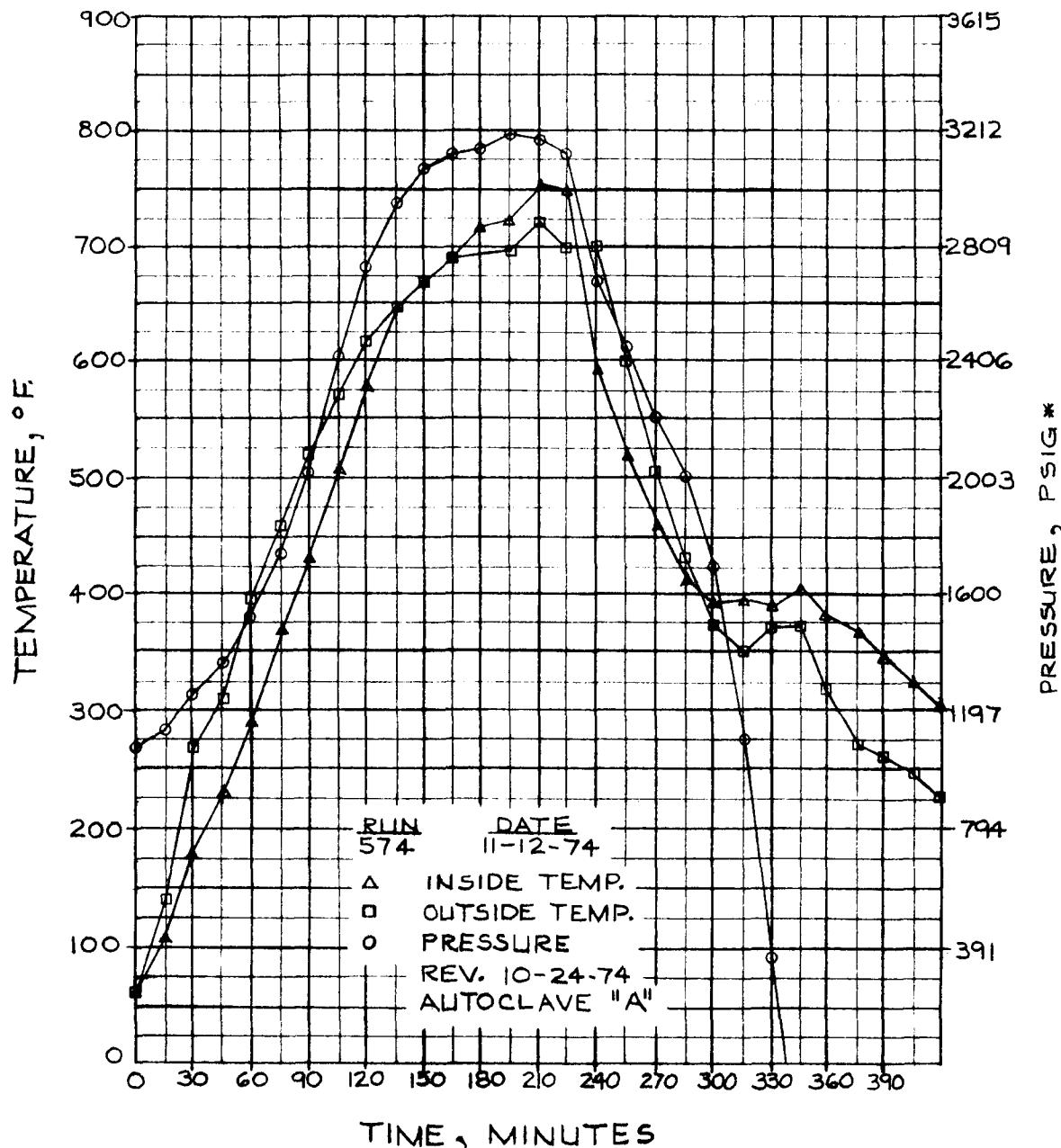
APPENDIX FIGURE A-2
AUTOCLAVE APPARATUS/ACCESSORY SCHEMATIC



APPENDIX FIGURE A-3

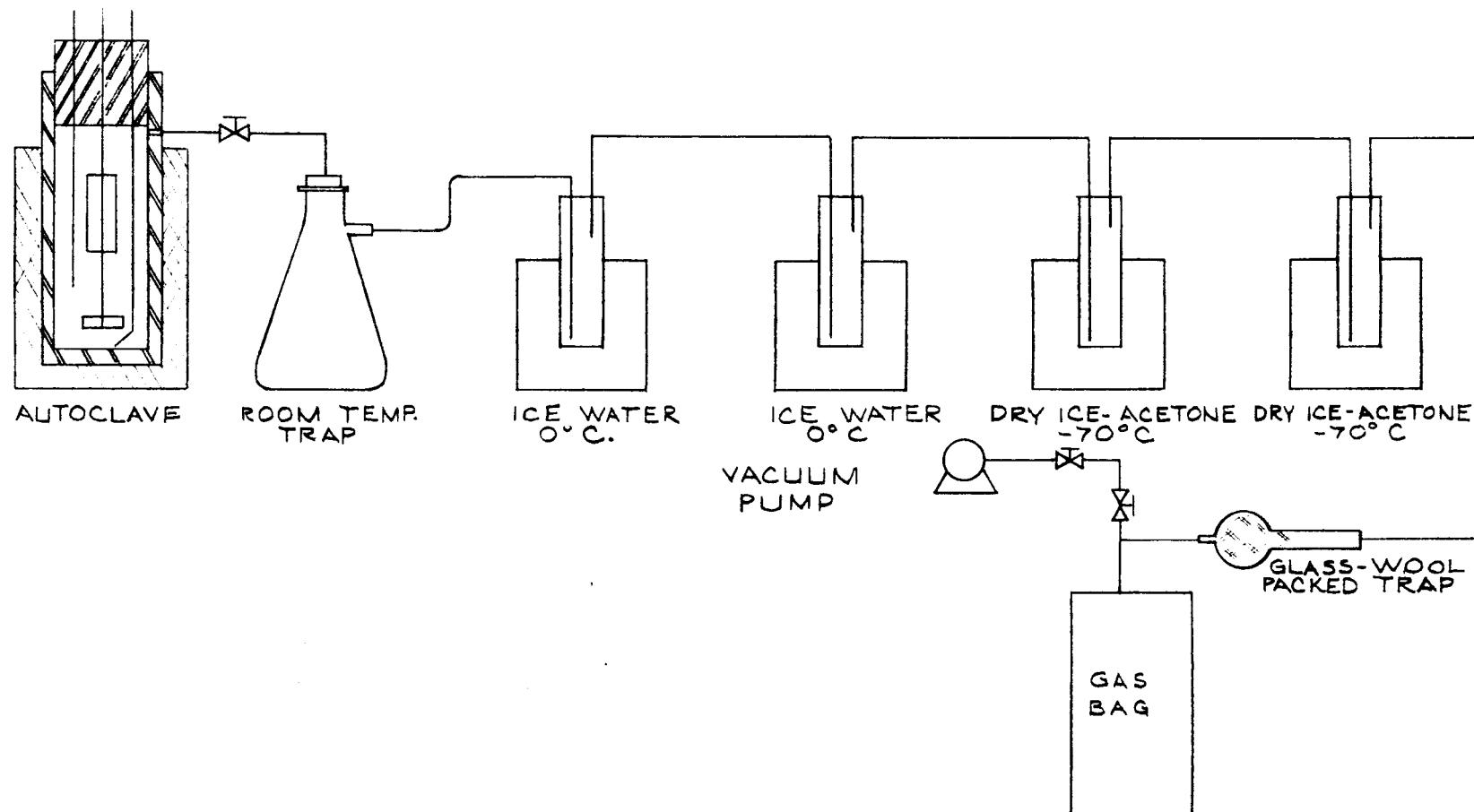


APPENDIX FIGURE A-4
 TYPICAL PRESSURE-TEMPERATURE
 PROFILE FOR BATCH AUTOCLAVE COAL
 LIQUEFACTION EXPERIMENTS

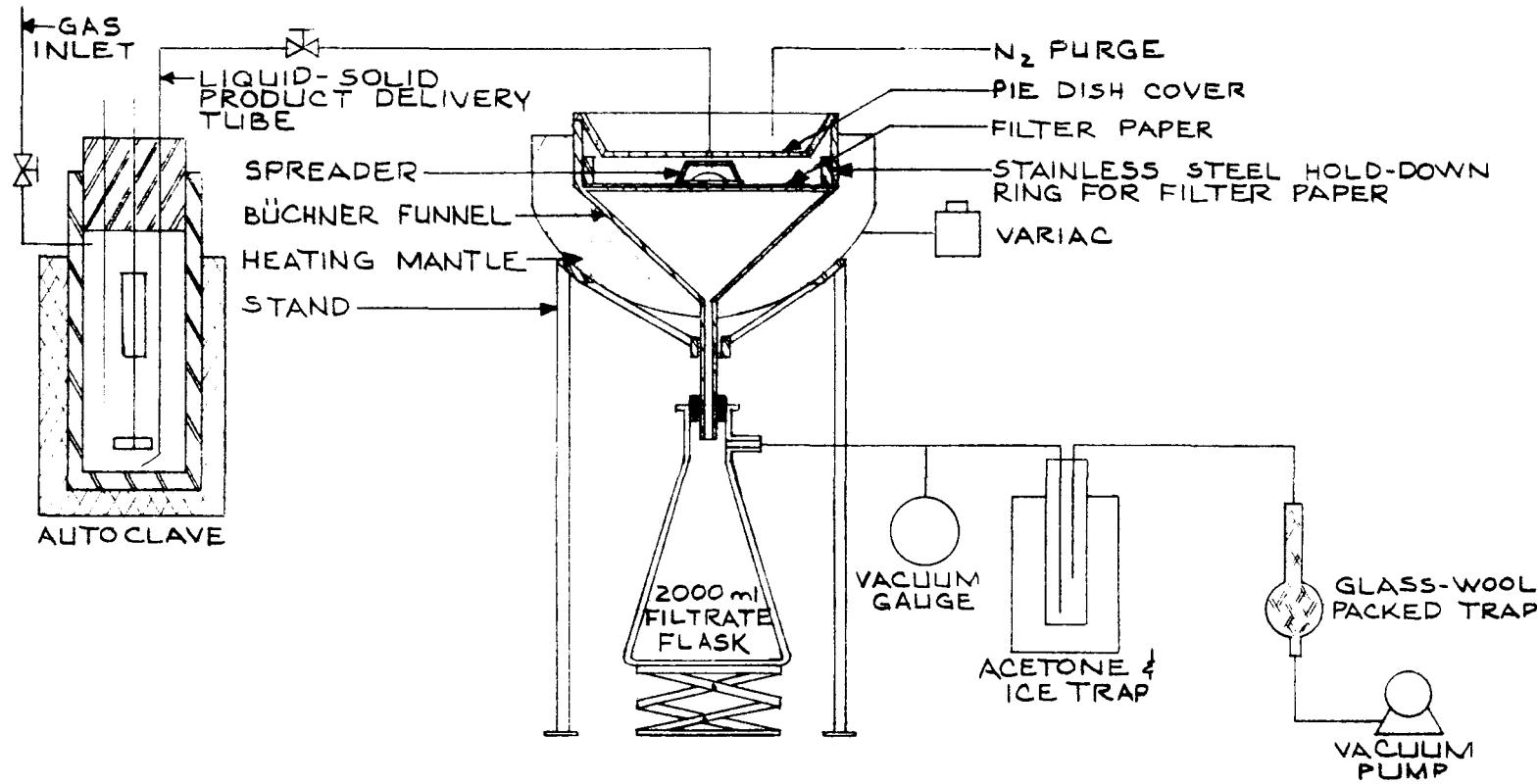


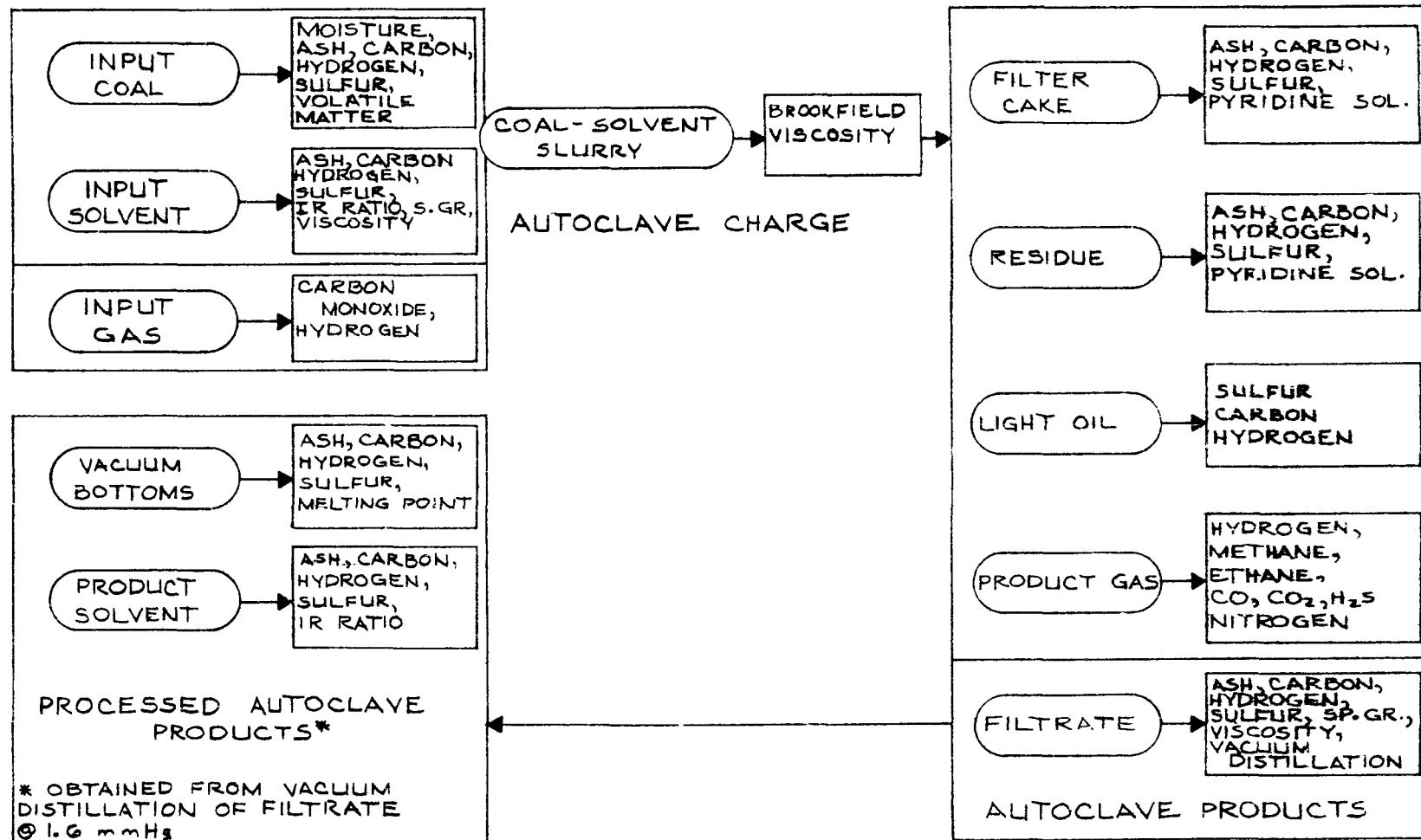
* NOTE: EACH PRESSURE DIV.
 EQUALS CA. 100.8 PSIG

APPENDIX FIGURE A-5
BATCH AUTOCLAVE TEST - PRODUCT GAS REMOVAL



APPENDIX FIGURE A-6
BATCH AUTOCLAVE TEST- LIQUID SOLID PRODUCT REMOVAL





APPENDIX FIGURE A-7
ANALYTICAL TEST SCHEMATIC
BATCH AUTOCLAVE LIQIEFACTION STUDIES

APPENDIX B

ANALYTICAL METHODS

1. Standard (ASTM) Procedures.

These procedures are used to characterize raw and solvent-refined lignite and intermediate liquid products obtained from the batch autoclave liquefaction experiments.

a. Moisture in the pulverized raw lignite to be tested in the autoclave is determined using ASTM D-3173; this method measures the weight loss of a sample after a one-hour exposure in an air-atmosphere drying oven at 105°C. ASTM D-95 is used occasionally as an alternate method for determining moisture content in lignite; the sample is distilled with xylene, which co-distills with water in the sample. The separated water settles in a graduated section of a special distillation trap. The water content of the sample is calculated from the measured volume of water collected during distillation.

b. Ash contents of raw lignite, solvent-refined lignite, input and reclaim solvent, pot residue, filter cake, and filtrate are determined using ASTM D-3174. The ash is measured by weighing the residue remaining after burning a 2.5 g sample according to the following heating program: room temperature to 400°C, 2-1/2 hrs., 400°C to 750°C, 1/2 to 1 hr., and 4 hrs. @ 750°C.

c. Volatile matter is determined on raw lignite using ASTM D-3175. Volatile matter (corrected for moisture content) is measured by establishing the loss in weight resulting from heating a 0.5 g sample at a carefully controlled heating rate from room temperature to 950°C in approximately 10 minutes.

d. Carbon and hydrogen contents were determined on all autoclave test starting materials and raw and processed coal products using ASTM D-271 beginning with Run 465. Duplicate determinations are made on each liquid or solid sample, using sample sizes ranging from 35 to 100 mg. Carbon and hydrogen are measured by burning a weighed quantity of sample at 850°C with O₂ in the presence of CuO catalyst. The H₂O and CO₂ from the combustion are freed of interfering substances and passed through absorption tubes containing magnesium perchlorate and potassium hydroxide, respectively. The weight gain of each absorption tube is used to calculate the carbon and hydrogen contents of the sample on a moisture-free basis.

e. Total sulfur is determined on all starting materials and raw and processed coal products by ASTM D-1552 using a LECO Model 521-500 induction furnace with a LECO Model 532 automatic titrator. Sample size varied from 35 mg to 100 mg. The sample is burned at high temperature in a stream of oxygen, which converts most of the sulfur present in the sample to SO₂. Products of combustion are swept into an automatic titra-

tion unit cell containing acidic KI and a starch solution. A deep blue color, developed in the cell by adding a small amount of KIO_3 , establishes an end point color for the electric eye sensor in the automatic titrator. As the combustion products from the sample are swept into the titrator cell, the SO_2 bleaches the starch-iodine color in the titration cell, causing additional KIO_3 titrant to be added automatically for restoration of the original starch-iodine end point color. Sodium azide is added to the HCl in the titration cell to react preferentially with any nitrogen in the sample, thus preventing interference with the iodometric reaction. The amount of KIO_3 titrant required to restore the titration cell to original end point color as indicated by the buret reading is a measure of the sulfur content of the sample. A standardization factor based on analysis of sulfur in $AlK(SO_4)_2 \cdot 12H_2O$ is used to obtain accurate results.

f. Total nitrogen was not determined routinely on any samples from the runs included in Interim Report No. 2. However, when total nitrogen in as-received lignite, solvent, or liquefaction products was determined, the Kjeldahl-Gunning method (ASTM D-271) is used. In this method, approximately 0.5 to 1.0 gram of sample is pre-reduced for 30 minutes at room temperature using 5 grams of sodium thiosulfate and 30 ml of concentrated sulfuric acid in an 800 ml Kjeldahl digestion flask. $HgO-K_2SO_4$ catalyst is then added to the digestion flask, and the flask is heated for 8 to 15 hours while acid digestion of the sample proceeds. Following digestion, a standard acid-base titration is used to determine the amount of nitrogen in the sample.

g. Specific gravity, $d_{60}/60$ F, of input solvent, reclaim solvent, and filtrate is measured using the method of ASTM D-1298. Readings are made using a glass hydrometer at room temperature and corrected to specific gravity values at 60°F using international standard tables.

h. Extractable hydrocarbons in the filter cake and residue products are determined by ASTM D-473, using pyridine as the extraction solvent. The sample is placed in a weighed cellulose Soxhlet thimble and extracted with pyridine for 6 to 8 hours. Following extraction, the cellulose thimble containing pyridine insoluble residue is placed in an oven and air-dried at 105°C for 8 to 12 hours. The thimble is cooled and weighed, and the content of pyridine extractable material is calculated on a moisture and ash free basis.

i. Calorific value of solvent-refined lignite is determined by ASTM D-2015 using a Parr series 1200 calorimeter.

2. Special Test Procedures

a. IR ratio is used to measure the relative intensity of the infra-red absorbances of aromatic and aliphatic hydrogens in liquefaction solvents at wave lengths of 3.28 and

3.41 microns, respectively. Thus, the degree of hydrogenation (or dehydrogenation) of a solvent during a liquefaction test can be approximated by comparing IR measurements before and after the liquefaction test. When the solvent is hydrogenated, the absorption intensity of the aliphatic hydrogen (3.41μ) increases and that of the aromatic hydrogen (3.28μ) decreases. With comparable solvent fractions, it has been found that the IR ratio can be correlated with the hydrogen content of the solvent which determined using ASTM D-271.(4)

A Perkin-Elmer Model 700 Infra-Red Spectrophotometer is used to measure the IR spectra of the sample over a wavelength range of 2.5 to 16 microns. A sodium chloride cell with 0.037 mm wall thickness contains the sample which is diluted 1:5 with carbon disulfide. At the completion of the IR scan, a baseline is drawn on the IR trace from approximately 2.5 microns to 5 microns. Intensity is read directly from the chart in absorbance units at both 3.28 and 3.41 microns. The IR ratio is computed by dividing the intensity (in absorbance units) at 3.28 microns by the intensity (in absorbance units) at 3.41 microns.

b. Blackness is an empirical test used to measure the relative amount of converted coal in solution in the liquefaction solvent. The sample used for this test is the filtrate from the suction filtration of the autoclave product. The test consists of dissolving approximately 50 mg of sample in pyridine and diluting to 100 ml with reagent grade pyridine. A Bausch and Lomb Spectronic 20 Spectrophotometer is used to measure absorbance of the solution at 550 μ . The absorbance reading is divided by the actual sample weight, yielding data in absorption/gram/100 ml. This is used to estimate the relative amount of coal in solution, and thus the extent of coal depolymerization in the autoclave.

c. Melting point is determined using a Model 304.5 Pacific Transducer Corporation Melting Point Meter designed to measure the melting points of substances from approximately 120°F to 500°F . The material whose melting point is most commonly measured is the vacuum bottoms, predominately solvent-refined lignite. The meter has a stainless steel platen bar with a temperature gradient from RT to 500°F . A finely ground sample is spread on the platen and a demarcation line between molten and solid sample particles is established in 60 seconds. A thermocouple junction is positioned on the demarcation point and the temperature read is the melting point of the sample.

d. Viscosity is determined at room temperature on input and reclaim coal liquefaction solvent, coal-solvent slurry, and filtrate.

A Brookfield model LVT Synchro-Electric viscometer with a model "C" Helipath stand is generally used to measure viscosity; however, the Helipath stand is not required for the input and reclaim coal liquefaction solvents. Because of the

heterogeneous nature of coal-solvent slurry, a size T-C "T-bar" viscometer spindle rotating at a speed of 12 RPM and traveling vertically through the sample on the Helipath stand was used to measure viscosity. To insure greater uniformity, both the filtrate and coal-solvent slurry samples are mixed for five minutes using a Brookfield counter-rotating mixer just prior to measuring viscosity. For input or reclaim solvents, samples are placed in a beaker at room temperature and viscosity is measured using a Brookfield No. 1 viscometer spindle rotating at 30 RPM. The viscosity is computed by multiplying the scale reading by 2.0, a factor which depends on the viscometer spindle and speed of rotation which were used.

e. Hydrogen sulfide is determined in the product gas immediately after recovery from the autoclave. A 500 ml sample of product gas is withdrawn from the gas bag using a Hamilton S-0500 super syringe. The syringe is then connected to a glass bubbler tube, and the product gas is bubbled through ammoniacal zinc sulfate solution to remove hydrogen sulfide. The amount of hydrogen sulfide absorbed is determined iodometrically and calculated as mol percent of H_2S in the product gas in accordance with ASTM D-2385.

f. Gas specific gravity is also measured on the product gas immediately following collection. A 200 ml glass bulb is evacuated using a high vacuum source and weighed; air is then admitted to the evacuated bulb and the bulb is reweighed to determine the weight of air occupying the bulb. Similarly, a glass bulb for the product gas is evacuated and weighed; the bulb is then connected to the product gas bag and product gas is admitted to the bulb until it attains atmospheric pressure. The bulb is reweighed to determine the weight of product gas. Specific gravity of the product gas sample is then determined by dividing the weight of the sample gas by the weight of a corresponding volume of air at atmospheric pressure. The specific gravity is used to calculate the average molecular weight of the gas and to calculate the weight of gas recovered for material balance purposes.

g. Gas chromatography is used to determine the composition of both input and product gas samples. Analyses are made using a Hewlett-Packard Model 700 Gas Chromatograph equipped with dual 1/4" ID x 4 meter stainless steel columns containing Porasil and 5A molecular sieves, respectively, and a thermal conductivity detector. Argon is used as the carrier gas. Both detector and column temperatures are thermostatically controlled at 100°C. The detector bridge current used is 100 ma, and the carrier gas flow rates are 30 and 60 cc/min for the Porasil and Molecular sieve columns, respectively. Gas samples are charged to the chromatograph under vacuum, which eliminates small errors that may be introduced to the system by fluctuations in atmospheric pressure. A Sargent Model SRG recording potentiometer is used to trace the chromatogram of the gas sample; an integrating device coupled to the recorder allows the analyst to determine areas rapidly

for each constituent peak in the chromatogram. The Porasil column resolves ethane and carbon dioxide and trace amounts of C₃ and C₄ hydrocarbons; the molecular sieve column resolves hydrogen, nitrogen, methane, and carbon monoxide.

Prior to the gas analysis, the gas chromatograph is calibrated with four different gas mixtures containing known compositions of H₂, CO, CO₂, methane, ethane, and nitrogen which bracket the compositions of these constituents in the unknown samples. Calibration curves for each constituent in the known mixtures are constructed and used to compute the volume (mole) percent of each constituent in the gas sample being analyzed.

APPENDIX C

DISTILLATION METHODS

1. Atmospheric Distillation

Atmospheric distillation is performed by ASTM D-246 on all input solvents to determine distillation properties such as initial boiling point and estimated true boiling point curves at atmospheric pressure. The distillation is conducted using the apparatus assembly for flame distillation. A 100 gram sample is distilled in a 300 ml flask. The initial boiling point is recorded, and fractions of RT to 210°C, 210° to 235°C, 235° to 270°C, 270° to 315°C, 315° to 355°C and residue above 355°C are collected and weighed. Distillation test temperatures are adjusted for barometric pressure using the pressure-temperature correction table published in ASTM D-246.

2. Vacuum Distillation

Liquefaction solvent used in the batch autoclave tests is obtained by vacuum distilling, at 1.6 mm Hg, raw solvent received from the supplier. The liquefaction solvent is the fraction boiling between 100° and 230°C.

The filtrate produced during suction filtration of the reactor slurry product is also vacuum distilled at 1.6 mm Hg with three fractions being taken: IBP to 100°C, considered the light oil fraction; 100° to 230°C, which is the reclaim solvent fraction; and 230° and higher (vacuum bottoms). A Vigreux glass fractionating column with an effective length of 15 cm and a straight-tube water-cooled condenser are used for the distillation, which employs a 500 ml flask with a 200 gram sample.

APPENDIX D

COMPUTERIZED DATA REDUCTION

A computer program is used to handle the data obtained from each experimental run. The purpose of the program is twofold: (1) to calculate material balances and (2) to provide parameters for characterizing the process. As the computer program has evolved from the data handling of many types of experiments it will not be discussed in detail.

1. Material Balances

As shown in the block flow diagram, Figure D-1, the output material is handled either as experimentally measureable outputs or as calculated products that would result if the experimental outputs were combined prior to separation. The calculated products are those that would be recovered from a facility that included separation equipment as well as a reactor.

Inputs include weighed quantities of water, solvent, and lignite. The gas inputs are calculated based on calibrations of the reactor made with gases of different compositions as described in the discussion of Autoclave operations in Appendix A.

Experimental outputs include gas, water, light oil, filtrate, cake, pot residue, and residue. The quantity of gas is determined from its volume and density. The water and oil are the weighed quantities collected in the cold traps. The filtrate and cake are weighed after the reactor discharge material is filtered. The pot residue is the quantity of material remaining in the reactor after emptying, while the residue is the material adhering to the distributions plate above the filter.

The calculated products include light oils, reclaimed solvent, vacuum bottoms, and mineral residues. The following describes how the distribution of these calculated products is obtained: The oil is added to the filtrate. The pot residue is extracted with pyridine and the soluble material considered filtrate with the insoluble material considered filter cake. Based on the measured weight fractions of filtrate and cake the residue is split between filtrate and cake. At this point all outputs except gas and water are either assigned to the filtrate or the cake. The pyridine soluble portion of the cake is then considered to be filtrate and the remainder of the cake mineral residues. A vacuum distillation of a portion of the filtrate is the basis for proportioning all the filtrate between light oil, reclaimed solvent, and vacuum bottoms. The vacuum bottoms fraction is sometimes referred to as solvent refined lignite, though in a commercial plant some of this material might have to be used as recycle solvent in order to obtain 100 percent solvent recycle.

Two types of material balances are made: (1) balances based on actual recovery data and (2) balances adjusted to 100 percent recovery. Four actual recovery material balances are made on the experimental outputs and include: total, organic, water, and ash. Percentage recoveries of all four quantities are also calculated and the total percentage recovered reported as "percent closure". The other actual material balance is made on the solvent after the quantity of recoverable solvent has been calculated.

A single material balance is made on a 100 percent recovery basis based on the input MAF lignite. To adjust the material balance to 100 percent it is assumed that all the losses are evenly distributed among the gas, water, and calculated products. The material balance is also an organic or MAF balance. The components of the balance are: gas, liquid, unconverted, and water. Water is included in an MAF balance because part of the input water is changed into hydrogen and carbon dioxide, some of the former is combined with organic material and the latter is removed as gas. Usually, because of the difficulty in analyzing the small samples of some of the products and because of changes due to chemical reactions, the ash input does not equal the ash output. Therefore, the water and ash are combined and the difference between the input and output is assumed to be combined into the organic products. The liquid refers to the MAF lignite converted to light oil and solvent refined lignite. Any reclaimed solvent in excess of that needed for recycle is counted as solvent refined lignite, and any shortage of solvent is made up from the vacuum bottoms and decreases the solvent and refined lignite yield.

2. Characterization Parameters

There are six characterization parameters and all are based on actual recoveries in percent:

- Closure
- Solvent Recoverable
- Vacuum Bottoms Yield
- Liquids Plus Vacuum Bottoms Yield
- Total Extracted
- Net Liquids

The "percentage closure" is based on an overall material balance, while the others are calculated on a MAF basis after normalizing the outputs to 100 percent material balance.

"Percentage closure" is an indication of the total recovery of all inputs and "percentage solvent recoverable" refers to the solvent available for recycle after the liquefaction reaction has been completed.

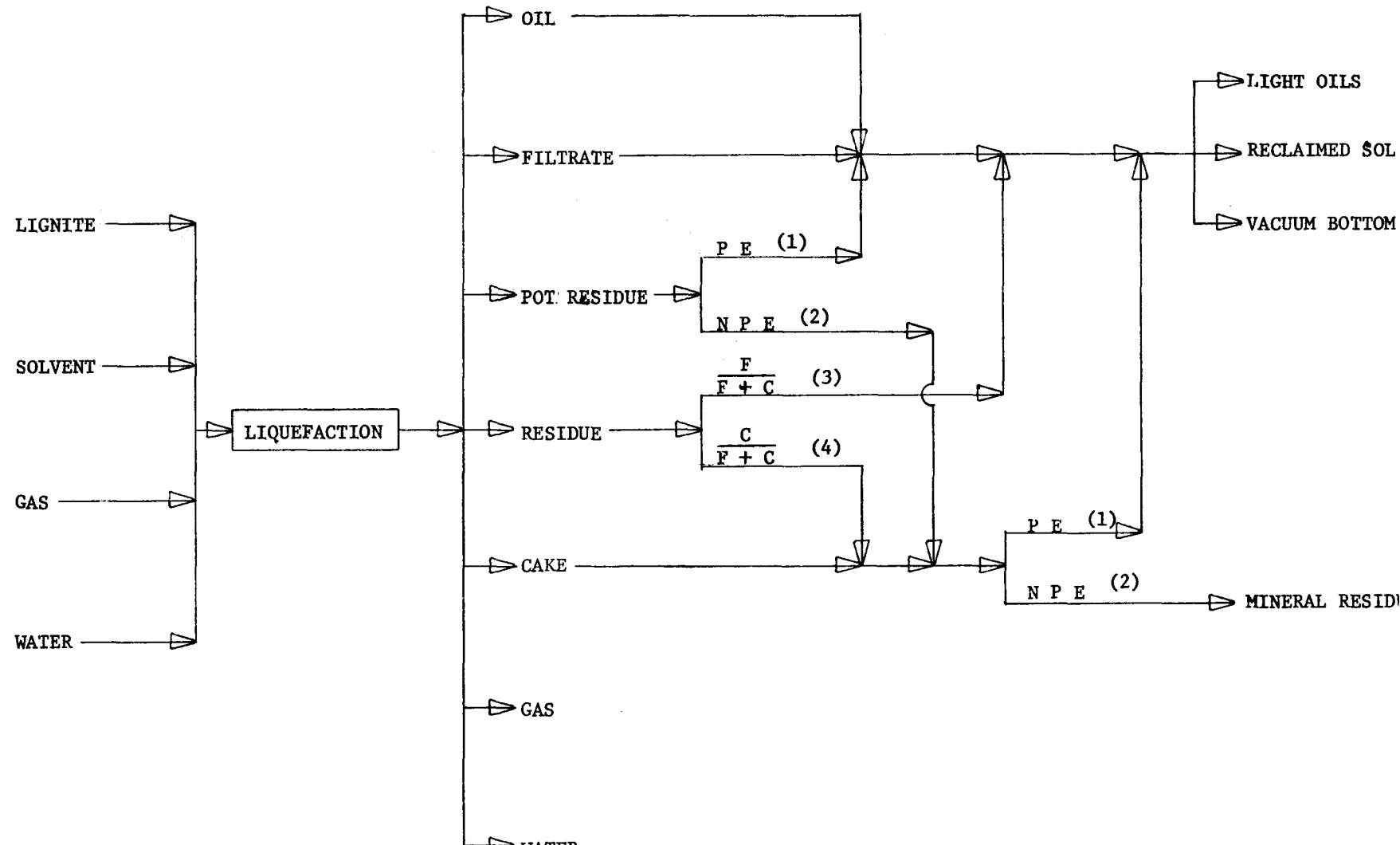
The other four characterization parameters refer to recoveries of the input lignite. "Percentage vacuum bottoms

"yield" is that fraction of the lignite that is converted into material remaining as residue in the vacuum distillation and which might be considered solvent refined lignite. The lignite converted into light oil is added to that converted into the vacuum bottoms to give "percentage liquids plus vacuum bottoms yield". All of the lignite that is converted is reported as "percentage total extracted;" the difference between this percentage and one hundred percent is the lignite that was not converted by liquefaction. The "percentage net liquids" refers to the sum of the light oils, reclaimed solvent, and solvent refined lignite minus the input solvent; thus it represents the net lignite conversion after recovering enough material to make up completely the amount of solvent charged.

INPUTS

EXPERIMENTAL OUTPUTS

CALCULATED PRODUCTS



(1) PYRIDINE EXTRACTABILITY

(2) NON-PYRIDINE EXTRACTABLE

(3) FILTRATE / (FILTRATE + CAKE)

(4) CAKE / (FILTRATE + CAKE)

FIG. . BLOCK FLOW DIAGRAM FOR MATERIAL BALANCE CALCULATIONS

APPENDIX E

COMPLETE AUTOCLAVE RUN DATA

Catalytic Effects of Cations on Lignite Liquefaction	56
Table 1. Alkali Metals and Alkaline Earths	56
Table 2. Transition Metals	57
Table 3. Miscellaneous Metals, Commercial Catalysts, and Base Case Runs	60
Evaluation of Liquefaction Solvents	62
Table 4. Anthracene Oils	62
Table 5. Creosote Oils	65
Table 6. Creosote Oils and Petroleum Derived Solvents	68
Table 7. Petroleum Derived Solvents	71
Table 19. Distillation Data on the Solvents	103
Solvent Recycle Series	74
Table 8. First Pass Tests	74
Table 9. Second and Third Pass Tests	77
Table 10. Fourth and Fifth Pass Tests	80
Effect of Storage on Lignite Reactivity	83
Table 11. 0-12 Weeks	83
Table 12. 24-36 Weeks	86
Table 13. Effect of Reducing Gas on Lignite Liquefaction	89
Table 14. Carbonization of Lignite	91
Table 15. Effect of Carbonization on Lignite Liquefaction	92
Table 16. Effect of Dehydration on Lignite Liquefaction	94
Table 17. Liquefaction of Miscellaneous Lignite Samples	97
Table 18. Liquefaction Product Work-ups Without Filtration	100

NOTE: All autoclave test tables have two parts, (1) Material Balance and Yield Data, and (2) Analytical Data.

TABLE 1 - PART 1: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

Run No.	ALKALI METALS AND ALKALINE EARTHS							
	415	425	426	423	453	416	431	456
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	768	750	750	755	750	766	750	755
Max. Press, psig	3390	3180	3170	3140	2830	3280	3210	2960
Gas Charged	CO/H ₂							
Solvent	UNDRAO-72							
Lignite	72-1X(Na1)	72-1X(Na2)	72-1X(Na3)	72-1X(K1)	72-1X3(K2)	72-1(Ca)	72-1X2(Ca2)	72-1X3(Ca3)
Metal Added	Sodium → Potassium → Calcium →							
Conc., Mmols/Kg	228	265	369	167	385	221	166	142
<u>Material In, grams</u>								
Coal & H ₂ O	317.3	310.2	313.3	308.8	314.8	316.0	310.9	309.5
Solvent	400.2	400.1	399.9	399.3	400.2	400.5	399.9	400.4
Gas	128.0	122.3	118.1	119.2	118.8	98.2	119.8	117.1
Total	845.5	832.6	831.5	827.3	833.8	814.7	830.6	827.0
<u>Material Out, grams</u>								
Filtrate	454.0	467.5	450.0	474.5	408.0	450.0	460.0	428.7
Filter Cake	35.5	43.0	33.5	30.5	32.5	40.5	64.5	69.1
Residues	22.0	22.5	45.5	24.5	29.5	21.5	14.5	30.7
Gas	183.8	187.4	194.9	189.6	207.7	169.1	157.1	177.8
H ₂ O	82.0	78.4	66.1	68.6	56.4	98.3	98.5	101.2
Light Oil	13.0	5.1	12.9	8.9	15.1	11.7	11.7	12.5
Total	790.3	803.9	802.9	796.6	749.2	793.1	806.3	820.0
% Recovery	93.5	96.6	96.6	96.3	89.9	97.3	97.1	99.1
<u>100% Recovery Basis</u>								
Products, grams	33.3	25.4	33.2	38.0	43.4	28.1	41.4	44.1
Light Oil	380.7	382.3	378.3	386.1	335.0	356.7	358.6	350.8
Solvent	122.6	127.8	124.9	114.1	137.8	125.9	141.2	123.9
Vacuum Btms	536.6	535.5	536.4	538.2	516.2	510.7	541.2	518.8
Total Liquid	136.4	135.4	136.5	138.9	116.0	110.2	141.3	118.4
Net Liquids	68.6	71.7	83.7	77.7	112.3	75.5	42.0	62.2
Net Gas	-20.8	-18.7	-31.2	-28.5	-37.0	-4.3	1.6	2.2
Net H ₂ O	1.4	-0.6	-3.3	0.3	-2.5	0.1	-0.3	0.3
Net Ash	14.5	12.0	13.8	11.2	9.9	18.8	15.3	17.3
Unconverted Coal (MAF)	<u>Yields-Wt% MAF Coal</u>							
Net Gas	34.3	35.9	42.0	38.9	56.1	37.7	21.0	31.0
Net Liquid	68.2	67.8	68.4	69.7	58.7	55.0	70.6	59.1
Unconverted	7.2	6.0	6.9	5.6	4.9	9.4	7.7	8.7
Net H ₂ O + Ash	-9.7	-9.7	-17.3	-14.2	-19.7	-2.1	0.7	1.2
<u>Total Liquid Composition (Wt%)</u>								
Light Oil	6.2	4.7	6.2	7.1	8.4	5.5	7.6	8.5
Solvent	70.9	71.4	70.5	71.7	64.9	69.8	66.3	67.6
Vacuum Btms	22.9	23.9	23.3	21.2	26.7	24.7	26.1	23.9
Net Liquid (Wt% MAF Coal)	<u>Net Recovery</u>							
Net Light Oil	16.6	12.7	16.6	19.0	21.7	14.0	20.7	22.0
Net SRL	51.6	55.1	51.8	50.7	37.0	41.0	49.9	37.1
Net Recovery	88.9	92.3	91.4	93.1	75.2	86.7	87.1	86.9
Wt% (100% Rec. Basis)	95.1	95.5	94.6	96.7	83.7	89.0	89.7	87.6

TABLE 1 - PART 2: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

Run No.	ALKALI METALS AND ALKALINE EARTHS							
	415	425	426	423	453	416	431	456
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	768	750	750	755	750	766	750	755
Max. Press., psig	3390	3180	3170	3140	2830	3280	3210	2960
Gas Charged	CO/H ₂							
Solvent	UNDRAO-72							
Lignite	72-1X(Na1)	72-1X(Na2)	72-1X(Na3)	72-1X(K1)	72-1X3(K2)	72-1(Ca)	72-1X2(Ca2)	72-1X3(Ca3)
Metal Added	Sodium			Potassium			Calcium	
Conc., Mmol/Kg	228	265	369	167	385	221	166	142
<u>Analytical Data</u>								
1. <u>Gas Analysis, Mol%</u>								
H ₂	42.1	39.2	44.0	41.0	42.9	40.0	37.1	38.1
CH ₄	4.0	1.7	1.5	1.7	2.7	3.2	1.3	2.9
CO	20.1	20.6	14.1	14.6	6.0	28.6	33.6	31.0
C ₂ H ₆	0.0	0.6	0.6	0.5	1.1	0.5	0.5	1.2
CO ₂	33.1	37.2	39.1	41.5	46.6	26.9	26.7	25.9
H ₂ S	0.7	0.7	0.7	0.7	0.7	0.8	0.8	0.9
Gas Specific Gravity @ RT	0.794	0.795	0.790	0.790	0.809	0.746	0.780	0.787
2. <u>Input Coal</u>								
Volatile Matter, Wt%	28.09	30.35	33.43	30.82	38.72	29.93	32.17	32.83
Ash, Wt%	2.75	3.45	4.57	3.14	4.70	3.32	3.56	3.17
Moisture, Wt%	34.20	28.31	30.80	29.63	31.49	33.31	31.36	27.49
Sulfur, Wt% (1)	1.30	1.22	1.18	1.27	0.45	1.40	1.29	---
3. <u>Input Solvent</u>								
IR ratio	1.30	1.46	1.46	1.46	1.53	1.50	1.52	1.53
4. <u>Coal-Solvent Slurry</u>								
5. <u>Cake</u>								
Ash, Wt%	23.35	19.63	15.96	23.25	23.32	16.81	14.45	12.67
Sulfur, Wt%	3.19	2.37	1.86	2.59	1.32	2.10	1.72	1.38
Pyridine Sol., Wt% (2)	53.46	68.83	67.88	55.41	69.29	52.94	75.34	74.40
6. <u>Filtrate</u>								
Ash, Wt%	0.00	0.00	0.01	0.02	0.09	0.01	0.02	0.00
7. <u>Vacuum Btms</u>								
Ash, Wt%	0.01	0.01	0.06	0.16	1.41	0.01	0.01	0.05
Sulfur, Wt%	0.31	0.30	0.40	0.31	0.25	0.25	0.35	0.33
8. <u>Residue</u>								
Ash, Wt%	5.48	5.53	14.91	11.52	17.26	19.18	9.98	2.54
Sulfur, Wt%	0.65	---	1.67	2.47	0.82	2.05	---	0.52
Pyridine Sol., Wt% (2)	95.90	95.16	87.32	99.99	96.30	84.31	91.23	98.22
9. <u>Light Oil</u>								
10. <u>Product Solvent</u>								
IR ratio	0.88	0.83	0.96	0.91	0.93	0.97	1.03	1.00

(1) Calculated on a moisture-free basis

(2) Calculated on a ash-free basis

TABLE 2 - PART 1: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

TRANSITION METALS								
Run No.	424	428	427	429	437	432	433	435
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	745	750	750	755	750	750
Max. Press, psia	3260	3180	3140	3230	3220	3270	3160	3050
Gas Charged	CO/H ₂				Hydrogen			
Solvent	UNDRAO-72							
Lignite	72-1X(Fe)	72-1X2(Fe2)	72-1X(Co)	72-1X2(Co2)	72-1X2(Ni)	72-1X2(Ni2)		
Metal Added	Iron		Cobalt		Nickel			
Conc. Mmols/Kg	152	232	107	320	320	140	314	314
<u>Material In, grams</u>								
Coal & H ₂ O	307.5	308.6	306.4	310.9	311.4	308.2	311.2	310.9
Solvent	399.5	400.2	398.9	399.6	400.4	399.6	399.9	399.9
Gas	124.8	116.1	116.9	119.4	14.7	120.1	120.3	14.7
Total	831.7	824.9	822.3	829.9	726.5	827.9	831.4	725.5
<u>Material Out, grams</u>								
Filtrate	467.5	472.5	470.0	436.5	451.5	455.0	464.0	464.5
Filter Cake	35.5	45.0	51.5	81.5	72.0	61.0	61.5	54.5
Residues	18.5	15.5	19.0	21.0	17.0	20.0	13.5	10.5
Gas	174.0	165.9	160.8	157.7	38.8	164.3	160.0	33.9
H ₂ O	87.2	91.8	97.3	104.3	123.4	95.2	95.0	122.1
Light Oil	13.8	10.2	7.7	10.7	11.1	8.3	11.5	15.9
Total	796.5	800.9	806.3	811.7	713.8	803.8	805.5	701.4
% Recovery	95.8	97.1	98.1	97.8	98.3	97.1	96.9	96.7
<u>100% Recovery Basis</u>								
<u>Products, grams</u>								
Light Oil	31.7	49.0	29.4	38.0	50.8	46.2	49.2	43.6
Solvent	373.5	352.9	372.7	350.8	342.7	351.2	355.2	362.3
Vacuum Btms	135.3	134.8	132.6	136.7	143.2	137.3	137.0	137.1
Total Liquid	540.5	536.7	534.7	525.5	536.7	534.7	541.4	543.0
Net Liquids	141.0	136.5	135.8	125.9	136.3	135.1	141.5	143.1
Net Gas	56.8	54.8	47.0	41.8	24.8	49.1	44.8	20.4
Net H ₂ O	-8.8	-5.3	0.0	6.9	25.7	-1.7	-1.7	26.6
Net Ash	-0.2	0.4	0.6	2.8	0.3	-0.2	0.1	0.5
Unconverted Coal (MAF)	10.9	13.7	15.0	22.4	13.1	17.3	15.6	9.3
<u>Yields-Wt% MAF Coal</u>								
Net Gas	28.5	27.4	23.7	20.9	12.4	24.6	22.4	10.2
Net Liquid	70.6	68.0	68.4	63.0	68.0	67.7	70.6	71.6
Unconverted	5.4	7.0	7.6	11.2	6.6	8.7	7.8	4.7
Net H ₂ O + Ash	-4.5	-2.4	0.3	4.9	13.0	-1.0	-0.8	13.5
<u>Total Liquid Composition (Wt%)</u>								
Light Oil	5.9	9.1	5.5	7.2	9.5	8.6	9.1	8.0
Solvent	69.1	65.8	69.7	66.8	63.8	65.7	65.6	66.7
Vacuum Btms	25.0	25.1	24.8	26.0	26.7	25.7	25.3	25.3
<u>Net Liquid (Wt% MAF Coal)</u>								
Net Light Oil	15.8	24.5	14.8	19.0	25.4	23.1	24.6	21.8
Net SRL	54.8	43.5	53.6	44.0	42.6	44.6	46.0	49.8
<u>Net Recovery</u>								
..%	89.6	85.6	91.7	85.9	84.1	85.3	86.1	87.6
Wt% (100% Rec. Basis)	93.5	88.2	93.5	87.8	85.6	87.8	88.9	90.6

TABLE 2 - PART 2: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

TRANSITION METALS								
Run No.	424	428	427	429	437	432	433	435
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	745	750	750	755	750	750
Max. Press, psia	3260	3180	3140	3230	3220	3270	3160	3050
Gas Charged	CO/H ₂				Hydrogen			
Solvent	UNDRAO-72							
Lignite	72-1X(Fe)	72-1X2(Fe2)	72-1X(Co)	72-1X2(Co2)	72-1X2(Ni)	72-1X2(Ni2)		
Metal Added	Iron		Cobalt		Nickel			
Conc. Mmols/Kg	152	232	107	320	320	140	314	314
<u>Analytical Data</u>								
1. <u>Gas Analysis, Mol%</u>								
H ₂	35.5	36.6	34.4	36.0	86.1	37.4	34.4	85.0
CH ₄	1.8	1.9	1.8	1.5	1.7	1.4	1.8	2.3
CO	31.3	27.2	37.6	43.1	0.0	35.9	35.9	0.0
C ₂ H ₆	0.6	0.4	0.5	0.5	0.5	0.6	0.8	0.8
CO ₂	30.0	33.3	25.2	18.8	11.4	24.3	26.7	11.5
H ₂ S	0.8	0.6	0.5	0.1	0.3	0.4	0.4	0.4
Gas Specific Gravity @ RT	0.789	0.803	0.783	0.748	0.223	0.759	0.779	0.233
2. <u>Input Coal</u>								
Volatile Matter, Wt%	33.79	31.42	32.34	30.52	30.74	31.17	31.43	31.29
Ash, Wt%	2.73	2.88	2.98	3.70	3.73	2.94	3.71	3.69
Moisture, Wt%	26.07	30.34	27.95	30.61	30.10	28.93	30.34	30.66
Sulfur, Wt% (1)	1.28	1.29	1.25	1.10	1.10	2.94	1.26	1.26
3. <u>Input Solvent</u>								
IR ratio	1.46	1.47	1.47	1.52	1.53	1.52	1.53	1.53
4. <u>Coal-Solvent Slurry</u>								
Brookfield Visc., cp	---	---	---	---	---	---	---	---
5. <u>Cake</u>								
Ash, Wt%	17.32	17.01	15.46	11.92	14.52	10.50	16.35	19.29
Sulfur, Wt%	2.86	3.76	3.19	2.55	2.98	2.09	3.53	3.98
Pyridine Sol, Wt% (2)	65.84	66.86	69.54	71.74	81.42	73.33	73.16	80.50
6. <u>Filtrate</u>								
Ash, Wt%	0.07	0.00	0.00	0.00	0.03	0.00	0.08	0.00
7. <u>Vacuum Btms</u>								
Ash, Wt%	0.14	0.00	0.00	0.00	0.05	0.00	0.00	0.00
Sulfur, Wt%	0.32	0.31	0.28	0.28	0.26	0.31	0.25	0.23
8. <u>Residue</u>								
Ash, Wt%	5.14	10.33	8.90	11.97	7.81	14.40	8.39	14.20
Sulfur, Wt%	1.43	---	---	---	---	---	---	---
Pyridine Sol, Wt% (2)	99.99	93.97	92.62	89.32	94.23	86.80	88.94	99.98
9. <u>Light Oil</u>								
10. <u>Product Solvent</u>								
IR ratio	0.85	0.94	0.95	1.14	1.14	1.03	1.00	0.88

(1) Calculated on a moisture-free basis

(2) Calculated on an ash-free basis

TABLE 3 - PART 1: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

MISCELLANEOUS METALS, COMMERCIAL CATALYSTS, AND BASE CASE RUNS

Run No.	434	436	438	439	421	455	454	520
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	745	755	750	755	750	750	772
Max. Press, psia	2880	2980	3120	2880	3120	2870	2880	3130
Gas Charged				CO/H ₂				
Solvent				UNDRAO-72				
Lignite				72-1X2(Mo) 72-1X2(Mo2)	72-1(F) 72-1X2(Ni-Mo)	72-1(D) 72-1(I)	72-1X3	UNDCAO-73 73-2(C)
Metal Added				Molybdenum	HDS-3A Cat	None		HDS-3A
Conc., Mmols/Kg	150	447	55Ni, 138Mo	18Ni, 48Mo	---	---	---	
<u>Material In, grams</u>								
Coal & H ₂ O	308.4	307.9	329.6	323.2	320.4	322.6	307.1	322.8
Solvent	400.2	399.6	399.4	399.9	399.6	399.6	400.2	399.3
Gas	121.4	119.3	122.0	119.9	120.9	119.9	118.5	158.0
Total	830.0	826.8	851.0	843.0	840.9	842.1	825.8	879.2
<u>Material Out, grams</u>								
Filtrate	384.5	356.0	382.0	386.5	418.5	434.9	435.0	392.2
Filter Cake	107.0	118.0	129.0	105.0	83.5	71.4	68.5	95.4
Residues	32.0	24.5	31.5	32.0	29.5	31.4	24.0	44.3
Gas	192.5	186.8	189.6	190.6	188.1	196.4	161.7	243.7
H ₂ O	67.3	69.4	57.8	60.6	71.3	73.1	91.2	52.7
Light Oil	11.7	13.1	8.2	39.4	10.7	9.9	13.3	6.6
Total	795.0	767.8	798.1	814.1	801.6	817.1	793.7	834.9
<u>% Recovery</u>								
	95.8	92.9	93.8	96.6	95.3	97.0	96.1	95.0
<u>100% Recovery Basis</u>								
<u>Products, grams</u>								
Light Oil	52.8	59.2	46.5	54.6	20.2	42.2	27.5	21.4
Solvent	372.2	325.2	360.0	353.8	398.4	367.9	364.4	380.9
Vacuum Btms	106.8	129.2	135.2	133.2	122.8	117.6	144.8	119.5
Total Liquids	531.8	513.6	541.7	541.6	541.4	527.7	536.7	521.8
Net Liquids	131.6	114.0	142.3	141.7	141.8	128.1	136.5	122.5
Net Gas	79.6	81.9	80.2	77.5	76.4	82.5	49.7	98.6
Net H ₂ O	-29.5	-25.0	-43.4	-36.9	-25.5	-24.3	-4.9	-44.1
Net Ash	1.6	7.3	-0.6	0.0	1.3	-3.0	0.2	0.9
Unconverted Coal (MAF)	16.8	21.6	16.0	17.8	6.3	16.7	18.7	21.0
<u>Yields-Wt% MAF Coal</u>								
Net Gas	39.8	41.0	41.2	38.7	38.1	41.2	24.8	49.6
Net Liquid	65.7	57.0	73.2	70.9	70.8	64.0	68.2	61.6
Unconverted	8.4	10.8	8.2	8.9	3.2	8.4	9.4	10.5
Net H ₂ O + Ash	-13.9	-8.8	-22.6	-18.5	-12.1	-13.6	-2.4	-21.7
<u>Total Liquid Composition (Wt%)</u>								
Light Oil	9.9	11.5	8.6	10.1	3.7	8.0	5.1	4.1
Solvent	70.0	63.3	66.4	65.3	73.6	69.7	67.9	73.0
Vacuum Btms	20.1	25.2	25.0	24.6	22.7	22.3	27.0	22.9
<u>Net Liquid (Wt% MAF Coal)</u>								
Net Light Oil	26.4	29.6	23.9	27.3	10.1	21.1	13.8	27.5
Net SRL	39.3	27.4	49.3	43.6	60.7	42.9	54.4	34.1
<u>Solvent Recovery</u>								
Wt%	89.1	75.6	84.5	85.5	95.0	89.3	87.5	8
Wt% (100% Rec. Basis)	93.0	81.4	90.1	88.5	99.7	92.0	91.0	+

TABLE 3 - PART 2: CATALYTIC EFFECTS OF CATIONS ON LIGNITE LIQUEFACTION

MISCELLANEOUS METALS, COMMERCIAL CATALYSTS, AND BASE CASE RUNS

Run No.	434	436	438	439	421	455	454	520
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	745	755	750	755	750	750	750
Max. Press, psia	2880	2980	3120	2880	3120	2870	2880	3130
Gas Charged				CO/H ₂				
Solvent					UNDRAO-72			
Lignite	72-1X2(Mo)	72-1X2(Mo2)	72-1(F)	72-1X2(Ni-Mo)	72-1(D)	72-1(I)	72-1X3	UNDCAO-73
Metal Added	Molybdenum		HDS-3A	Cat		None		73-2(C)
Conc., Mmols/Kg	150	447	55Ni,138Mo	18Ni,48Mo	---	---	---	HDS-3A
<u>Analytical Data</u>								
1. <u>Gas Analysis, Mol%</u>								
H ₂	34.5	39.0	41.9	38.1	40.6	39.0	34.4	41.7
CH ₄	2.6	2.1	1.8	2.4	1.5	2.5	3.1	1.7
CO	7.5	7.4	9.1	6.6	15.4	18.5	33.5	11.7
C ₂ H ₆	0.7	0.7	0.6	0.4	0.8	0.9	1.0	0.9
CO ₂	54.4	50.8	46.1	51.6	41.1	38.5	27.2	43.4
H ₂ S	0.3	0.0	0.5	0.9	0.6	0.6	0.8	0.6
Specific Gravity @ RT	0.947	0.915	0.837	0.928	0.796	0.815	0.815	0.824
2. <u>Input Coal</u>								
Volatile Matter, Wt%	29.96	29.97	30.33	29.59	31.07	30.30	31.07	30.49
Ash, Wt%	2.84	2.86	9.18	7.44	6.25	7.19	2.31	7.38
Moisture, Wt%	29.94	29.40	31.01	28.86	31.32	30.52	32.33	29.53
Sulfur, Wt% (1)	1.15	1.36	0.78	0.82		0.68	0.73	0.85
3. <u>Input Solvent</u>								
IR ratio	1.53	1.53	1.53	1.53		1.53	1.53	1.67
4. <u>Coal-Solvent Slurry</u>								
5. <u>Cake</u>								
Ash, Wt%	7.97	11.14	19.98	17.81	21.27	25.53	8.58	18.45
Sulfur, Wt%	2.31	2.65	1.34	1.75	1.62	1.99	1.49	0.89
Pyridine Sol., Wt% (2)	86.46	83.33	90.56	83.00	93.60	75.10	74.90	78.80
6. <u>Filtrate</u>								
Ash, Wt%	0.06	0.09	0.00	0.00	0.29	0.03	0.03	0.03
7. <u>Vacuum Btms</u>								
Ash, Wt%	0.16	0.20	0.17	0.11	0.78	0.03	0.02	0.17
Sulfur, Wt%	0.17	0.25	0.16	0.04		0.26	0.30	0.20
8. <u>Residue</u>								
Ash, Wt%	3.59	5.95	6.61	18.87	4.86	3.76	7.07	16.16
Sulfur, Wt%	---	---	---	---		0.64	0.70	1.33
Pyridine Sol., Wt% (2)	96.34	97.09	98.37	99.99	99.99	93.24	93.01	95.58
9. <u>Light Oil</u>								
10. <u>Product Solvent</u>								
IR ratio	0.68	0.74	0.77	0.77		0.90	0.90	0.81

(1) Calculated on a moisture-free basis

(2) Calculated on an ash-free basis

TABLE 4 - PART 1: EVALUATION OF LIQUEFACTION SOLVENTS

ANTHRACENE OILS									
Run No.	455	511	468	465	483	484	510	471	480
<u>Test Conditions</u>									
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	750	755	750	755	755	755	755	755	750
Max. Press, psia	2870	3170	3130	3060	3130	3130	3220	3200	3200
Gas Charged	CO/H ₂								
Solvent	UNDRAO-73	UNDHRAO-73	72-1(K)	72-1(K)	UNDCAO-73	72-1(L)	72-1(S)	UNDHCAO-73	72-1(K)
Fraction	Standard	Topped	Standard	Standard	Standard	Standard	Topped	Standard	Standard
Lignite	72-1(I)	72-1(T)							
<u>Material In, grams</u>									
Coal & H ₂ O	322.6	325.9	322.5	323.3	322.2	323.3	327.8	320.9	322.3
Solvent	399.6	399.6	400.2	399.1	399.4	399.9	399.8	400.2	399.6
Gas	119.9	145.6	141.1	145.9	142.0	146.5	148.8	143.1	151.9
Catalyst	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	842.1	871.1	863.7	868.3	863.6	869.7	867.4	864.3	873.8
<u>Material Out, grams</u>									
Filtrate	434.9	363.1	327.0	351.4	394.7	437.9	374.4	456.5	419.1
Filter Cake	71.4	122.6	195.0	134.0	91.3	60.0	116.6	43.6	67.1
Residues	31.4	49.2	45.5	45.3	31.9	36.5	43.6	50.4	44.1
Gas	191.8	220.3	214.7	211.1	224.2	228.1	221.4	217.0	234.6
H ₂ O	73.1	74.4	70.1	69.9	73.0	74.9	79.9	77.2	77.0
Light Oil	9.9	7.6	8.6	8.3	9.2	9.3	4.4	7.7	6.8
Catalyst Gain	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	812.5	837.2	860.9	820.0	824.3	846.7	840.3	852.4	848.7
<u>% Recovery</u>									
	96.5	96.1	99.7	94.4	95.4	97.4	95.9	98.6	97.1
<u>100% Recovery Basis</u>									
<u>Products, grams</u>									
Light Oil	42.4	31.6	20.7	26.1	26.0	30.3	38.3	31.9	23.3
Solvent	370.0	343.0	404.1	384.7	368.9	381.8	338.4	402.0	380.1
Vac. Btms	118.3	140.6	116.9	123.0	117.8	109.5	138.4	100.8	116.1
Total Liquid	530.7	515.2	541.7	533.8	512.7	521.6	515.1	534.7	519.5
Net Liquids	131.1	115.6	141.5	134.7	113.3	121.7	115.3	134.5	119.9
Net Gas	78.9	83.6	74.5	77.7	82.2	87.7	82.1	77.0	89.7
Net H ₂ O	-23.8	-22.3	-29.4	-25.6	-23.1	-22.8	-16.4	-21.5	-20.3
Net Ash	-2.9	-4.8	-7.1	-4.2	-1.3	-3.1	-2.2	-0.7	-1.5
Unconverted Coal (MAF)	16.9	27.5	20.0	15.9	17.8	16.1	21.5	11.4	94.1
<u>Yields-Wt% MAF Coal</u>									
Net Gas	39.5	41.9	37.2	39.0	46.5	43.9	41.1	38.4	44.8
Net Liquid	65.5	57.9	70.9	67.5	56.9	61.0	57.6	67.0	60.0
Unconverted	8.4	13.8	10.0	8.4	8.9	8.0	10.6	5.7	6.1
Net H ₂ O + Ash	-13.4	-13.6	-18.1	-14.9	-12.3	-12.9	-9.3	-11.1	-10.9
<u>Total Liquid Composition (Wt%)</u>									
Light Oil	8.0	6.1	3.8	4.9	5.1	5.8	7.4	6.0	4.5
Solvent	69.7	66.6	74.6	72.1	71.9	73.2	65.7	75.2	73.2
Vac. Btms.	22.3	27.3	21.6	23.0	23.0	21.0	26.9	18.8	22.3
<u>Net Liquid (Wt% MAF Coal)</u>									
Net Light Oil	21.2	2.6	10.3	13.1	13.0	15.1	12.3	15.9	11.7
Net SRL	44.3	55.3	60.6	54.4	43.9	45.9	45.3	51.1	48.3
<u>% Recovery</u>									
	89.3	82.5	100.7	91.0	88.2	93.0	81.2	99.0	92.4
... (100% Rec. Basis)	92.6	85.9	101.0	96.4	92.4	95.5	84.6	100.4	95.1

TABLE 4 - PART 2: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	ANTHRACENE OILS								
	455	511	468	465	483	484	510	471	480
<u>Test Conditions</u>									
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	750	755	750	755	755	755	755	755	750
Max. Press, psia	2870	3170	3130	3060	3130	3130	3220	3200	3200
Gas Charged				CO/H ₂					
Solvent	UNDRAO-73	UNDRAO-73	UNDCAO-73		UNDCAO-73		UNDHCAO-73		UNDHCAO-73
Fraction	Standard	Topped	Standard	Standard	Standard	Standard	Topped	Standard	Standard
Lignite	72-1(I)	72-1(T)		72-1(K)		72-1(L)	72-1(S)		72-1(K)
<u>Analytical Data</u>									
1. <u>Gas Analysis</u>									
H ₂	39.0	41.5	46.6	40.0	42.3	42.1	41.7	45.0	40.3
CH ₄	2.5	2.1	1.9	1.6	2.1	2.1	1.5	2.3	1.8
CO	18.5	22.4	21.2	18.8	20.6	22.3	22.7	19.7	23.5
C ₂ H ₆	0.9	0.6	0.8	0.7	1.0	0.9	0.7	0.9	0.8
CO ₂	38.5	32.7	29.0	38.3	33.3	32.0	32.8	31.5	33.3
H ₂ S	0.6	0.7	0.5	0.6	0.7	0.6	0.6	0.6	0.3
Gas Specific Gravity @ RT	0.815	0.810	0.786	0.814	0.813	0.820	0.801	0.786	0.802
2. <u>Input Coal</u>									
Volatile Matter, Wt%	30.30	32.13	30.15	30.00	30.49	31.60	31.01	32.16	31.11
Ash, Wt%	7.19	8.60	7.09	7.53	7.27	7.80	8.82	6.76	7.28
Moisture, Wt%	30.52	25.98	30.06	30.04	28.84	25.87	28.51	28.29	28.75
Carbon, Wt% (1)	---	64.27	63.57	---	63.08	62.25	63.22	---	63.08
Hydrogen, Wt% (1)	---	3.83	3.99	---	4.43	4.09	3.64	---	4.43
Sulfur, Wt% (1)	0.68	1.28	1.10	0.95	1.16	1.14	1.14	1.10	1.10
3. <u>Input Solvent</u>									
Carbon, Wt%	---	89.99	91.39	90.78	91.36	91.36	91.09	91.58	91.75
Hydrogen, Wt%	---	5.75	6.30	5.92	6.00	6.00	6.08	6.33	6.40
Sulfur, Wt%	---	0.57	0.23	0.48	0.55	0.55	0.59	0.25	0.21
Specific Gravity 60/60	---	1.157	1.095	1.112	1.116	1.116	1.124	1.100	1.101
IR ratio	1.53	1.54	0.97	1.66	1.65	1.65	1.67	0.93	0.96
4. <u>Coal-Solvent Slurry</u>									
Brookfield Visc., cp (2)	---	---	---	---	---	---	---	---	---
5. <u>Cake</u>									
Ash, Wt%	25.53	15.36	7.11	12.00	20.39	28.45	19.18	29.79	27.29
Carbon, Wt%	---	75.86	85.16	79.98	68.73	61.08	72.55	61.89	63.05
Hydrogen, Wt%	---	5.06	6.02	5.49	4.51	3.89	4.76	3.99	4.25
Sulfur, Wt%	1.99	1.58	0.55	1.24	1.85	2.78	1.78	3.11	2.39
Pyridine Sol., Wt%	75.10	82.58	96.07	95.47	81.77	67.70	86.28	74.31	83.06

(1) Calculated on a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C

TABLE 4 - PART 2 CONT.: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	455	511	468	465	483	484	510	471	480
6. <u>Filtrate</u>									
Ash, Wt%	0.00	0.00	0.03	0.31	0.02	0.00	0.06	0.02	0.00
Carbon, Wt%	--	89.16	90.21	89.50	90.21	89.59	84.59	90.35	90.00
Hydrogen, Wt%	--	6.28	6.37	6.26	6.35	6.38	6.21	6.52	6.49
Sulfur, Wt%	--	0.46	0.20	0.40	0.33	0.33	0.39	0.18	0.18
Specific Gravity 60/60	--	1.135	1.131	1.150	1.136	1.126	1.141	1.160	1.131
7. <u>Vacuum Btms.</u>									
Ash, Wt%	0.03	0.11	0.70	1.49	0.09	0.09	0.19	0.05	0.06
Carbon, Wt%	--	88.24	87.41	86.26	87.70	88.13	88.23	87.76	87.17
Hydrogen, Wt%	--	5.71	5.36	5.63	5.53	5.74	5.56	5.70	5.76
Sulfur, Wt%	0.26	0.28	0.20	0.36	0.24	0.19	0.28	0.20	0.18
8. <u>Residue</u>									
Ash, Wt%	3.76	3.57	7.15	4.66	7.09	12.83	8.22	25.26	9.11
Carbon, Wt%	--	86.35	88.12	88.76	86.68	78.56	83.82	64.86	79.16
Hydrogen, Wt%	--	6.10	6.32	6.32	6.19	6.97	5.82	4.83	5.73
Sulfur, Wt%	0.64	0.66	0.37	0.44	0.76	0.54	0.96	1.16	0.30
Pyridine Sol., Wt%	93.24	95.14	99.99	98.90	92.24	99.14	94.58	90.38	98.33
9. <u>Light Oil</u>									
Carbon, Wt%	--	81.36	84.78	--	84.99	85.09	86.52	84.66	86.35
Hydrogen, Wt%	--	8.86	8.93	--	8.50	8.39	8.24	9.15	9.03
Sulfur, Wt%	--	0.34	0.30	--	0.29	0.38	0.42	0.23	0.20
10. <u>Product Solvent</u>									
Carbon, Wt%	--	90.99	91.24	91.00	91.33	92.46	90.16	91.36	91.10
Hydrogen, Wt%	--	6.62	6.45	6.31	6.52	5.66	6.34	6.43	6.39
Sulfur, Wt%	--	0.46	0.13	0.36	0.39	0.42	0.41	0.21	0.23
Specific Gravity 60/60	--	1.105	1.097	1.105	1.105	1.106	1.109	1.097	1.102
IR Ratio	0.90	0.87	0.79	0.91	0.87	0.86	0.84	0.73	0.76

TABLE 5 - PART 1: EVALUATION OF LIQUEFACTION SOLVENTS

CREOSOTE OILS									
Run No.	447	491	463	479	464	475	462	474	448
<u>Test Conditions</u>									
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	755	750	750	750	755	755	755	750	770
Max. Press, psia	3310	3040	3080	3280	3140	3160	3220	3220	3080
Gas Charged									
Solvent	UNDRCO-72(2)	UNDRCO-73	UNDRCO-73	UNDRCO-72	UNDRCO-73	UNDRCO-73	UNDRCO-73	UNDRCO-73	UNDRCO-72(3)
Fraction	Standard	Standard	Topped	Standard	Standard	Standard	Standard	Standard	Standard
Lignite	72-1(G)	72-1(M)	72-1(K)	72-1(K)	72-1(H)	72-1(K)	72-1(K)	72-1X, 2(B)	
<u>Material In, grams</u>									
Coal & H ₂ O	321.2	321.2	324.3	322.2	323.7	320.5	317.5	321.1	307.6
Solvent	399.6	397.7	400.5	399.6	399.6	399.0	399.6	399.9	399.8
Gas	120.0	142.6	133.5	142.6	145.0	145.1	138.9	144.7	120.9
Total	840.8	861.5	858.3	864.3	868.3	864.6	856.0	865.7	828.3
<u>Material Out, grams</u>									
Filtrate	406.5	438.5	426.5	426.3	380.0	457.0	421.8	449.0	278.0
Filter Cake	36.0	48.0	82.5	56.5	101.0	65.5	50.0	46.5	66.0
Residues	65.0	50.5	35.4	58.7	47.0	24.9	29.0	55.8	96.0
Gas	181.2	213.4	208.7	206.9	218.8	221.8	214.0	217.0	159.4
H ₂ O	71.5	70.0	64.3	86.4	63.4	70.7	73.9	73.7	108.0
Light Oil	38.5	3.3	11.2	6.9	10.4	4.9	27.6	8.2	70.5
Total	798.7	823.7	828.6	841.7	820.6	844.8	816.3	850.2	777.9
<u>% Recovery</u>	95.0	95.6	96.5	97.4	94.5	97.7	95.4	98.2	93.9
<u>100% Recovery Basis</u>									
<u>Products, grams</u>									
Light Oil	40.5	46.0	120.3	98.5	103.3	72.3	30.2	103.8	75.1
Solvent	330.4	372.1	291.0	307.1	298.0	342.8	278.7	322.3	207.1
Vac. Btms	153.6	110.7	122.4	115.7	120.1	110.3	206.2	109.7	184.4
Total Liquid	524.5	528.8	533.7	521.3	521.4	525.4	515.1	535.8	466.6
Net Liquids	124.9	131.1	133.2	121.7	121.8	126.4	115.5	135.9	66.8
Net Gas	70.7	80.6	82.8	69.8	86.5	81.9	85.4	76.3	48.8
Net H ₂ O	-24.4	-26.2	-33.2	-10.8	-32.6	-27.2	-22.1	-24.7	15.2
Net Ash	-0.3	-0.9	0.4	0.8	-1.9	1.9	-0.5	-1.5	1.0
Unconverted Coal (MAF)	28.9	14.6	17.4	18.4	26.1	17.1	18.6	14.3	68.3
<u>Yields - Wt% MAF Coal</u>									
Net Gas	35.5	40.5	41.3	35.0	43.3	41.0	43.4	38.1	24.4
Net Liquid	62.7	65.8	66.4	60.8	60.9	63.2	58.7	67.9	33.4
Unconverted	14.6	7.3	8.7	9.2	13.1	8.5	9.4	7.1	34.1
Net H ₂ O + Ash	-12.8	-13.6	-16.4	-5.0	-17.3	-12.7	-11.5	-13.1	8.1
<u>Total Liquid Composition (Wt%)</u>									
Light Oil	7.7	8.7	22.5	18.9	19.8	13.8	5.9	19.4	16.1
Solvent	63.0	70.4	54.5	58.9	57.2	65.2	54.1	60.1	44.4
Vacuum Btms.	29.3	20.9	23.0	22.2	23.0	21.0	40.0	20.5	39.5
<u>Net Liquid (Wt% MAF Coal)</u>									
Net Light Oil	20.3	23.1	6.5	49.2	0.0	36.1	0.0	51.9	37.5
Net SRL	42.4	42.7	59.9	11.6	60.9	27.1	58.7	16.0	-4.1
<u>Solvent Recovery</u>									
Wt%	78.5	89.4	70.1	74.8	70.5	83.9	66.5	79.1	48.7
Wt% (100% Rec. Basis)	82.6	93.5	72.7	76.8	74.6	85.9	69.7	80.5	51.9

TABLE 5 - PART 2: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	<u>CREOSOTE OILS</u>								
	447	491	463	479	464	475	462	474	448
<u>Test Conditions</u>									
Time	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	755	750	750	750	755	755	755	750	770
Max. Press, psia	3310	3040	3080	3280	3140	3160	3220	3220	3080
Gas Charged	CO/H ₂								
Solvent	UNDRCO-72(2)	UNDRCO-73	UNDHRCO-73	UNDHRCO-72	UNDCCO-73	UNDHCCO-73	UNDRCO-72(3)		
Fraction	Standard	Standard	Standard	Standard	Standard	Standard			
Lignite	72-1(G)	72-1(M)	72-1(K)	72-1(K)	72-1(H)	72-1(K)	72-1X, 2(B)		
<u>Analytical Data</u>									
1. <u>Gas Analysis</u>									
H ₂ , Mol%	41.9	41.3	44.2	43.5	48.0	42.8	42.1	44.4	37.0
CH ₄ ,	1.7	2.3	1.3	1.9	3.9	1.8	1.7	1.7	1.9
CO	19.4	19.7	19.3	21.1	15.4	21.8	19.9	23.4	40.7
C ₂ H ₆	0.5	0.9	0.6	0.8	0.7	0.7	0.8	0.7	0.6
CO ₂	36.1	35.1	34.1	32.4	31.8	32.4	34.8	29.5	19.0
H ₂ S	0.4	0.7	0.5	0.3	0.2	0.5	0.7	0.3	0.8
Gas Specific Gravity @ RT	0.780	0.826	0.802	0.788	0.789	0.793	0.806	0.783	0.735
2. <u>Input Coal</u>									
Volatile Matter, Wt%	30.33	33.11	29.98	31.08	30.15	30.21	26.01	30.27	33.41
Ash, Wt%	6.77	7.78	7.52	7.27	7.56	6.74	6.58	6.75	2.71
Moisture, Wt%	30.96	23.94	30.09	28.82	29.69	29.91	30.15	29.78	26.09
Carbon, Wt% (1)	--	62.18	--	63.08	63.57	--	--	--	--
Hydrogen, Wt% (1)	--	4.22	--	4.43	3.99	--	--	--	--
Sulfur, Wt% (1)	1.21	1.02	0.96	1.10	0.95	1.10	--	1.10	--
3. <u>Input Solvent</u>									
Carbon, Wt%	--	90.00	--	90.68	91.20	90.31	--	91.09	--
Hydrogen, Wt%	--	6.03	--	6.68	6.75	6.17	--	6.63	--
Sulfur, Wt%	--	0.47	--	0.12	0.12	0.48	--	0.12	--
Specific Gravity 60/60	--	1.107	--	1.071	1.066	1.087	--	1.065	--
IR ratio	--	1.76	1.83	0.96	0.86	1.96	1.72	0.86	--
4. <u>Coal-Solvent Slurry</u>									
Brookfield Visc., cp (2)	--	--	--	--	--	--	--	--	--
5. <u>Cake</u>									
Ash, Wt%	29.32	23.61	26.77	27.11	18.53	31.38	35.15	29.87	5.89
Carbon, Wt%	--	66.28	--	62.91	70.86	57.68	--	59.79	--
Hydrogen, Wt%	--	4.22	--	4.19	5.24	3.72	--	3.96	--
Sulfur, Wt%	2.16	2.90	2.35	2.66	1.60	3.19	2.71	2.81	0.97
Pyridine Sol., Wt%	50.05	72.92	78.70	68.01	78.35	67.62	51.67	68.83	38.33

(1) Calculated on a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C

TABLE 5 - PART 2 CONT.: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	CREOSOTE OILS								
	447	491	463	479	464	475	462	474	448
6. <u>Filtrate</u>									
Ash, Wt%	0.00	0.00	0.02	0.00	0.04	0.00	0.03	0.00	0.00
Carbon, Wt%	---	89.71	---	89.27	88.68	89.33	---	90.51	---
Hydrogen, Wt%	---	6.56	---	6.74	6.81	6.57	---	6.89	---
Sulfur, Wt%	---	0.34	---	0.18	0.16	0.39	---	0.17	---
Specific Gravity 60/60	---	1.136	---	1.107	1.101	1.103	---	1.092	---
7. <u>Vacuum Btms.</u>									
Ash, Wt%	0.55	0.01	0.08	0.06	0.18	0.05	0.26	0.03	0.02
Carbon, Wt%	---	86.73	---	87.37	86.16	86.45	---	86.49	---
Hydrogen, Wt%	---	5.62	---	5.88	6.05	5.67	---	6.08	---
Sulfur, Wt%	0.24	0.25	0.26	0.21	0.13	0.23	0.32	0.23	---
8. <u>Residue</u>									
Ash, Wt%	15.49	24.32	1.86	21.82	6.76	13.96	10.00	17.76	4.72
Carbon, Wt%	---	63.60	---	66.62	82.76	74.54	---	73.54	---
Hydrogen, Wt%	---	4.76	---	5.19	6.40	5.70	--	5.53	---
Sulfur, Wt%	---	1.64	0.58	1.60	0.53	0.45	0.95	0.33	---
Pyridine Sol., Wt%	69.90	89.16	92.13	90.36	95.05	94.70	91.72	91.22	71.00
9. <u>Light Oil</u>									
Carbon, Wt%	---	65.91	---	89.06	86.97	81.06	---	87.18	---
Hydrogen, Wt%	---	9.10	---	8.69	9.19	8.62	---	8.51	---
Sulfur, Wt%	---	0.48	---	0.19	0.19	0.56	---	0.21	---
10. <u>Product Solvent</u>									
Carbon, Wt%	---	90.42	---	88.97	90.36	89.95	---	90.44	---
Hydrogen, Wt%	---	6.53	---	6.69	6.79	6.56	---	6.62	---
Sulfur, Wt%	---	0.36	---	0.17	0.16	0.41	---	0.20	---
Specific Gravity 60/60	---	1.095	---	1.086	1.086	1.093	---	1.088	---
IR ratio	1.37	0.77	0.78	0.70	0.59	0.80	0.93	0.73	1.73

TABLE 6 - PART 1: EVALUATION OF LIQUEFACTION SOLVENTS

CREOSOTE OILS AND PETROLEUM DERIVED SOLVENTS										
Run No.	503	507	508	485	497	514	473	478	486	513
<u>Test Conditions</u>										
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	755	755	750	752	754	765	750	750	750	750
Max. Press, psia	3160	3080	2870	3200	3180	3250	3200	3140	3260	3320
Gas Charged	CO/H ₂									
Solvent	USSLTCO-73	USSMHCO-73	GulfFS120-73		UNDF06-73		UNDF05-73			
Fraction	As Received	As Received	Standard	As Received	Standard	As Received	72-1(M)	As Received		
Lignite	72-1(Q)	72-1(R)	72-1(L)	72-1(P)	72-1(U)	72-1(K)	72-1(M)	72-1(U)		
<u>Material In, grams</u>										
Coal & H ₂ O	320.7	333.3	324.6	323.6	321.2	322.4	321.6	322.1	321.8	322.7
Solvent	399.3	400.6	399.3	400.2	399.3	399.0	400.4	399.3	398.8	399.3
Gas	143.2	138.9	139.6	145.2	147.7	149.5	143.0	138.2	142.6	155.1
Total	863.2	872.8	863.5	869.0	868.2	871.0	865.0	859.6	863.1	877.1
<u>Material Out, grams</u>										
Filtrate	439.3	444.6	402.9	438.2	437.8	432.0	347.0	393.7	376.9	348.4
Filter Cake	60.8	71.3	95.2	71.7	68.5	71.9	51.0	20.2	22.7	69.7
Residues	35.1	32.8	44.0	30.4	35.8	34.0	145.6	128.5	129.0	114.0
Gas	208.8	216.2	205.8	226.0	217.0	228.6	215.5	218.0	216.9	228.2
H ₂ O	72.4	75.2	68.4	75.1	74.9	77.1	76.9	76.3	81.8	84.1
Light Oil	8.0	3.9	3.8	5.6	6.6	2.5	16.7	16.6	5.8	4.6
Total	823.8	844.0	820.1	847.0	840.6	846.1	854.6	853.3	833.1	849.0
% Recovery	95.4	96.7	95.0	97.5	96.8	97.1	98.6	99.3	96.5	96.8
<u>100% Recovery Basis</u>										
<u>Products, grams</u>										
Light Oil	182.1	11.8	10.5	15.4	21.3	21.8	57.2	69.1	19.0	17.8
Solvent	247.2	367.3	347.3	406.5	397.9	285.9	397.8	386.6	432.2	246.3
Vac. Btms	101.5	152.6	174.0	99.1	106.8	205.5	68.7	65.6	51.0	181.0
Total Liquid	530.8	531.7	531.8	521.0	526.0	513.2	523.7	521.3	502.2	445.1
Net Liquid	131.5	131.1	132.5	120.8	126.7	114.2	123.3	122.0	103.4	45.8
Net Gas	75.0	84.7	77.0	86.6	76.5	85.9	75.6	81.3	82.2	80.6
Net H ₂ O	-23.8	-29.8	-27.7	-22.9	-22.2	-20.2	-21.9	-22.8	-14.8	-12.8
Net Ash	0.3	-3.0	-2.7	-1.2	-0.5	0.2	-1.1	-1.1	-3.1	-1.9
Unconverted Coal (MAF)	16.8	17.4	20.3	16.6	19.5	19.7	24.3	20.1	31.3	88.1
<u>Yields - Wt% MAF Coal</u>										
Net Gas	37.5	42.2	38.6	43.3	38.2	43.0	37.7	40.8	41.3	40.4
Net Liquid	65.8	65.5	66.4	60.4	63.4	57.2	61.7	61.1	52.0	22.9
Unconverted	8.4	8.7	10.2	8.3	9.8	9.8	12.1	10.1	15.7	44.1
Net H ₂ O + Ash	-11.7	-16.4	-15.2	-12.0	-11.4	-10.0	-11.5	-12.0	-9.0	-7.4
<u>Total Liquid Composition (Wt%)</u>										
Light Oil	34.3	2.2	2.0	3.0	4.0	4.3	10.9	13.2	3.8	4.0
Solvent	46.6	69.1	65.3	78.0	75.7	55.7	76.0	74.2	86.1	55.3
Vacuum Btms	19.1	28.7	32.7	19.0	20.3	40.0	13.1	12.6	10.1	40.7
<u>Net Liquid (Wt% MAF Coal)</u>										
Net Light Oil	15.0	5.9	5.3	7.7	10.6	9.5	28.6	34.6	9.5	7.3
Net SRL	50.8	59.6	61.1	52.7	52.8	47.7	33.1	26.5	42.5	15.6
<u>% Recovery</u>										
% (100% Rec. Basis)	59.1	88.7	82.6	99.0	96.5	69.6	98.0	96.1	104.6	61.7
Wt% (100% Rec. Basis)	61.9	91.7	87.0	101.6	99.7	71.6	99.4	96.8	108.4	

TABLE 6 - PART 2: EVALUATION OF LIQUEFACTION SOLVENTS

CREOSOTE OILS AND PETROLEUM DERIVED SOLVENTS

Run No.	503	507	508	485	497	514	473	478	486	513
<u>Test Conditions</u>										
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	755	755	750	750	755	765	750	750	750	750
Max. Press, psia	3160	3080	2870	3200	3180	3250	3200	3140	3260	3320
Gas Charged	CO/H ₂	CO/H ₂	1:1CO/H ₂	1:1CO/H ₂			CO/H ₂	CO/H ₂	CO/H ₂	CO/H ₂
Solvent	USS Lt Crst	USS MH CRST	UNDF120-73	Gulf FS-120	UNDF06-73	UNDF05-73	UNDF06-73	UNDF05-73	UNDF05-73	UNDF05-73
Fraction	As Received	As Received	Standard	As Received	Standard	As Received	Standard	Standard	As Received	As Received
Lignite	72-1(Q)	72-1(R)	72-1(L)	72-1(P)	72-1(U)	72-1(K)	72-1(M)	72-1(M)	72-1(U)	72-1(U)
<u>Analytical Data</u>										
1. Gas Analysis Mol%										
H ₂	42.6	42.2	40.1	42.8	38.7	38.6	45.5	43.0	44.0	43.1
CH ₄	2.0	1.9	1.8	3.4	3.6	3.4	1.9	2.4	2.0	2.1
CO	24.3	21.0	20.2	22.3	20.8	20.3	24.7	23.4	25.8	23.9
C ₂ H ₆	0.7	1.1	1.0	1.2	1.2	1.3	0.8	1.1	0.8	0.9
CO ₂	29.7	32.9	36.1	29.8	35.1	35.9	26.7	29.6	27.0	29.5
H ₂ S	0.7	0.9	0.8	0.5	0.6	0.5	0.4	0.5	0.4	0.5
Gas Spec. Gravity @RT	0.780	0.823	0.824	0.790	0.792	0.801	0.767	0.769	0.760	0.784
2. Input Coal										
Volatile Matter, Wt%	31.13	29.61	30.55	31.52	31.06	31.60	30.35	30.90	32.91	31.67
Ash, Wt%	6.75	7.57	7.81	7.78	6.99	7.55	6.77	7.23	7.73	7.57
Moisture, Wt%	29.67	32.29	30.14	26.06	29.05	27.24	29.60	29.24	24.39	27.07
Carbon, Wt% (1)	62.65	64.36	64.36	62.25	61.53	63.14	---	63.08	62.18	63.14
Hydrogen, Wt% (1)	3.98	3.77	3.77	4.09	4.18	3.32	---	4.43	4.22	3.32
Sulfur, Wt% (1)	1.35	1.24	1.24	1.14	1.13	1.14	1.10	1.10	1.02	1.14
3. Input Solvent										
Carbon, Wt%	89.85	91.78	92.03	88.93	88.99	89.57	86.22	86.22	87.39	85.26
Hydrogen, Wt%	6.24	5.37	5.50	8.37	8.35	7.81	10.37	10.37	11.69	11.03
Sulfur, Wt%	0.82	0.94	0.94	2.02	2.26	2.27	2.77	2.77	1.14	1.28
Specific Grav. 60/60	1.062	1.204	1.204	1.031	1.034	1.071	0.948	0.950	0.917	0.950
IR ratio	1.89	2.14	2.56	0.23	0.21	0.23	0.11	0.10	0.06	0.08
4. Coal-Solvent Slurry										
Brookfield Visc., cp(2)	--	--	--	--	--	--	--	--	--	--
5. Cake										
Ash, Wt%	27.06	27.62	20.53	27.16	26.48	28.71	1.92	7.92	5.73	3.87
Carbon, Wt%	62.39	63.25	67.92	67.66	63.49	62.90	84.20	78.38	81.43	84.11
Hydrogen, Wt%	4.05	3.80	4.21	3.32	4.66	4.42	8.19	6.00	6.35	8.15
Sulfur, Wt%	2.92	2.55	1.81	3.13	3.01	3.14	2.00	1.87	1.16	1.32
Pyridine Sol., Wt%	69.56	72.54	82.42	74.48	68.94	68.86	98.59	85.69	87.56	7.14

(1) Calculated on a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C

TABLE 6 - PART 2 CONT.: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	CREOSOTE OILS AND PETROLEUM DERIVED SOLVENTS									
	503	507	508	485	497	514	473	478	486	513
6. <u>Filtrate</u>										
Ash, Wt%	0.02	0.00	0.00	0.00	0.01	0.04	0.01	0.00	0.00	0.03
Carbon, Wt%	88.24	89.46	89.20	89.22	88.24	88.44	86.34	85.89	87.19	86.52
Hydrogen, Wt%	6.55	6.15	5.88	8.24	7.98	7.31	10.15	9.99	11.66	10.18
Sulfur, Wt%	0.49	0.63	0.62	1.61	1.81	1.79	2.29	2.05	1.00	1.04
Specific Grav.60/60	1.087	1.160	1.160	1.069	1.066	1.119	0.969	0.975	0.929	0.997
7. <u>Vacuum Btms</u>										
Ash, Wt%	0.13	0.07	0.10	0.11	0.08	0.11	0.06	0.04	1.32	0.08
Carbon, Wt%	87.56	89.02	87.77	86.76	86.82	89.27	85.90	88.33	86.06	87.63
Hydrogen, Wt%	5.66	4.85	4.97	5.86	5.90	5.81	6.64	6.62	7.45	8.74
Sulfur, Wt%	0.28	0.38	0.40	0.41	0.42	0.96	0.65	0.45	0.51	1.34
8. <u>Residue</u>										
Ash, Wt%	19.84	11.75	3.71	10.52	12.42	10.53	21.66	20.95	16.05	19.76
Carbon, Wt%	70.06	78.91	86.24	78.58	75.30	82.81	65.39	68.36	71.87	68.55
Hydrogen, Wt%	5.18	5.28	5.88	6.97	6.65	7.50	5.51	5.40	5.83	5.46
Sulfur, Wt%	1.95	1.24	0.89	1.75	2.45	2.16	2.79	2.63	1.80	2.08
Pyridine Sol, Wt%	93.31	99.17	97.27	90.24	91.63	94.29	76.65	78.76	71.19	73.22
9. <u>Light Oil</u>										
Carbon, Wt%	89.68	82.45	82.58	82.04	82.75	80.66	83.97	78.75	78.20	76.54
Hydrogen, Wt%	7.15	9.87	9.57	11.46	11.58	11.68	11.99	11.91	11.14	11.52
Sulfur, Wt%	0.68	0.59	0.53	0.33	0.34	0.35	0.96	0.45	0.10	0.09
10. <u>Product Solvent</u>										
Carbon, Wt%	89.51	91.34	91.18	88.88	88.32	88.59	86.25	86.43	87.19	86.51
Hydrogen, Wt%	6.62	6.05	6.09	8.47	8.29	8.16	10.39	10.19	11.57	11.50
Sulfur, Wt%	0.46	0.67	0.68	1.84	2.07	2.00	2.62	2.60	1.01	1.08
Specific Grav.60/60	1.085	1.155	1.155	1.033	1.034	1.045	0.951	0.959	0.917	0.925
IR ratio	0.75	0.98	1.03	0.23	0.21	0.21	0.11	0.10	0.06	0.06

TABLE 7 - PART 1: EVALUATION OF LIQUEFACTION SOLVENTS

PETROLEUM DERIVED SOLVENTS									
Run No.	519	495	499	496	509	494	501	500	506
<u>Test Conditions</u>									
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp, °F	755	750	750	755	755	755	755	755	750
Max. Press, psia	3430	3150	3200	3130	3140	3190	3220	3070	3210
Gas Charged	CO/H ₂								
Solvent	Exxon HAN-73	Exxon ATS-2-73	Exxon AC-73						
Fraction	As Received	Standard	As Received	Standard	As Received				
Lignite	72-1(W)	72-1(O)	72-1(P)	72-1(O)	72-1(R)	72-1(O)	72-1(P)	72-1(Q)	
<u>Material In, grams</u>									
Coal & H ₂ O	324.2	322.6	321.8	320.3	322.1	322.0	321.2	318.6	319.9
Solvent	400.3	400.2	400.2	396.5	396.3	399.2	399.9	396.8	395.7
Gas	151.1	142.2	138.7	146.1	140.3	141.6	143.2	139.3	140.7
Total	875.6	865.0	860.7	862.9	858.7	862.8	864.3	854.7	856.3
<u>Material Out, grams</u>									
Filtrate	384.7	426.3	418.6	405.7	391.4	420.9	414.7	413.8	381.4
Filter Cake	58.2	75.9	70.9	76.9	100.9	78.7	80.0	75.9	89.5
Residues	77.9	26.0	37.9	35.5	28.5	46.3	32.3	36.2	34.9
Gas	226.1	212.2	215.4	217.1	220.8	233.4	223.6	213.2	222.0
H ₂ O	80.4	75.3	75.5	75.7	69.4	69.3	68.4	75.4	77.0
Light Oil	20.7	12.5	8.0	16.2	12.3	2.8	3.9	7.1	10.0
Total	848.0	828.2	826.3	827.1	823.3	851.4	822.9	821.6	814.8
% Recovery	96.8	95.7	96.0	95.8	95.9	98.7	95.2	96.1	95.2
<u>100% Recovery Basis</u>									
<u>Products, grams</u>									
Light Oil	274.4	52.7	51.6	59.1	49.1	12.3	10.7	14.4	16.9
Solvent	149.7	358.1	352.6	256.9	260.5	392.8	360.9	219.3	209.1
Vac. Btms	84.9	108.7	109.3	197.8	201.1	107.5	143.2	274.9	268.5
Total Liquid	509.0	519.5	513.5	513.8	510.7	512.6	514.8	508.6	494.5
Net Liquids	108.7	119.3	113.3	117.3	114.4	113.4	114.9	111.8	98.8
Net Gas	82.3	79.5	85.7	80.5	89.9	94.9	91.7	82.6	92.5
Net H ₂ O	-16.8	-21.1	-21.1	-20.1	-26.5	-28.1	-27.5	-20.5	-17.9
Net Ash	-1.2	0.3	-0.1	1.9	-1.3	0.3	-0.7	3.2	2.3
Unconverted Coal (MAF)	27.2	22.6	22.4	18.3	20.2	20.9	21.6	21.6	21.7
<u>Yields - Wt% MAF Coal</u>									
Net Gas	41.1	39.7	42.8	40.4	45.4	47.2	45.8	41.5	46.8
Net Liquid	54.3	59.4	56.6	59.5	58.4	56.5	57.4	56.3	50.1
Unconverted	13.6	11.3	11.2	9.2	10.2	10.4	10.8	10.9	11.0
Net H ₂ O + Ash	-9.0	-10.4	-10.6	-9.1	-14.0	-14.1	-14.0	-8.7	-7.9
<u>Total Liquid Composition (Wt%)</u>									
Light Oil	53.9	10.1	10.0	11.7	9.8	2.4	2.1	2.8	3.4
Solvent	29.4	69.0	68.7	49.9	50.9	76.6	70.1	43.1	42.3
Vacuum Btms.	16.7	20.9	21.3	38.4	39.3	21.0	27.8	54.1	54.3
<u>Net Liquid (Wt% MAF Coal)</u>									
Net Light Oil	11.9	26.3	25.8	29.7	24.8	6.1	5.3	7.3	8.6
Net SRL	42.4	33.1	30.8	29.8	33.6	50.4	52.1	49.0	41.5
<u>Solvent Recovery</u>									
Wt%	36.2	85.6	84.6	62.1	63.0	97.1	85.9	53.1	50.3
Wt% (100% Rec. Basis)	37.4	89.4	88.1	64.8	65.7	98.4	90.2	54.9	52.8

TABLE 7 - PART 2: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	PETROLEUM DERIVED SOLVENTS									
	519	495	499	496	509	494	501	500	506	
<u>Test Conditions</u>										
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	
Avg. Temp, °F	755	750	750	755	755	755	755	755	750	
Max. Press, psia	3430	3150	3200	3130	3140	3190	3220	3070	3210	
Gas Charged	1:1CO/H ₂		CO/H ₂		CO/H ₂		CO/H ₂		CO/H ₂	
Solvent	Exxon HAN-73		Exxon ATS2-73		Exxon AC-73		Exxon AC-73		Exxon AC-73	
Fraction	As Received	Standard	As Received	As Received	Standard	As Received	As Received	As Received	As Received	
Lignite	72-1(W)	72-1(O)	72-1(P)	72-1(O)	72-1(R)	72-1(O)	72-1(P)	72-1(Q)	72-1(Q)	
<u>Analytical Data</u>										
1. Gas Analysis, Mol%	45.4	42.6	41.3	38.0	40.2	42.2	41.8	38.1	39.3	
H ₂	1.4	2.8	2.5	4.1	3.7	3.8	4.2	4.4	4.5	
CH ₄	23.6	22.6	22.6	22.9	22.0	22.0	22.5	24.0	23.1	
CO	0.7	1.0	1.0	1.3	1.5	1.3	1.5	1.6	1.6	
C ₂ H ₆	28.4	30.4	32.2	33.2	32.0	30.2	29.5	31.4	30.9	
CO ₂	0.5	0.6	0.4	0.5	0.6	0.5	0.5	0.5	0.6	
H ₂ S	0.789	0.778	0.783	0.812	0.813	0.823	0.787	0.801	0.795	
Gas Specific Gravity @RT										
2. Input Coal	30.35	31.86	31.02	31.70	30.54	32.35	31.26	30.83	31.67	
Volatile Matter, Wt%	7.57	7.13	6.98	7.09	7.81	7.24	7.03	6.68	7.45	
Ash, Wt%	29.63	29.80	29.17	30.15	30.17	28.72	28.63	30.35	28.75	
Moisture, Wt%	62.99	62.77	61.53	62.77	64.36	62.77	61.53	61.16	62.65	
Carbon, Wt%	3.91	4.21	4.18	4.21	3.77	4.21	4.18	3.98	3.98	
Hydrogen, Wt%	1.20	1.09	1.13	1.09	1.24	1.09	0.90	1.35	1.35	
Sulfur, Wt%										
3. Input Solvent	89.06	73.62	89.72	90.37	90.85	89.33	88.38	88.67	89.10	
Carbon, Wt%	10.10	7.53	8.50	7.49	7.45	8.43	8.35	8.05	8.14	
Hydrogen, Wt%	0.25	1.28	1.37	1.28	1.23	1.92	2.05	2.05	2.18	
Sulfur, Wt%	0.937	1.020	1.020	1.084	1.084	1.038	1.040	1.065	1.066	
Specific Grav. 60/60	0.21	0.30	0.32	0.34	0.35	0.17	0.18	0.18	0.18	
IR ratio										
4. Coal-Solvent Slurry	---	---	---	---	---	---	---	---	---	
Brookfield Visc., cp (2)	---	---	---	---	---	---	---	---	---	
5. Cake	20.44	26.36	24.73	27.51	21.13	25.06	23.64	27.25	24.03	
Ash, Wt%	66.65	62.18	65.71	62.82	68.95	63.91	64.19	63.97	67.31	
Carbon, Wt%	5.07	4.44	4.68	4.25	5.16	5.08	4.67	4.47	4.90	
Hydrogen, Wt%	1.68	2.75	2.50	2.80	2.21	2.70	2.69	3.13	3.12	
Sulfur, Wt%	70.57	65.34	65.97	74.55	80.49	73.23	71.72	69.17	75.95	
Pyridine Sol., Wt%										

(1) Calculated at a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C

TABLE 7 - PART 2 CONT.: EVALUATION OF LIQUEFACTION SOLVENTS

Run No.	PETROLEUM DERIVED SOLVENTS								
	519	495	499	496	509	494	501	500	506
6. <u>Filtrate</u>									
Ash, Wt%	0.03	0.00	0.07	0.05	0.00	0.07	0.00	0.04	0.03
Carbon, Wt%	86.86	87.99	88.17	90.35	89.14	88.47	87.84	87.65	88.54
Hydrogen, Wt%	9.37	7.90	7.89	7.46	7.22	7.83	7.74	7.60	5.61
Sulfur, Wt%	0.23	1.22	1.15	1.04	1.05	1.58	1.60	1.65	1.72
Specific Gravity 60/60	0.966	1.060	1.059	1.101	1.100	1.075	1.060	1.135	1.157
7. <u>Vacuum Btms</u>									
Ash, Wt%	0.09	0.13	0.29	0.04	0.06	0.15	0.00	0.07	0.08
Carbon, Wt%	86.93	86.93	87.10	88.41	90.10	86.56	87.03	89.52	88.86
Hydrogen, Wt%	7.01	5.89	5.85	5.87	5.79	6.01	5.74	6.50	6.37
Sulfur, Wt%	0.24	0.33	0.38	0.49	0.57	0.47	0.46	1.08	1.18
8. <u>Residue</u>									
Ash, Wt%	13.10	10.99	15.04	9.03	6.64	8.94	5.06	13.97	15.17
Carbon, Wt%	73.87	67.04	74.57	74.62	83.48	79.85	82.91	71.93	75.12
Hydrogen, Wt%	6.98	6.50	6.69	6.71	6.87	6.78	7.26	5.68	6.41
Sulfur, Wt%	1.19	1.44	1.75	1.69	1.43	1.98	1.89	2.45	2.53
Pyridine Sol, Wt%	81.24	93.67	93.13	94.72	95.20	94.78	98.06	90.18	92.06
9. <u>Light Oil</u>									
Carbon, Wt%	83.47	84.15	85.27	82.23	86.04	79.64	78.96	75.45	77.06
Hydrogen, Wt%	10.73	9.85	10.38	9.36	10.01	11.44	12.50	11.89	12.14
Sulfur, Wt%	0.24	0.34	0.31	0.43	0.36	0.34	0.45	0.43	0.48
10. <u>Product Solvent</u>									
Carbon, Wt%	88.86	76.51	89.78	89.10	88.93	88.72	87.27	88.28	88.54
Hydrogen, Wt%	9.33	7.57	8.48	7.97	8.06	8.34	8.27	8.25	8.45
Sulfur, Wt%	0.30	1.40	1.50	1.40	1.42	1.77	1.84	1.74	1.87
Specific Gravity 60/60	0.972	1.024	1.024	1.030	1.057	1.040	1.040	1.043	1.044
IR ratio	0.20	0.27	0.27	0.27	0.29	0.18	0.20	0.19	0.18

TABLE 8 - PART 1: SOLVENT RECYCLE SERIES - FIRST PASS TESTS

MATERIAL BALANCE AND YIELD DATA										
Run No.	527	528	533	536	537	546	547	548	557	560
<u>Test Conditions</u>										
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	755	750	755	750	750	750	750	750	752	752
Max. Press, psia	3190	3180	3190	3160	3160	3150	3210	3170	3140	3180
Gas Charged	CO/H ₂									
Starting Solvent	UNDFS120-74									
Lignite	73-2(C)			73-2(D)			73-2(F)			
<u>Material In, grams</u>										
Coal & H ₂ O	322.6	321.5	319.4	319.1	318.8	303.9	319.1	319.7	322.0	321.7
Solvent	399.0	397.6	400.2	399.9	400.0	400.4	400.2	400.5	400.5	400.2
Gas	161.5	153.3	152.8	151.1	148.6	152.7	152.7	159.5	150.7	151.3
Total	883.1	872.4	872.4	870.1	867.4	857.0	872.0	879.7	873.2	873.2
<u>Material Out, grams</u>										
Filtrate	425.0	403.5	447.4	431.3	448.0	439.8	444.1	434.6	454.0	453.1
Filter Cake	116.9	131.0	83.5	91.2	80.3	85.7	83.0	83.8	82.4	87.2
Residues	17.7	32.1	20.7	30.3	26.6	19.2	23.8	39.9	26.5	25.4
Gas	226.5	230.9	219.7	219.3	220.5	213.6	216.6	217.8	214.4	217.0
H ₂ O	71.2	72.4	74.2	78.8	77.3	78.2	80.7	76.4	77.9	78.0
Light Oil	2.5	1.6	1.6	0.6	0.8	0.6	1.0	1.6	0.6	1.7
Total	859.8	871.5	847.1	851.5	853.5	837.1	849.2	854.1	855.8	862.3
% Recovery	97.4	99.9	97.1	97.9	98.4	97.7	97.4	97.1	98.0	98.8
<u>Yields 100% Recovery Basis</u>										
<u>Product, grams</u>										
Light Oil	13.0	11.1	15.6	12.8	15.7	11.1	11.9	11.4	11.3	14.3
Solvent	407.0	397.9	396.3	403.7	403.7	406.8	409.8	409.5	393.7	405.9
Vac. Btms	106.4	109.6	115.9	108.9	105.1	103.2	105.3	114.8	126.1	110.6
Total liquid	526.4	518.6	527.8	525.4	524.5	521.1	527.0	535.7	531.1	530.8
Net liquids	127.4	121.0	127.6	125.5	124.5	120.7	126.8	135.2	130.6	130.6
Net Gas	71.1	77.9	73.5	72.9	75.5	66.0	69.7	64.8	68.1	68.4
Net H ₂ O	-26.4	-26.6	-23.5	-19.3	-21.2	-19.8	-17.0	-21.5	-20.4	-20.8
Net Ash	-0.5	-1.4	0.2	-1.0	0.0	0.0	-0.3	-0.2	-1.0	-0.7
Unconverted Coal (MAF)	27.5	27.9	22.9	21.9	21.3	19.6	20.9	21.8	23.0	22.7
<u>Yields Wt% MAF Coal</u>										
Net Gas	35.6	39.1	36.6	36.5	37.7	35.4	34.8	32.4	34.0	34.2
Net Liquid	64.1	61.0	63.6	62.7	62.2	64.7	63.3	67.4	65.1	65.2
Unconverted	13.8	14.0	11.4	10.9	10.7	10.5	10.5	10.9	11.5	11.3
Net H ₂ O + Ash	-13.5	-14.1	-11.6	-10.1	-10.6	-10.6	-8.6	-10.7	-10.6	-10.7
<u>Total Liquid Composition (Wt%)</u>										
Light Oil	2.5	2.2	2.9	2.4	3.0	2.1	2.3	2.1	2.1	2.7
Solvent	77.3	76.7	75.1	76.9	77.0	78.1	77.7	76.4	74.1	76.5
Vacuum Btms.	20.2	21.1	22.0	20.7	20.0	19.8	20.0	21.5	23.8	20.8
<u>Net Liquid (Wt% MAF Coal)</u>										
Net Light Oil	6.5	5.6	7.8	6.4	7.8	5.9	5.9	5.7	5.6	7.1
Net SRL	57.6	55.4	55.8	56.3	54.4	58.8	57.4	61.7	59.5	58.1
<u>Solvent Recovery</u>										
%	99.3	100.0	96.2	98.8	99.3	99.2	99.7	99.3	96.3	100.2
(100% Rec. Basis)	102.0	100.1	99.1	100.9	100.9	101.6	102.4	102.3	98.3	1.4

TABLE 8 - PART 2 : SOLVENT RECYCLE SERIES - FIRST PASS TESTS

<u>Run No.</u>	<u>ANALYTICAL DATA</u>										
	527	528	533	536	537	546	547	548	557	560	
<u>Test Conditions</u>											
Time	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	
Avg. Temp., °F	755	750	755	750	750	750	750	750	752	752	
Max. Press., psia	3190	3180	3190	3160	3160	3150	3210	3170	3140	3180	
Gas Charged	CO/H ₂										
Starting Solvent	UNDFS120-74										
Lignite	73-2(C) → ← 73-2(D) → ← 73-2(F) →										
<u>Analytical Data</u>											
1. <u>Gas Analysis, Mol%</u>	42.0	45.0	48.5	42.3	44.0	45.7	45.9	45.4	44.0	58.0	
H ₂	1.2	1.4	1.4	0.9	1.5	1.4	1.4	1.5	1.5	1.4	
CH ₄	25.8	24.1	23.9	24.7	23.9	25.3	26.2	24.6	26.9	33.0	
CO	0.6	0.9	0.6	0.6	0.6	0.5	0.4	0.7	0.5	0.1	
C ₂ H ₆	30.0	28.1	25.3	31.1	29.8	26.8	25.7	27.5	26.7	6.9	
CO ₂	0.4	0.5	0.3	0.4	0.2	0.3	0.4	0.3	0.4	0.6	
H ₂ S	0.759	0.763	0.752	0.753	0.754	0.743	0.750	0.756	0.762	0.757	
Gas Specific Gravity @RT											
2. <u>Input Coal</u>	30.64	30.57	30.81	31.19	30.60	31.18	31.88	31.55	34.67	34.59	
Volatile Matter, Wt%	7.40	7.39	6.29	6.37	6.24	6.30	6.43	6.37	7.06	7.07	
Ash, Wt%	29.36	29.53	28.33	27.44	27.85	27.45	25.83	26.60	27.58	27.74	
Moisture, Wt%	63.80	63.80	65.38	65.38	64.26	66.04	67.52	66.82	62.48	62.48	
Carbon, Wt% (1)	4.42	4.42	4.34	4.34	4.45	4.13	4.23	4.18	4.36	4.36	
Hydrogen, Wt% (1)	0.80	0.80	0.71	0.71	0.69	0.63	0.63	0.63	0.94	0.94	
Sulfur, Wt% (1)											
3. <u>Input Solvent</u>	--	--	--	--	0.01	0.02	0.01	--	--	--	
Ash, Wt%	88.76	88.46	88.24	88.12	88.16	88.37	86.43	86.63	89.26	89.22	
Carbon, Wt%	8.11	8.03	8.01	8.37	8.18	8.28	8.48	8.23	8.09	7.85	
Hydrogen, Wt%	2.51	2.57	2.51	2.59	2.63	2.48	2.39	2.44	2.37	2.44	
Sulfur, Wt%	1.041	1.041	1.035	1.035	1.042	1.034	1.049	1.056	1.033	1.049	
Specific Grav. 60/60	--	--	--	33.0	33.0	25.4	22.4	33.2	51.0	32.4	
Brookfield Visc., cp (2)	0.20	0.20	0.21	0.20	0.21	0.21	0.19	0.21	0.21	0.21	
IR Ratio											
4. <u>Coal-Solvent Slurry</u>	Brookfield Visc., cp (2)	--	--	--	3500	3490	2580	3480	4170	4282	4998
5. <u>Cake</u>	Ash, Wt%	18.75	15.75	21.13	18.35	21.95	19.31	20.69	20.26	23.23	22.38
Carbon, Wt%	69.46	72.22	67.74	68.72	66.19	69.33	66.65	66.29	64.50	66.56	
Hydrogen, Wt%	5.25	5.84	5.15	5.48	4.88	5.42	5.23	5.20	4.74	4.77	
Sulfur, Wt%	2.04	2.01	1.86	1.77	1.89	1.87	1.70	1.73	2.21	2.23	
Pyridine Sol., Wt%	74.80	80.29	69.92	76.75	71.15	75.68	72.92	75.11	68.75	70.90	

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 8 - PART 2 CONT.: SOLVENT RECYCLE SERIES - FIRST PASS TESTS

Run No.	ANALYTICAL DATA									
	527	528	533	536	537	546	547	548	557	560
6. <u>Filtrate</u>										
Ash, Wt%	--	0.07	0.12	0.03	0.04	0.04	0.04	0.02	0.03	0.03
Carbon, Wt%	87.93	87.73	87.61	86.37	86.73	87.37	86.51	84.35	85.66	86.65
Hydrogen, Wt%	8.01	7.86	7.91	7.84	7.79	8.04	7.94	7.99	7.50	7.69
Sulfur, Wt%	2.04	2.07	2.06	1.95	2.01	2.07	2.04	1.98	1.93	1.81
Specific Grav. 60/60	1.085	1.076	1.070	1.071	1.073	1.073	1.080	1.090	1.080	1.087
Brookfield Visc., cp (2)	--	--	--	2120	1380	1150	1270	1900	3282	3890
Blackness, abs./g. @550 m μ	6.68	5.41	3.61	4.30	4.89	4.66	4.40	4.65	4.27	4.29
7. <u>Vacuum Btms</u>										
Ash, Wt%	0.17	0.22	0.21	0.17	0.18	0.15	0.18	0.14	0.27	0.23
Carbon, Wt%	85.26	85.89	85.64	84.00	85.58	85.36	84.39	84.43	84.40	85.09
Hydrogen, Wt%	6.20	6.32	6.13	5.86	6.17	6.25	6.41	5.94	6.13	6.04
Sulfur, Wt%	0.38	0.44	0.37	0.35	0.39	0.28	0.43	0.45	0.36	0.48
Melting Point, °F	321	309	314	305	315	292	328	322	306.5	264.2
8. <u>Residue</u>										
Ash, Wt%	6.08	7.07	4.10	3.57	3.14	5.02	5.57	4.27	5.78	5.78
Carbon, Wt%	82.69	80.42	83.70	83.88	85.01	82.48	83.37	81.03	81.91	82.50
Hydrogen, Wt%	7.25	7.03	7.10	7.62	7.72	7.98	7.85	7.62	7.31	7.31
Sulfur, Wt%	2.13	2.14	1.99	1.84	1.92	1.95	1.77	1.94	1.90	1.85
Pyridine Sol, Wt%	93.62	94.92	96.82	97.33	96.47	94.11	95.25	96.36	96.34	95.19
9. <u>Light Oil</u>										
Carbon, Wt%	--	--	--	79.82	80.74	78.74	79.98	79.06	82.95	82.86
Hydrogen, Wt%	--	--	--	10.90	11.56	11.62	10.90	10.83	9.56	10.45
Sulfur, Wt%	--	--	0.84	0.87	0.60	--	0.75	0.76	--	--
10. <u>Product Solvent</u>										
Ash, Wt%	--	--	--	--	0.06	0.02	0.02	--	--	--
Carbon, Wt%	87.61	88.67	87.09	87.91	88.23	87.63	86.26	87.53	87.83	88.40
Hydrogen, Wt%	8.18	8.23	7.82	8.34	8.26	8.29	8.53	8.78	8.24	7.91
Sulfur, Wt%	2.43	2.37	2.34	2.49	2.37	2.35	2.42	2.32	2.40	2.33
Specific Gravity 60/60	1.040	1.045	1.031	1.036	1.043	1.050	1.051	1.050	1.043	1.045
Brookfield Visc, cp (2)	--	--	--	37.4	37.0	24.0	23.8	35.2	37.0	52.0
IR Ratio	0.20	0.19	0.19	0.20	0.19	0.19	0.19	0.20	0.19	0.18

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 9 - PART 1: SOLVENT RECYCLE SERIES - SECOND AND THIRD PASS TESTS

MATERIAL BALANCE AND YIELD DATA													
Run No.	529	534	543	551	552	558	535	544	553	559			
<u>Test Conditions</u>													
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5			
Avg. Temp., °F	755	765	750	750	752	752	755	750	752	752			
Max. Press, psia	3200	3300	3170	3160	3130	3150	3210	3120	3200	3150			
Gas Charged	CO/H ₂												
Starting Solvent	UNDFS120-74												
Lignite	73-2(C)	73-2(D)			73-2(F)			73-2(D)					
	Second Pass				Third Pass				73-2(F)				
<u>Material In, grams</u>													
Coal & H ₂ O	322.7	319.2	319.7	318.8	319.0	322.1	319.3	319.7	319.4	320.9			
Solvent	399.1	399.3	400.4	400.4	400.4	400.7	400.0	400.4	400.7	400.1			
Gas	160.5	155.9	160.6	152.4	153.8	149.7	157.8	157.2	149.7	151.2			
Total	882.3	874.4	880.7	871.6	873.2	872.5	877.1	877.3	869.8	872.2			
<u>Material Out, grams</u>													
Filtrate	421.6	458.6	439.6	434.2	437.6	470.5	462.0	458.9	462.7	452.6			
Filter Cake	121.9	69.9	83.1	88.5	79.8	71.2	73.3	66.3	78.5	79.7			
Residues	12.6	19.2	28.2	31.5	35.1	26.3	27.4	28.1	20.2	29.3			
Gas	222.4	223.2	220.0	226.6	216.9	212.5	225.6	217.5	217.0	213.8			
H ₂ O	74.2	79.6	75.7	78.4	80.2	82.5	76.8	76.9	80.7	79.2			
Light Oil	1.3	1.3	1.2	0.7	1.3	0.2	1.4	1.1	0.3	1.1			
Total	854.0	851.8	847.8	859.9	850.9	863.2	866.5	848.8	859.4	855.7			
<u>% Recovery</u>	96.8	97.4	96.3	98.7	97.5	98.9	98.8	96.7	98.8	98.1			
<u>Yields 100% Recovery Basis</u>													
<u>Products, grams</u>													
Light Oil	10.0	13.2	11.0	10.5	11.4	10.9	14.1	13.5	8.7	12.7			
Solvent	405.6	406.9	410.3	406.5	407.5	412.9	403.6	406.8	406.3	405.3			
Vac. Btms	111.0	105.7	110.5	105.6	109.8	109.6	114.4	113.7	111.3	111.5			
Total liquid	526.6	525.8	531.8	522.6	528.7	533.4	532.1	534.0	526.3	529.5			
Net liquids	127.5	126.5	131.4	122.2	128.3	132.7	132.1	133.6	125.6	129.4			
Net Gas	69.3	73.2	67.9	77.3	68.7	65.1	70.6	67.6	69.9	66.7			
Net H ₂ O	-22.9	-18.1	-21.3	-20.3	-17.6	-16.5	-22.1	-20.3	-18.2	-19.1			
Net Ash	0.4	-1.2	-0.3	-0.2	-0.5	-1.2	-1.0	-1.6	-0.5	-0.7			
Unconverted Coal (MAF)	25.6	19.4	22.3	20.9	20.7	20.3	20.3	21.0	23.7	23.0			
<u>Yields Wt% MAF Coal</u>													
Net Gas	34.7	36.6	33.9	38.6	34.4	32.5	35.3	33.7	34.9	33.5			
Net Liquid	63.8	63.3	65.8	61.2	64.2	66.2	66.1	66.8	62.6	64.9			
Unconverted	12.8	9.7	11.1	10.4	10.4	10.1	10.1	10.5	11.8	11.5			
Net H ₂ O +Ash	-11.3	-9.6	-10.8	-10.2	-9.0	-8.8	-11.5	-11.0	-9.3	-9.9			
<u>Total Liquid Composition (Wt%)</u>													
Light Oil	1.9	2.5	2.1	2.0	2.1	2.0	2.6	2.5	1.7	2.4			
Solvent	77.0	77.4	77.1	77.8	77.1	77.4	75.9	76.2	77.2	76.5			
Vacuum Btms	21.1	20.1	20.8	20.2	20.8	20.6	21.5	21.3	21.2	21.1			
<u>Net Liquid (Wt% MAF Coal)</u>													
Net light Oil	5.0	6.6	5.5	5.3	5.7	5.4	7.0	6.7	4.3	6.4			
Net SRL	58.8	56.7	60.3	55.9	58.5	60.8	59.1	60.1	58.4	58.5			
<u>Solvent Recovery</u>													
Wt% (100% Rec. Basis)	98.4	99.3	98.7	100.2	99.2	102.0	99.7	98.3	101.2	99.4			
Wt% (100% Rec. Basis)	101.7	101.9	102.5	101.6	101.8	103.1	100.9	101.6	101.4	101.3			

TABLE 9 - PART 2: SOLVENT RECYCLE SERIES - SECOND AND THIRD PASS TESTS

ANALYTICAL DATA													
Run No.	529	534	543	551	552	558	535	544	553	559			
<u>Test Conditions</u>													
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5			
Avg. Temp. °F	755	765	750	750	752	752	755	750	752	752			
Max. Press., psia	3200	3300	3170	3160	3130	3150	3210	3120	3200	3150			
Gas Charged	CO/H ₂												
Starting Solvent	UNDFS120-74												
Lignite	73-2(C)	73-2(D)	73-2(F)	73-2(D)	73-2(F)	Third Pass							
Second Pass													
<u>Analytical Data</u>													
1. <u>Gas Analysis, Mol%</u>	42.8	43.2	44.6	42.6	46.1	44.9	43.6	44.9	46.5	52.9			
H ₂	1.3	1.7	1.3	1.5	1.7	1.4	1.4	1.5	1.3	1.4			
CH ₄	25.1	25.4	26.0	28.4	24.4	24.9	25.5	24.2	24.9	26.2			
CO	0.5	0.6	0.5	0.5	0.7	0.5	0.6	0.6	0.4	0.3			
C ₂ H ₆	29.9	28.8	27.3	26.6	26.8	27.9	28.5	28.6	26.7	18.8			
CO ₂	0.4	0.3	0.3	0.4	0.3	0.4	0.4	0.2	0.2	0.4			
H ₂ S	0.752	0.753	0.752	0.787	0.770	0.754	0.767	0.759	0.748	0.759			
Gas Specific Grav. @RT													
2. <u>Input Coal</u>	30.64	31.54	30.77	31.54	32.65	34.71	31.08	30.98	32.63	34.60			
Volatile Matter, Wt%	7.41	6.44	6.36	6.30	6.31	7.07	6.35	6.39	6.33	7.05			
Ash, Wt%	29.35	26.64	28.39	26.60	26.46	27.49	27.70	28.03	26.50	27.73			
Moisture, Wt%	63.80	65.38	65.19	63.54	63.66	62.48	65.38	65.52	63.66	62.48			
Carbon, Wt% (1)	4.42	4.34	4.08	3.82	3.83	4.36	4.34	4.10	3.83	4.36			
Hydrogen, Wt% (1)	0.80	0.71	0.63	0.63	0.63	0.94	0.71	0.63	0.63	0.94			
Sulfur, Wt% (1)													
3. <u>Input Solvent</u>	--	--	--	--	0.02	--	0.01	0.04	--	--			
Ash, Wt%	88.19	87.22	87.39	88.07	88.07	87.84	85.86	87.88	88.05	87.61			
Carbon, Wt%	8.20	8.12	8.40	8.36	8.00	8.48	8.00	8.43	8.35	8.22			
Hydrogen, Wt%	2.41	2.44	2.43	2.42	2.28	2.31	2.25	2.26	2.18	2.21			
Sulfur, Wt%	1.045	1.063	1.038	1.043	1.046	1.040	1.041	1.038	1.039	1.045			
Specific Gravity 60/60	--	--	--	30.2	37.8	43.2	40.0	31.6	42.0	31.0			
Brookfield Visc., cp (2)	0.20	0.20	0.20	0.21	0.20	0.19	0.21	0.19	0.20	--			
IR Ratio													
4. <u>Coal-Solvent Slurry</u>	--	--	4080	3570	3382	3807	3770	3890	5539	--			
Brookfield Visc., cp (2)													
5. <u>Cake</u>	18.28	24.31	20.99	19.43	20.96	26.20	23.56	23.54	22.01	24.01			
Ash, Wt%	72.30	62.83	67.10	67.00	67.01	61.60	63.93	64.27	63.22	63.88			
Carbon, Wt%	5.65	4.39	5.09	4.66	5.13	4.61	4.72	4.95	4.33	4.68			
Hydrogen, Wt%	2.06	1.82	1.85	1.70	1.74	2.30	1.84	1.79	1.70	2.25			
Sulfur, Wt%	77.26	67.65	72.13	76.33	74.09	66.13	69.10	65.14	65.05	67.53			
Pyridine Sol., Wt%													

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 9 - PART 2 CONT.: SOLVENT RECYCLE SERIES - SECOND AND THIRD PASS TESTS

Run No.	ANALYTICAL DATA									
	529	534	543	551	552	558	535	544	553	559
6. <u>Filtrate</u>										
Ash, Wt%	0.09	--	0.05	0.02	0.01	0.03	--	0.05	0.03	0.04
Carbon, Wt%	85.92	87.38	87.28	86.61	87.00	87.20	87.28	83.22	85.63	86.16
Hydrogen, Wt%	7.78	7.67	8.11	7.69	7.81	7.88	7.89	7.94	7.85	7.61
Sulfur, Wt%	1.97	1.90	1.92	1.94	1.42	1.79	1.79	1.85	1.81	1.74
Specific Grav. 60/60	1.075	1.074	1.076	1.073	1.073	1.080	1.075	1.076	1.080	1.078
Brookfield Visc. cp (2)	--	--	2250	1810	1674	2224	1850	1800	2357	3015
Blackness, abs./g. @550 m μ	4.21	4.32	4.57	4.48	--	4.41	4.58	5.04	--	4.80
7. <u>Vacuum Btms</u>										
Ash, Wt%	0.15	0.14	0.19	0.19	0.14	0.28	0.19	0.20	0.18	0.24
Carbon, Wt%	81.07	85.80	84.95	84.64	84.97	84.06	85.84	85.41	84.14	84.90
Hydrogen, Wt%	6.04	6.18	6.36	6.22	6.28	6.37	6.28	6.27	5.78	6.37
Sulfur, Wt%	0.38	0.34	0.40	0.33	0.36	0.35	0.31	0.38	0.33	0.37
Melting Point, °F	298.0	315.0	283.0	297.0	277.9	282.8	307.0	284.0	283.5	265.9
8. <u>Residue</u>										
Ash, Wt%	8.50	3.52	4.23	4.15	4.12	11.44	2.79	10.35	6.19	4.80
Carbon, Wt%	78.72	83.09	82.00	82.03	83.52	75.70	84.63	77.25	79.72	82.27
Hydrogen, Wt%	7.43	7.58	7.65	7.74	7.84	6.70	7.29	6.92	7.31	7.37
Sulfur, Wt%	2.27	1.81	1.72	1.87	1.87	2.03	1.75	1.90	1.78	1.66
Pyridine Sol., Wt%	92.92	97.88	95.46	96.34	96.62	94.39	98.61	90.38	92.49	96.45
9. <u>Light Oil</u>										
Carbon, Wt%	--	77.54	79.52	79.53	74.91	53.26	79.38	79.54	79.39	78.97
Hydrogen, Wt%	--	10.19	9.89	10.37	9.92	10.68	10.51	10.50	10.08	10.46
Sulfur, Wt%	0.61	0.74	0.45	1.0	0.86	--	0.51	--	--	0.66
10. <u>Product Solvent</u>										
Ash, Wt%	--	--	0.03	0.01	0.03	--	0.11	0.03	--	--
Carbon, Wt%	88.07	87.94	86.09	88.08	87.49	87.90	87.74	87.93	88.43	87.33
Hydrogen, Wt%	8.24	8.17	8.29	8.62	8.22	8.25	8.35	8.44	8.12	8.17
Sulfur, Wt%	2.29	2.35	2.32	2.34	2.22	--	2.24	2.35	2.18	2.24
Specific Grav. 60/60	1.042	1.041	1.038	1.036	1.046	--	1.041	1.043	1.042	1.045
Brookfield Visc. cp (2)	--	--	29.8	40.8	31.6	--	--	32.4	27.6	31.0
IR Ratio	0.19	0.21	0.20	0.19	0.17	--	0.20	0.19	0.19	0.20

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 10 - PART 1: SOLVENT RECYCLE SERIES - FOURTH AND FIFTH PASS TEST

MATERIAL BALANCE AND YIELD DATA

<u>Run No.</u>	545	554	565
<u>Test Conditions</u>			
Time, hrs.	0.5	0.5	0.5
Avg. Temp, °F	750	752	752
Max. Press, psia	3130	3150	3125
Gas Charged	CO/H ₂		
Starting Solvent	UNDFS120-74		
Lignite	73-2(D)	73-2(E)	73-2(F)
	Fourth Pass		Fifth Pass
<u>Material In, grams</u>			
Coal & H ₂ O	319.2	320.1	321.6
Solvent	400.3	400.5	399.8
Gas	154.0	150.2	144.7
Total	873.5	870.8	866.1
<u>Material Out, grams</u>			
Filtrate	463.5	448.3	467.4
Filter Cake	68.3	84.1	73.2
Residues	23.3	14.9	15.3
Gas	213.2	216.4	208.2
H ₂ O	77.1	80.1	80.3
Light Oil	1.6	1.1	1.5
Total	847.0	844.9	845.9
<u>% Recovery</u>	97.0	97.0	97.7
<u>Yields-100% Recovery Basis</u>			
<u>Products, grams</u>			
Light Oil	10.4	16.1	13.4
Solvent	399.4	397.8	407.6
Vac. Btms	124.4	111.4	107.7
Total Liquid	534.2	525.3	528.7
Net Liquids	133.9	124.9	128.9
Net Gas	65.9	72.9	68.4
Net H ₂ O	-20.4	-17.0	-17.5
Net Ash	-0.7	-1.2	-1.2
Unconverted Coal (MAF)	21.3	20.4	21.4
<u>Yields-Wt. % MAF Coal</u>			
Net Gas	32.9	36.5	34.2
Net Liquid	66.9	62.4	64.4
Unconverted	10.7	10.2	10.7
Net H ₂ O + Ash	-10.5	-9.1	-9.3
<u>Total Liquid Composition (Wt%)</u>			
Light Oil	1.9	3.2	2.5
Solvent	74.8	74.4	77.1
Vac. Btms.	23.3	22.4	20.4
<u>Net Liquid (Wt% MAF Coal)</u>			
Net Light Oil	5.2	8.0	6.7
Net SRL	61.7	54.4	57.7
<u>Solvent Recovery</u>			
Wt%	96.7	96.3	99.6
Wt% (100% Rec. Basis)	99.7	99.3	101.9

TABLE 10 - PART 2: SOLVENT RECYCLE SERIES - FOURTH AND FIFTH PASS TEST

ANALYTICAL DATA

<u>Run No.</u>	545	554	565
<u>Test Conditions</u>			
Time	0.5	0.5	0.5
Avg. Temp, °F	750	752	752
Max. Press, psia	3130	3150	3125
Gas Charged	CO/H ₂		
Starting Solvent	UNDFS120-74		
Lignite	73-2(D)	73-2(E)	73-2(F)
	Fourth Pass		Fifth Pass
<u>Analytical Data</u>			
1. <u>Gas Analysis, Mol %</u>			
H ₂	47.2	42.9	41.6
CH ₄	1.6	1.2	0.9
CO	22.5	27.6	20.6
C ₂ H ₆	0.6	0.4	0.5
CO ₂	27.8	27.5	35.9
H ₂ S	0.3	0.4	0.5
Gas Specific Gravity @ RT	0.750	0.755	0.748
2. <u>Input Coal</u>			
Volatile Matter, Wt. %	31.15	30.40	34.64
Ash, Wt. %	6.29	6.71	7.11
Moisture, Wt. %	27.52	28.66	27.64
Carbon, Wt. % (1)	65.98	67.15	62.48
Hydrogen, Wt. % (1)	4.13	4.47	4.36
Sulfur, Wt. % (1)	0.63	0.87	1.60
3. <u>Input Solvent</u>			
Ash, Wt. %	0.04	0.02	0.01
Carbon, Wt. %	87.85	88.22	87.91
Hydrogen, Wt. %	8.31	8.30	8.25
Sulfur, Wt. %	2.08	2.23	2.13
Specific Gravity 60/60	1.043	1.042	1.043
Brookfield Visc. cp (2)	33.4	38.0	39.0
IR Ratio	0.20	0.19	0.18
4. <u>Coal-Solvent Slurry</u>			
Brookfield Visc., cp (2)	4500	3575	5864
5. <u>Cake</u>			
Ash, Wt%	24.85	21.58	26.21
Carbon, Wt%	62.02	66.04	61.83
Hydrogen, Wt%	4.72	4.92	4.47
Sulfur, Wt%	1.84	2.06	2.28
Pyridine Sol., Wt%	63.63	72.11	64.09

(1) Calculated on a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 10 - PART 2 (CONT.): SOLVENT RECYCLE SERIES - FOURTH AND FIFTH PASS TESTS

<u>ANALYTICAL DATA</u>			
<u>Run No.</u>	545	554	565
<u>6. Filtrate</u>			
Ash, Wt%	0.04	0.05	0.04
Carbon, Wt%	86.57	87.16	85.63
Hydrogen, Wt%	7.98	7.96	7.74
Sulfur, Wt%	1.68	1.70	1.64
Specific Grav., 60/60	1.077	1.083	1.080
Brookfield Visc., cp (2)	1560	2585	4773
Blackness, abs./g. @550 m μ	5.47	4.77	4.67
<u>7. Vacuum Btms</u>			
Ash, Wt%	0.17	0.25	0.25
Carbon, Wt%	84.43	84.24	85.24
Hydrogen, Wt%	6.35	6.41	6.08
Sulfur, Wt%	0.31	0.31	0.33
Melting Point, °F	286.0	304.1	301.0
<u>8. Residue</u>			
Ash, Wt%	3.73	6.34	10.51
Carbon, Wt%	81.23	79.72	76.80
Hydrogen, Wt%	7.63	7.39	6.86
Sulfur, Wt%	1.81	1.70	1.93
Pyridine Sol., Wt%	96.90	97.76	92.17
<u>9. Light Oil</u>			
Carbon, Wt%	75.80	78.27	--
Hydrogen, Wt%	10.98	10.49	--
Sulfur, Wt%	0.74	0.65	0.43
<u>10. Product Solvent</u>			
Ash, Wt%	0.03	0.02	0.01
Carbon, Wt%	87.93	88.22	86.50
Hydrogen, Wt%	8.38	8.30	7.90
Sulfur, Wt%	2.23	2.20	2.05
Specific Grav. 60/60	1.039	1.033	1.045
Brookfield Visc., cp (2)	34.2	62.0	46.0
IR Ratio	0.18	0.20	0.20

(1) Calculated on a moisture-free basis

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 11 - PART 1: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 0-12 WEEKS

Run No.	MATERIAL BALANCE AND YIELD DATA									
	↔ BASE CASE ↔		↔ 6-WEEKS STORAGE ↔				↔ 12-WEEKS STORAGE ↔			
Test Conditions										
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	755	750	755	760	750	750	755	755	750
Max. Press., psia	3020	3110	3270	3230	3120	3210	3160	3180	3220	2960
Gas Charged	↔ CO/H ₂ ↔									
Solvent	↔ UNDCAO-73 ↔									
Lignite	↔ 73-2(A) ↔		↔ 73-2(B) ↔				↔ 73-2(B) ↔			
Material In, grams			H ₂ O Strg	Air Strg	N ₂ Strg	N ₂ Strg	Air Strg	N ₂ Strg	H ₂ O Strg	Air Strg
Coal & H ₂ O	322.0	323.1	315.9	323.3	323.5	323.0	323.4	323.7	317.3	323.4
Solvent	399.6	399.9	399.2	398.9	399.6	399.0	400.0	399.9	399.0	400.1
Gas	144.2	140.8	153.5	158.3	152.8	161.3	150.7	155.7	155.9	157.0
Total	865.8	863.8	868.6	880.5	875.9	883.3	874.1	879.3	873.2	880.5
Material Out, grams										
Filtrate	439.3	444.3	415.3	433.4	436.3	448.3	429.3	445.0	444.7	445.8
Filter Cake	65.6	63.0	75.2	68.0	71.5	65.0	104.0	76.3	66.4	99.6
Residues	29.9	37.6	42.5	34.0	33.9	27.0	20.7	27.7	27.0	17.6
Gas	203.5	210.3	221.4	233.2	219.6	228.5	218.5	229.1	227.4	211.6
H ₂ O	75.2	72.3	68.7	73.7	72.2	72.7	73.4	73.5	64.9	74.7
Light Oil	6.7	8.3	6.7	7.8	6.8	8.6	5.1	5.8	4.0	4.1
Total	820.2	835.8	829.8	850.1	840.3	850.1	851.0	857.4	834.4	853.4
% Recovery	94.7	96.8	95.5	96.6	95.9	96.3	97.4	97.5	95.7	96.9
Yields 100% Recovery Basis										
Product, grams										
Light Oil	41.3	56.0	28.2	31.9	32.8	33.0	23.7	25.2	23.7	21.4
Solvent	397.4	367.0	375.5	380.1	381.5	376.4	388.1	388.5	383.1	398.5
Vac. Btms	92.7	110.3	124.7	112.4	116.7	112.5	122.6	117.0	125.6	121.0
Total Liquid	531.4	533.3	528.4	524.4	531.0	521.9	534.4	530.7	532.4	540.9
Net Liquids	131.8	133.4	129.2	125.5	131.4	122.9	134.4	130.8	133.4	140.8
Net Gas	70.6	76.5	78.3	83.2	76.1	76.1	73.7	79.3	81.7	61.3
Net H ₂ O	-20.0	-25.0	-27.7	-23.4	-24.4	-24.1	-24.4	-24.4	-31.8	-22.7
Net Ash	-0.6	-2.6	-1.3	-2.9	-1.4	-2.2	-2.4	-3.8	-1.4	0.2
Unconverted Coal (MAF)	17.6	17.6	21.3	17.5	18.0	26.8	18.7	18.3	17.5	20.8
Yield Wt% MAF Coal										
Net Gas	35.4	38.3	39.2	41.6	38.1	38.2	36.8	39.6	40.9	30.6
Net Liquid	66.0	66.7	64.6	62.7	65.8	61.5	67.3	65.4	67.0	70.3
Unconverted	8.9	8.8	10.7	8.8	9.0	13.4	9.3	9.1	8.7	10.4
Net H ₂ O + Ash	-10.3	-13.8	-14.5	-13.1	-12.9	-13.1	-13.4	-14.1	-16.6	-11.3
Total Liquid Composition (Wt%)										
Light Oil	7.8	10.5	5.3	6.1	6.2	6.3	4.5	4.8	4.5	3.9
Solvent	74.8	68.8	71.1	72.5	71.8	72.1	72.6	73.2	71.9	73.7
Vacuum Btms	17.4	20.7	23.6	21.4	22.0	21.6	22.9	22.0	23.6	22.4
Net Liquid (Wt% MAF Coal)										
Net Light Oil	20.7	28.0	14.1	16.0	16.4	16.5	11.9	12.6	11.9	10.7
Net SRL	45.3	38.7	50.5	46.7	49.4	45.0	55.4	52.8	55.1	59.6
Solvent Recovery										
Wt%	94.2	88.8	89.9	92.0	91.6	90.8	94.5	94.7	91.9	96.5
Wt% (100% Rec. Basis)	99.4	91.8	94.1	95.3	95.5	94.3	97.1	97.1	96.0	99.6

TABLE 11 - PART 2: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 0-12 WEEKS

Run No.	ANALYTICAL DATA									
	BASE CASE		6 WEEKS STORAGE				12 WEEKS STORAGE			
Test Conditions	504	505	515	516	517	518	521	522	523	524
Time	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	755	750	755	760	750	750	755	755	750
Max. Press., psia	3020	3110	3270	3230	3120	3210	3160	3180	3220	2960
Gas Charged	CO/H ₂									
Solvent	UNDCAO-73									
Lignite	73-2(A)		73-2(B)				73-2(C)			
	H ₂ O Strg	Air Strg	N ₂ Strg	N ₂ Strg	Air Strg	N ₂ Strg	H ₂ O Strg	Air Strg		
Analytical Data										
1. Gas Analysis, Mol%										
H ₂	42.7	45.1	44.5	40.7	40.8	40.3	43.8	44.4	46.0	42.9
CH ₄	2.0	1.7	1.5	1.9	1.7	1.9	0.8	1.2	0.8	0.9
CO	22.6	22.6	21.3	21.7	20.5	22.2	23.7	21.7	20.5	23.7
C ₂ H ₆	0.9	0.9	0.8	0.9	0.9	0.8	0.4	0.5	0.4	0.5
CO ₂	31.1	29.1	31.4	34.1	35.4	34.2	30.9	31.7	31.9	31.4
H ₂ S	0.7	0.6	0.5	0.7	0.7	0.6	0.4	0.5	0.4	0.6
Gas Specific Grav. @RT	0.788	0.776	0.762	0.810	0.800	0.800	0.769	0.773	0.748	0.795
2. Input Coal										
Volatile Matter, Wt%	31.20	31.26	30.26	30.14	29.64	29.64	29.38	27.33	31.78	28.10
Ash, Wt%	7.34	7.33	5.36	7.38	7.41	7.41	7.59	7.64	5.96	7.33
Moisture, Wt%	29.80	29.90	30.15	29.54	30.52	30.52	27.51	28.36	28.16	30.03
Carbon, Wt% (1)	63.06	63.06	65.70	64.13	65.07	65.07	62.88	64.34	65.57	64.21
Hydrogen, Wt% (1)	4.17	4.17	4.74	3.77	4.10	4.10	4.27	4.38	4.70	4.05
Sulfur, Wt% (1)	0.79	0.79	0.53	0.95	0.94	0.94	0.90	0.96	0.52	0.98
3. Input Solvent										
Carbon, Wt%	91.58	90.71	91.04	90.96	90.92	90.92	90.98	90.58	91.05	90.76
Hydrogen, Wt%	5.98	5.94	5.98	5.91	5.93	5.93	6.04	5.89	6.00	5.92
Sulfur, Wt%	0.63	0.59	0.58	0.59	0.61	0.61	0.53	0.62	0.58	0.60
Specific Grav. 60/60	1.112	1.115	1.120	1.120	1.124	1.124	1.116	1.118	1.118	1.118
IR Ratio	1.59	1.67	1.72	1.66	1.66	1.66	1.71	1.62	1.74	1.68
4. Coal-Solvent Slurry										
5. Cake										
Ash, Wt%	27.17	26.13	17.95	26.83	26.98	29.11	18.60	23.61	21.04	21.65
Carbon, Wt%	58.71	61.65	69.56	62.19	64.54	60.54	--	--	--	--
Hydrogen, Wt%	3.71	3.84	4.28	3.75	4.15	3.68	--	--	--	--
Sulfur, Wt%	1.66	1.54	0.82	1.79	1.48	1.67	1.25	1.37	0.77	1.50
Pyridine Sol., Wt%	68.22	70.94	74.54	71.56	71.92	65.96	82.07	74.52	74.32	77.50

(1) Calculated on moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 11 - PART 2 Cont.: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 0-12 WEEKS

ANALYTICAL DATA

<u>Run No.</u>	504	505	515	516	517	518	521	522	523	524
6. <u>Filtrate</u>										
Ash, Wt%	0.02	--	0.03	0.02	0.01	0.07	0.03	0.08	0.08	0.12
Carbon, Wt%	89.90	89.60	89.56	89.75	88.99	89.45	--	--	--	--
Hydrogen, Wt%	6.30	6.30	6.12	6.20	6.29	6.33	--	--	--	--
Sulfur, Wt%	0.41	0.43	0.45	0.41	0.45	0.42	0.49	0.51	0.47	0.48
Specific Grav. 60/60	1.108	1.102	1.140	1.130	1.130	1.127	1.139	1.130	1.134	1.130
7. <u>Vacuum Btms</u>										
Ash, Wt%	0.13	0.07	0.16	0.11	0.09	0.14	0.20	0.22	0.23	0.29
Carbon, Wt%	85.57	88.06	87.86	87.52	88.09	87.29	85.68	85.34	85.86	85.29
Hydrogen, Wt%	5.62	5.70	5.40	5.46	5.83	5.74	5.99	6.07	5.85	5.90
Sulfur, Wt%	0.26	0.26	0.22	0.27	0.24	0.25	0.26	0.25	0.23	0.25
8. <u>Residue</u>										
Ash, Wt%	19.96	19.30	1.32	6.36	9.97	9.15	11.38	12.53	16.76	8.70
Carbon, Wt%	69.65	68.67	85.39	83.10	78.25	80.69	--	--	--	--
Hydrogen, Wt%	4.75	4.79	5.87	5.69	5.53	5.71	--	--	--	--
Sulfur, Wt%	1.50	1.28	0.54	0.88	0.71	0.99	1.13	1.47	0.51	1.88
Pyridine Sol., Wt%	91.31	88.22	93.81	95.95	94.44	97.41	94.22	97.04	79.12	78.83
9. <u>Light Oil</u>										
Carbon, Wt%	82.11	85.55	84.60	86.41	85.97	84.83	--	--	--	--
Hydrogen, Wt%	8.29	8.74	8.52	8.59	8.53	8.63	--	--	--	--
Sulfur, Wt%	0.59	0.67	0.46	0.40	0.46	0.48	0.49	0.45	0.39	0.45
10. <u>Product Solvent</u>										
Carbon, Wt%	89.72	91.15	90.93	91.15	91.39	89.89	90.65	90.51	89.25	90.43
Hydrogen, Wt%	6.27	6.28	6.22	6.32	6.40	6.37	6.21	6.45	6.23	6.31
Sulfur, Wt%	0.53	0.52	0.48	0.44	0.48	0.48	0.58	0.47	0.48	0.51
Specific Grav. 60/60	1.117	1.106	1.105	1.104	1.104	1.105	1.108	1.108	1.108	1.114
IR ratio	0.93	0.94	1.04	0.92	0.94	0.93	0.98	0.91	1.02	1.00

TABLE 12 - PART 1: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 24-36 WEEKS

MATERIAL BALANCE AND YIELD DATA								
Run No.	24-WEEKS STORAGE				36-WEEK STORAGE			
	539	540	541	542	561	562	563	564
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	750	750	752	752	752	752
Max. Press., psia	3070	2970	3110	3000	3075	3125	3125	3145
Gas Charged	CO/H ₂							
Solvent	UNDCAO-74							
Lignite	73-2(B)							
<u>Material In, grams</u>	N ₂ Strg	H ₂ O Strg	Air Strg	H ₂ O Strg	N ₂ Strg	Air Strg	Air Strg	H ₂ O Strg
Coal & H ₂ O	323.1	318.0	323.5	321.9	323.5	324.2	324.4	316.9
Solvent	399.8	400.2	399.9	400.0	400.5	400.4	400.7	399.9
Gas	151.2	156.7	153.5	150.7	153.4	153.3	150.4	152.0
Total	874.1	874.9	876.9	872.6	877.4	877.9	875.5	868.8
<u>Material Out, grams</u>								
Filtrate	458.5	448.8	429.3	450.1	447.0	413.3	427.9	437.2
Filter Cake	79.3	68.5	104.1	84.7	91.9	104.3	105.5	96.6
Residues	17.7	39.5	16.4	16.9	21.0	20.7	24.9	20.5
Gas	216.7	225.6	213.7	218.5	204.3	214.3	215.8	215.3
H ₂ O	78.8	59.1	79.4	72.7	74.6	78.9	78.3	70.0
Light Oil	3.0	4.3	4.0	2.8	2.1	3.4	4.1	2.4
Total	854.0	845.8	846.9	845.7	840.9	834.9	856.5	842.0
<u>% Recovery</u>	97.7	96.7	96.6	96.9	95.8	95.1	97.8	96.9
<u>Yields 100% Recovery Basis</u>								
<u>Product, grams</u>								
Light Oil	29.1	22.0	29.5	20.6	29.4	36.6	29.2	34.5
Solvent	379.0	405.8	382.4	395.3	397.3	367.5	388.3	379.7
Vac. Btms	126.3	105.2	117.6	122.4	118.6	120.0	114.0	126.1
Total Liquid	534.4	533.0	529.5	538.3	545.3	524.1	531.5	540.3
Net Liquids	134.6	132.8	129.6	138.3	144.8	123.7	130.8	140.4
Net Gas	70.6	76.7	67.8	74.7	59.8	72.0	70.2	70.2
Net H ₂ O	-19.1	-38.7	-17.6	-24.8	-22.0	-16.9	-19.9	-27.8
Net Ash	-3.0	--	-0.6	-2.3	-1.6	-0.5	-1.9	-1.0
Unconverted Coal (MAF)	16.7	12.6	20.8	13.9	19.5	22.1	21.2	18.4
<u>Yields Wt% MAF Coal</u>								
Net Gas	35.3	41.7	33.8	37.4	29.8	36.0	35.0	35.1
Net Liquid	67.5	72.4	64.8	69.1	72.2	61.7	65.3	70.1
Unconverted	8.3	6.9	10.4	7.0	9.8	11.0	10.6	9.2
Net H ₂ O + Ash	-11.1	-21.0	-9.0	-13.5	-11.8	-8.7	-10.9	-14.4
<u>Total Liquid Composition (Wt%)</u>								
Light Oil	5.5	4.1	5.6	3.8	5.4	7.0	5.5	6.4
Solvent	70.9	76.1	72.2	73.5	72.9	70.0	73.1	70.3
Vacuum Btms	23.6	19.8	22.2	22.7	21.7	23.0	21.4	23.3
<u>Net Liquid (Wt% MAF Coal)</u>								
Net Light Oil	14.5	12.0	14.7	10.3	14.7	18.2	14.6	17.2
Net SRL	53.0	60.4	50.1	58.8	57.5	43.5	50.7	52.9
<u>Solvent Recovery</u>								
Wt%	92.6	98.0	92.3	95.8	95.0	87.3	94.8	92.0
% (100% Rec. Basis)	94.8	101.4	95.6	98.8	95.5	91.8	96.9	94.9

TABLE 12 - PART 2: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 24-36 WEEKS

Run No.	ANALYTICAL DATA							
	24 WEEKS STORAGE				36-WEEK STORAGE			
Test Conditions	539	540	541	542	561	562	563	564
Time	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	750	750	752	752	752	752
Max. Press., psia	3070	2970	3110	3000	3075	3125	3125	3145
Gas Charged	CO/H ₂							
Solvent	UNDCAO-74							
Lignite	73-2(B)							
	N ₂ Strg	H ₂ O Strg	Air Strg	H ₂ O Strg	N ₂ Strg	Air Strg	Air Strg	H ₂ O Strg
Analytical Data								
1. <u>Gas Analysis, Mol%</u>								
H ₂	42.6	41.7	44.7	42.5	42.1	43.1	40.5	43.3
CH ₄	1.0	0.9	0.8	1.0	0.9	1.0	0.7	0.9
CO	23.0	16.6	23.0	20.7	22.3	22.4	23.5	18.6
C ₂ H ₆	0.5	0.4	0.3	0.6	0.4	0.4	0.4	0.4
CO ₂	32.5	38.6	30.8	34.5	33.8	32.8	34.5	36.2
H ₂ S	0.4	1.8	0.4	0.7	0.5	0.3	0.4	0.6
Gas Specific Gravity @RT	0.774	0.791	0.761	0.796	0.777	0.773	0.775	0.746
2. <u>Input Coal</u>								
Volatile Matter, Wt%	27.88	29.82	29.37	32.67	29.21	32.39	32.11	29.80
Ash, Wt%	7.25	11.45	7.50	7.23	7.49	8.12	8.05	5.44
Moisture, Wt%	30.51	26.89	29.15	26.78	27.72	23.90	24.57	30.07
Carbon, Wt% (1)	63.56	59.48	62.32	61.52	64.90	63.14	63.14	66.16
Hydrogen, Wt% (1)	4.48	4.03	4.45	4.63	3.85	3.85	3.85	4.07
Sulfur, Wt% (1)	0.99	6.70	1.00	1.50	1.03	1.04	1.08	0.75
3. <u>Input Solvent</u>								
Ash, Wt%	0.06	0.06	0.05	0.05	0.04	--	--	--
Carbon, Wt%	90.55	91.18	90.89	90.94	91.67	91.00	90.81	89.50
Hydrogen, Wt%	6.03	5.99	5.97	6.07	5.92	5.84	6.02	5.94
Sulfur, Wt%	0.61	0.62	0.65	0.62	0.55	0.59	0.58	0.53
Specific Grav. 60/60	1.125	1.116	1.117	1.116	1.114	1.119	1.124	1.154
IR Ratio	1.7	1.8	1.8	1.8	1.77	1.77	1.77	1.77
4. <u>Coal-Solvent Slurry</u>								
5. <u>Cake</u>								
Ash, Wt%	24.07	33.41	20.51	21.23	21.54	20.40	19.31	15.15
Carbon, Wt%	64.57	60.44	69.23	67.11	67.91	69.07	67.95	72.71
Hydrogen, Wt%	4.25	4.02	4.46	4.60	4.35	4.60	4.71	4.83
Sulfur, Wt%	1.57	6.61	1.64	2.36	1.42	1.63	1.45	0.64
Pyridine Sol., Wt%	76.01	77.30	78.41	82.62	77.23	78.24	79.89	81.69

(1) Calculated on a moisture-free basis.

TABLE 12 - PART 2 Cont.: EFFECT OF STORAGE ON LIGNITE REACTIVITY - 24-36 WEEKS

Run No.	ANALYTICAL DATA							
	539	540	541	542	561	562	563	564
6. Filtrate								
Ash, Wt%	--	0.08	0.08	0.14	0.03	0.04	0.02	0.04
Carbon, Wt%	89.44	88.99	87.80	89.12	88.66	89.15	87.60	88.84
Hydrogen, Wt%	6.42	6.54	6.37	6.48	6.31	6.28	6.26	6.22
Sulfur, Wt%	0.53	0.53	0.49	0.49	0.46	0.44	0.41	0.39
Blackness abs./g. @550 mu	--	--	--	--	5.78	6.66	6.54	7.77
7. Vacuum Btms								
Ash, Wt%	0.10	0.57	0.24	0.55	0.17	0.14	0.20	0.12
Carbon, Wt%	85.39	83.47	83.96	84.07	85.92	85.26	85.76	84.70
Hydrogen, Wt%	6.15	6.55	6.03	6.36	5.88	6.04	5.80	5.81
Sulfur, Wt%	0.26	0.48	0.30	0.33	0.27	0.28	0.29	0.28
Melting Point, °F	--	--	--	--	297.4	288.1	308.5	286.6
8. Residue								
Ash, Wt%	15.70	33.16	8.46	6.29	7.60	4.83	7.77	3.39
Carbon, Wt%	72.95	60.87	78.35	81.93	79.17	85.26	80.91	76.66
Hydrogen, Wt%	5.23	4.56	5.76	6.02	5.25	6.11	5.73	5.31
Sulfur, Wt%	1.23	8.94	1.06	2.03	1.04	0.59	0.65	0.54
Pyridine Sol., Wt%	93.48	95.13	95.59	98.64	100.0	92.45	93.52	97.87
9. Light Oil								
Carbon, Wt%	80.73	--	83.15	77.26	84.64	83.92	84.07	--
Hydrogen, Wt%	9.32	--	8.92	10.48	8.53	8.55	8.50	--
Sulfur, Wt%	0.44	--	0.45	0.60	0.39	0.47	0.54	0.44
10. Product Solvent								
Ash, Wt%	0.02	0.03	0.02	0.02	0.03	0.01	--	0.02
Carbon, Wt%	91.27	90.37	90.19	90.78	90.38	90.85	88.81	87.69
Hydrogen, Wt%	6.66	6.50	6.28	6.54	6.46	6.35	6.21	5.93
Sulfur, Wt%	0.51	0.57	0.55	0.52	0.55	0.45	0.42	0.47
Specific Grav. 60/60	1.105	1.102	1.106	1.105	1.119	1.114	1.114	1.113
IR Ratio	0.97	0.81	0.99	0.89	0.95	0.97	0.98	0.99

TABLE 13 PART 1: EFFECT OF REDUCING GAS ON LIGNITE LIQUEFACTION

MATERIAL BALANCE AND YIELD DATA						
Run No.	410	422	412	411	414	413
<u>Test Conditions</u>						
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	776	755	770	764	765	776
Max. Press., psia	3310	3130	3400	3520	3340	3330
Gas Charged	CO	CO/H ₂	H ₂	CO	CO/H ₂	H ₂
Starting Solvent	UNDRAO-72					
Lignite	72-1			72-1X		
	Raw Coal			Extracted Coal		
<u>Material In, grams</u>						
Coal	326.0	319.7	320.8	301.0	303.8	290.8
Water	0.0	0.0	1.3	5.3	2.8	15.2
Solvent	400.5	399.3	400.2	400.0	400.2	400.2
Gas	239.1	123.0	17.2	240.3	121.2	17.3
Total	965.6	842.0	739.5	946.6	838.0	723.5
<u>Material Out, grams</u>						
Filtrate	307.5	418.5	346.5	399.0	464.5	400.5
Filter Cake	101.5	91.0	120.5	78.0	22.0	81.5
Residues	58.5	30.0	54.0	41.0	17.0	34.5
Gas	324.8	184.9	41.0	271.2	164.0	42.3
Water	40.5	67.0	111.0	78.0	93.5	116.4
Light Oil	7.5	13.5	12.0	17.5	13.0	14.1
Total	840.3	804.9	685.0	884.7	774.0	689.3
% Recovery	87.0	95.6	92.6	93.5	93.5	95.3
<u>Yields, 100% Rec. Basis</u>						
<u>Products, grams</u>						
Light Oil	18.7	26.1	29.0	22.6	24.9	28.7
Solvent	334.1	343.7	310.0	314.7	355.1	329.9
Vac. Btms	133.2	166.7	176.8	182.8	139.5	157.2
Total liquid	486.0	536.0	515.8	520.1	519.5	515.8
Net Liquids	85.5	136.7	115.6	120.1	119.3	115.6
Net Gas	134.1	70.4	27.1	49.8	54.2	27.1
Net Water	-56.2	-29.8	20.0	-16.4	0.2	22.6
Net Ash	2.9	2.5	1.8	1.3	2.3	3.0
Unconverted Coal (MAF)	34.5	19.9	35.6	45.1	24.3	31.6
<u>Yields, Wt% MAF Coal</u>						
Net Gas	66.8	35.2	13.6	24.9	27.1	13.6
Net Liquid	42.6	68.4	57.7	60.0	59.6	57.8
Unconverted	17.2	10.0	17.8	22.6	12.1	15.8
Net H ₂ O & Ash	-26.6	-13.6	10.9	-7.5	1.2	12.8
<u>Total Liquid Composition, Wt%</u>						
Light Oil	3.9	4.9	5.6	4.3	4.8	5.5
Solvent	68.7	64.0	60.1	60.5	68.3	64.0
Vac. Btms	77.4	31.1	34.3	35.2	26.9	30.5
<u>Net Liquid Yields, Wt% MAF Coal</u>						
Net Light Oil	9.3	13.1	14.5	11.3	12.4	14.4
Net SRL	33.3	55.3	43.2	48.7	47.2	43.4
<u>Solvent Recovery</u>						
Wt%	72.6	82.3	71.7	73.7	85.1	78.6
Wt% (100% Rec.)	83.4	86.1	77.4	78.8	91.1	82.4

TABLE 13 PART 2: EFFECT OF REDUCING GAS ON LIGNITE LIQUEFACTION

ANALYTICAL DATA

Run No.	410	422	412	411	414	413			
<u>Test Conditions</u>									
Time, hrs	0.5	0.5	0.5	0.5	0.5	0.5			
Avg. Temp., °F	776	755	770	764	765	776			
Max. Press., psia	3310	3130	3400	3520	3340	3330			
Gas Charged	CO	CO/H ₂	H ₂	CO	CO/H ₂	H ₂			
Starting Solvent	UNDRAO-72								
Lignite	72-1			72-1X					
Raw Coal	Extracted Coal								
<u>Analytical Data</u>									
1. <u>Gas Analysis, Mol%</u>									
H ₂	12.7	41.2	80.4	3.1	41.1	85.0			
CH ₄	0.0	1.5	3.9	1.0	3.2	0.0			
CO	36.3	16.0	4.4	66.4	29.8	6.6			
C ₂ H ₆	0.0	0.5	0.0	0.0	0.0	0.0			
CO ₂	50.6	40.3	10.7	28.8	26.2	7.5			
H ₂ S	0.4	0.5	0.6	0.7	0.7	0.9			
Gas Specific Gravity @ RT	1.148	0.798	0.296	1.092	0.769	0.244			
2. <u>Input Coal</u>									
Volatile Matter, Wt%	31.14	31.24	31.34	31.40	31.15	32.47			
Ash, Wt%	6.90	6.26	6.91	2.16	2.14	2.23			
Moisture, Wt%	31.50	31.24	30.70	31.38	31.92	29.04			
Fixed Carbon, Wt%	30.46	31.26	31.05	35.06	34.79	36.26			
3. <u>Input Solvent</u>									
IR Ratio	1.29	1.46	1.29	1.29	1.29	1.20			
4. <u>Coal-Solvent Slurry</u>									
5. <u>Cake</u>									
Ash, Wt%	19.02	21.66	11.99	5.86	15.19	7.58			
Sulfur, Wt%	1.50	1.62	0.90	1.00	2.20	1.30			
Pyridine Solubles, Wt% (2)	72.31	79.59	75.50	55.50	41.13	67.28			
6. <u>Filtrate</u>									
Ash, Wt%	0.02	0.11	0.01	0.28	0.63	0.01			
7. <u>Vacuum Bottoms</u>									
Ash, Wt%	0.04	0.29	0.01	0.57	2.21	0.01			
Sulfur, Wt%	0.22	0.19	0.25	0.30	0.57	0.24			
8. <u>Residues</u>									
Ash, Wt%	4.77	4.70	16.30	4.53	12.37	11.61			
Sulfur, Wt%	0.41	0.66	1.41	0.68	1.38	1.32			
Pyridine Solubles, Wt% (2)	99.99	86.44	86.54	82.51	85.40	81.86			
9. <u>Light Oil</u>									
10. <u>Product Solvent</u>									
IR Ratio	0.87	0.85	1.03	0.97	0.97	1.06			

(1) Calculated on a moisture-free basis

(2) Calculated on an ash-free basis

TABLE 14: CARBONIZATION OF LIGNITE
MATERIAL BALANCE, YIELD, AND ANALYTICAL DATA

Run No.	C-1	C-2	C-3	C-4	C-5					
<u>Test Conditions</u>										
Time, hrs.	0.5	1.75	3.0	1.7	1.0					
Max. Temp., °F	745	990	775	740	777					
Avg. Temp., °F	701	817	706	706	742					
Pressure, psig	0	0	0	0	0					
Lignite	72-2(A) (2)	72-2(A) (2)	72-2(B) (2)	72-1(J) (3)	72-1(L&N) (3)					
<u>Material In, Grams</u>										
Lignite	600.0	800.0	800.0	800.0	775.0					
<u>Material Out, Grams</u>										
Char	341.0	366.0	397.5	430.0	429.7					
Residue	0.0	0.5	3.9	5.0	1.5					
Water	208.0	301.0	323.3	260.4	264.4					
Oil	24.0	28.8	12.2	49.9	60.9					
Gas	27.0 (by diff)	98.6	67.7	49.9	60.9					
Total	600.0	794.9	804.6	751.0	762.3					
<u>% Recovery</u>										
	100	99.36	100.58	93.88	98.36					
<u>100% Recovery Basis</u>										
Products	Grams	Wt%	Grams	Wt%	Grams	Wt%	Grams	Wt%	Grams	Wt%
Char	341.0	56.8	368.9	46.1	399.1	49.9	463.4	57.9	438.4	56.5
Water	208.0	34.7	302.9	37.9	321.5	40.2	277.4	34.7	268.8	34.7
Oil	24.0	4.0	29.0	3.6	12.1	1.5	6.1	0.8	5.9	0.8
Gas	27.0	4.5	99.2	12.4	67.3	8.4	53.1	6.6	61.9	8.0
<u>Analytical Data</u>										
Lignite or Char, Wt%	Lignite	Char	Lignite	Char	Lignite	Char	Lignite	Char	Lignite	Char
Volatile Matter	28.30	32.97	28.30	20.60	30.05	27.13	28.94	31.61	30.87	30.42
Ash	6.45	8.96	6.45	11.12	6.37	10.13	7.30	12.83	7.79	13.64
Moisture	34.58	0.70	33.96	0.30	34.09	0.29	24.55	0.83	28.96	0.64
Fixed Carbon	30.67	57.37	31.29	67.98	29.49	62.45	39.21	54.73	32.38	55.30
Carbon (dry basis)	--	--	--	--	--	--	--	--	62.11	69.70
Hydrogen (dry Basis)	--	--	--	--	--	--	--	--	3.97	3.70
Sulfur (dry basis)	0.62	0.60	0.56	0.41	0.61	0.49	0.70	0.53	1.21	1.30
Product Gas	Mol%	Grams	Mol%	Grams	Mol%	Grams	Mol%	Grams	Mol%	Grams
	(1)	(1)								
H ₂	1.4	0.0	35.4	2.9	2.6	0.1	5.1	0.1	13.7	0.5
CH ₄	10.8	1.2	13.7	9.0	11.2	3.1	9.3	2.1	9.6	2.8
C ₂ H ₆	1.6	0.3	1.4	1.7	2.8	1.5	1.8	0.8	1.5	0.8
CO	11.0	2.2	5.2	6.0	5.2	2.6	9.3	3.7	7.2	3.7
CO ₂	72.4	22.6	43.2	78.1	75.9	58.6	72.5	45.4	67.0	53.5
H ₂ S	2.8	0.7	1.1	1.5	2.3	1.4	2.0	1.0	1.0	0.6
Spec. Grav. Calc.	1.319 (Ar-Free)	0.836			1.329		1.291		1.180	
Volume CF	4.860 (inc. Ar)	3.588			1.550		1.176		1.571	

(1) Argon Free.

(2) 72-2 Lignite is from the Glenharrold Mine of Consolidation Coal Co.

(3) 72-1 Lignite is from the Indianhead Mine of North American Coal Co.

TABLE 15 PART 1: EFFECT OF PRE-CARBONIZATION ON LIGNITE LIQUEFACTION

Run No.	MATERIAL BALANCE AND YIELD DATA							
	441	430	440	451	450	452	457	493
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	749	749	750	765	760	759	755	753
Max. Press., psia	3740	3000	3120	3100	3000	2550	2610	2610
Gas Charged	Argon			CO/H ₂				
Solvent				UNDRAO-72				UNDCAO-72
Lignite	72-1(F)	72-1(E)	72-1(G)	72-1(H)	C-4 Char	C-5 Char		
Carbonization	-	In Solvent @500°F	In Solvent @600°F	In Solvent @700°F	Without Solv. @700°F	Without Solv. @700°F	Separate	
<u>Material In, grams</u>								
Coal	318.8	321.9	313.8	318.6	320.0	293.5	232.3	233.6
H ₂ O	2.0	0.2	9.2	3.4	2.4	28.2	98.1	98.5
Solvent	400.2	399.1	400.2	400.4	399.5	400.0	400.7	399.7
Gas	344.8	113.4	107.8	119.1	119.1	103.6	119.5	141.0
Total	1065.8	834.6	831.0	841.5	838.6	825.3	850.6	872.8
<u>Material Out, grams</u>								
Filtrate	} 466.0	424.5	430.5	395.0	357.0	344.0	} 535.0	400.3
Filter Cake		94.0	79.0	93.0	139.0	123.0		160.4
Residues	71.5	19.5	26.0	41.5	25.0	28.5	31.5	31.3
Carbonization Gas	--	26.6	14.5	26.4	65.0	58.1	--	--
Liquefaction Gas	364.0	169.5	166.7	147.9	224.7	142.3	186.5	186.9
H ₂ O	109.8	68.5	79.7	80.8	68.1	66.3	60.5	56.4
Light Oil	8.7	10.0	7.8	9.2	14.4	15.7	11.0	4.9
Total	1020.0	812.6	804.2	793.8	828.2	719.8	824.5	840.2
% Recovery	95.7	97.4	96.8	94.3	98.8	94.3	96.9	96.3
<u>Yields 100% Recovery Basis</u>								
<u>Products, grams</u>								
Light Oil	9.1	49.3	19.8	24.8	29.0	23.2	12.7	33.1
Solvent	} 342.5	350.1	329.3	319.1	349.3	352.5	223.1	337.6
Vac Btms		127.2	173.3	170.9	92.9	92.5	292.4	126.1
Total liquid	351.6	526.6	522.4	514.8	471.2	468.2	528.2	536.8
Net Liquids	-48.6	127.5	122.2	114.4	71.7	68.2	127.5	137.1
Net Gas	35.6	88.0	79.4	65.7	109.1	97.5	72.9	-53.2
Net H ₂ O	15.0	-29.1	-12.5	-14.2	-30.8	-28.3	-37.6	-41.4
Net Ash	2.7	-0.5	-2.5	-1.3	-2.0	2.2	-2.6	0.4
Unconverted Coal (MAF)	195.4	13.8	18.5	35.7	52.7	61.0	40.3	50.8
<u>Yields, Wt% MAF Coal</u>								
Net Gas	17.8	44.1	39.7	32.8	54.4	48.6	36.4	26.6
Net Liquid	-24.3	63.8	61.1	57.1	35.7	34.0	63.6	68.5
Unconverted	97.7	6.9	9.2	17.8	26.3	30.4	20.1	25.4
Net H ₂ O + Ash	8.8	-14.8	-10.0	-7.7	-16.4	-13.0	-20.1	-20.5
<u>Total Liquid Composition Wt%</u>								
Light Oil	2.6	9.4	3.8	4.8	6.2	5.0	2.4	6.2
Solvent	} 97.4	66.5	63.0	62.0	74.1	75.3	42.2	70.3
Vacuum Btms		24.1	33.2	33.2	19.7	19.7	55.4	23.5
<u>Net Liquid Yields, Wt% MAF Coal</u>								
Net Light Oil	4.5	24.7	9.9	12.4	14.4	11.6	6.3	16.5
Net SRL	--	39.1	51.2	44.7	21.3	22.4	57.3	52.0
<u>Solvent Recovery</u>								
Wt%	81.9	85.4	79.6	75.2	86.1	81.5	54.0	~ 9
Wt% (100% Rec. Basis)	87.9	87.7	82.3	79.7	87.4	88.1	55.7	

TABLE 15 PART 2: EFFECT OF PRE-CARBONIZATION ON LIGNITE LIQUEFACTION

Run No.	ANALYTICAL DATA							
	441	430	440	451	450	452	457	493
<u>Test Conditions</u>								
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	749	749	752	765	760	760	755	753
Max. Press., psia	3740	3000	3120	3100	3000	2550	2610	2610
Gas Charged	Argon			CO/H ₂				
Solvent				UNDRAO-72				
Lignite	72-1(F)	72-1(E)	72-1(G)	72-1(H)	C-4 Char	UNDCAO-72		
Carbonization	-	In Solvent @500°F	In Solvent @600°F	Without Solv @700°F	Without Solv @700°F	C-5 Char	Separate	
<u>Analytical Data</u>								
1. <u>Gas Analysis, Mol%</u>								
H ₂	0.5	53.4	47.9	48.8	48.1	46.9	40.0	43.6
CH ₄	0.3	2.0	1.6	1.8	1.7	1.3	3.6	2.3
CO	2.5	21.6	16.5	21.5	25.1	27.6	14.6	16.8
C ₂ H ₆	0.3	0.6	0.5	0.8	0.5	0.4	1.5	1.1
CO ₂	6.5	21.6	33.0	26.7	24.0	23.3	39.5	35.6
H ₂ S	0.5	0.8	0.5	0.4	0.6	0.5	0.8	0.6
Argon	89.4	--	--	--	--	--	--	--
Gas Specific Gravity	1.369	0.768	0.745	0.672	0.671	0.656	0.857	0.817
2. <u>Input Coal</u>								
Volatile Matter, Wt%	29.50	30.83	34.42	30.33	30.33	28.12	31.61	30.42
Ash, Wt%	6.58	7.14	7.34	6.84	6.82	7.12	12.83	13.64
Moisture, Wt%	30.66	30.84	28.89	30.28	30.49	24.47	0.83	0.64
Fixed Carbon, Wt%	33.26	31.19	29.35	32.55	32.36	40.29	54.73	55.30
Sulfur, Wt% (1)	1.13	1.10	1.13	0.98	0.98	0.78	--	1.30
3. <u>Input Solvent</u>								
IR Ratio	1.53	1.53	1.53	1.53	1.53	1.53	1.53	1.58
4. <u>Coal-Solvent Slurry</u>								
5. <u>Cake</u>	(3)						(3)	
Ash, Wt%	4.09	21.81	21.0 ^o	16.30	12.24	14.65	4.68	18.29
Sulfur, Wt%	0.88	2.05	1.68	1.57	0.95	1.38	0.66	1.50
Pyridine Sol., Wt% (2)	64.38	84.12	74.88	63.29	60.64	50.98	92.76	67.10
6. <u>Filtrate</u>								
Ash, Wt%	--	0.00	0.00	0.00	0.00	0.00	--	0.00
7. <u>Vacuum Btms</u>								
Ash, Wt%	--	0.00	0.01	0.01	0.00	0.14	9.37	0.09
8. <u>Residue</u>								
Ash, Wt%	5.36	11.18	16.36	13.80	13.28	16.66	5.56	7.08
Sulfur, Wt%	0.80	--	1.27	1.03	--	0.97	0.64	0.79
Pyridine Sol, Wt% (2)	77.63	97.40	92.13	87.78	87.28	82.63	92.50	94.24
9. <u>Light Oil</u>								
10. <u>Product Solvent</u>								
IR Ratio	--	1.00	1.00	1.04	0.93	0.90	0.77	0.90
11. <u>Gas from Carbonization Mol%</u>								
H ₂	--	23.2	0.7	1.3	6.2	10.9	--	--
CH ₄	--	0.4	2.3	8.3	11.8	22.3	--	--
CO	--	3.4	1.5	1.3	21.8	2.0	--	--
C ₂ H ₆	--	0.0	0.3	3.2	5.4	3.3	--	--
CO ₂	--	73.0	95.2	85.9	54.8	61.5	--	--
Gas Specific Gravity	--	1.163	1.480	1.400	1.174	1.121	--	--

(1) Calculated on a moisture-free basis. (2) Calculated on an ash-free basis. (3) Total slurry product.

TABLE 16 PART 1: EFFECT OF DEHYDRATION ON LIGNITE LIQUEFACTION

MATERIAL BALANCE AND YIELD DATA							
Run No.	442	445	530(1)	532(1)	531(1)	525	526
<u>Test Conditions</u>							
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	755	760	750	750	750	758	755
Max. Press., psia	3180	3140	2390	2450	2370	3030	3180
Gas Charged	CO/H_2		H_2		CO/H_2		
Solvent	UNDRAO-72		UNDCAO-73		UNDFS120-74		
Lignite	72-1(F) Bone dry	72-1(G) Bone dry	73-2(D)			73-2(C)	
<u>Material In, grams</u>							
Coal	223.8	222.3	302.2	300.3	299.7	317.5	317.2
H_2O	100.0	99.9	18.4	19.6	20.8	5.2	5.9
Solvent	400.7	399.7	399.9	399.1	399.9	399.2	399.0
Gas	122.0	126.0	156.6	19.2	149.2	154.5	164.2
Total	846.5	847.9	877.1	738.2	869.6	876.4	886.3
<u>Material Out, grams</u>							
Filtrate	384.5	364.0	212.1	380.2	371.8	417.6	422.3
Filter Cake	123.0	125.0	303.8	158.9	172.0	114.2	120.7
Residues	29.5	26.5	26.3	17.9	18.7	25.9	9.2
Gas	190.8	188.5	172.2	44.2	187.7	221.5	230.3
H_2O	64.8	62.0	107.3	117.7	101.6	65.5	72.2
Light Oil	8.7	11.0	10.2	2.4	7.3	1.5	1.2
Total	801.3	777.0	831.9	721.3	859.1	846.2	855.9
% Recovery	94.7	91.6	94.9	97.7	98.8	96.6	96.6
<u>Yields, 100% Recovery Basis</u>							
<u>Products, grams</u>							
Light Oil	16.0	13.9	29.2	23.0	19.3	14.2	13.3
Solvent	292.4	281.1	355.5	351.7	375.9	410.9	404.0
Vac Btms	227.3	238.8	107.1	132.5	76.1	105.9	108.2
Total Liquid	535.7	533.8	491.8	507.2	471.3	531.0	525.5
Net Liquids	135.0	134.1	91.9	108.1	71.4	131.8	126.5
Net Gas	79.6	79.7	24.9	26.0	40.8	74.9	74.3
Net H_2O	-31.5	-32.3	13.3	20.9	3.1	-31.3	-24.9
Net Ash	3.8	-0.7	-1.1	-3.3	0.7	-1.7	-1.1
Unconverted Coal (MAF)	15.5	20.1	70.7	47.6	83.1	26.0	24.9
<u>Yields, Wt% MAF Coal</u>							
Net Gas	39.3	39.7	12.5	13.1	20.5	37.5	37.1
Net Liquid	66.7	66.8	46.1	54.2	36.0	66.2	63.4
Unconverted	7.7	10.0	35.3	23.9	41.6	13.0	12.5
$\text{Net H}_2\text{O} + \text{Ash}$	-13.7	-16.5	6.1	8.8	1.9	-16.7	-13.0
<u>Total Liquid Composition (Wt%)</u>							
Light Oil	3.0	2.6	5.9	4.6	4.1	2.7	2.5
Solvent	54.6	52.7	72.3	69.3	79.8	77.4	76.9
Vacuum Btms	42.4	44.7	21.8	26.1	16.1	19.9	20.6
<u>Net Liquid Composition (Wt%) MAF Coal</u>							
Net Light Oil	7.9	6.9	14.6	11.5	9.7	7.1	6.7
Net SRL	58.8	59.9	31.5	42.7	26.3	59.1	56.7
<u>Solvent Recovery</u>							
Wt%	69.1	64.4	84.4	86.1	92.9	99.4	97.8
Wt% (100% Rec. Basis)	73.0	70.3	88.9	88.1	94.0	102.9	101.2

TABLE 16 PART 2: EFFECT OF DEHYDRATION ON LIGNITE LIQUEFACTION

<u>Run No.</u>	<u>ANALYTICAL DATA</u>							
	442	445	530	532	531	525	526	
<u>Test Conditions</u>								
Time	0.5	0.5	0.5	0.5	0.5	0.5	0.5	
Avg. Temp., °F	755	760	750	750	750	758	755	
Max. Press., psia	3180	3140	2390	2450	2370	3030	3180	
Gas Charged	CO/H ₂		H ₂		CO/H ₂		CO/H ₂	
Solvent	UNDRAO-72		UNDCAO-73		UNDFS120-74		UNDFS120-74	
Lignite	72-1(F)	72-1(G)	73-2(D)	73-2(C)	73-2(C)	73-2(C)	73-2(C)	
	Bone dry	Bone dry						
<u>Analytical Data</u>								
1. <u>Gas Analysis, Mol%</u>								
H ₂	41.0	39.5	49.1	97.6	45.5	43.8	43.1	
CH ₄	1.3	1.9	0.6	1.0	1.0	1.6	1.3	
CO	17.7	19.8	37.4	0.4	39.2	22.1	28.0	
C ₂ H ₆	0.5	0.7	0.3	0.3	0.6	0.7	0.5	
CO ₂	38.9	37.5	12.3	0.1	13.5	31.3	26.6	
H ₂ S	0.6	0.7	0.3	0.6	0.2	0.4	0.4	
Gas Specific Gravity @ RT	0.809	0.825	0.602	0.181	0.680	0.769	0.755	
2. <u>Input Coal</u>								
Volatile Matter, Wt%	29.50	30.33	32.45	32.59	32.72	30.40	30.48	
Ash, Wt%	10.30	9.94	6.83	6.86	6.89	7.36	7.38	
Moisture, Wt%	0.00	0.06	26.94	26.64	26.34	29.73	29.56	
Carbon, Wt% (1)	--	--	65.64	65.64	65.64	63.80	63.80	
Hydrogen, Wt% (1)	--	--	4.31	4.31	4.31	4.42	4.42	
Sulfur, Wt% (1)	1.27	1.21	0.71	0.71	0.71	0.80	0.80	
3. <u>Input Solvent</u>								
Carbon, Wt%	--	--	91.15	90.63	88.66	89.03	89.11	
Hydrogen, Wt%	--	--	6.19	6.00	8.28	7.91	8.02	
Sulfur, Wt%	--	--	0.60	0.57	2.63	2.54	2.53	
Specific Gravity 60/60	--	--	1.110	1.117	1.035	1.035	1.035	
IR Ratio	1.53	1.53	1.73	1.77	0.19	0.22	0.24	
4. <u>Coal-Solvent Slurry</u>								
5. <u>Cake</u>								
Ash, Wt%	18.12	14.04	5.72	9.95	11.95	17.70	17.45	
Carbon, Wt%	--	--	83.38	72.67	75.79	71.23	70.96	
Hydrogen, Wt%	--	--	5.45	4.69	5.08	5.66	5.53	
Sulfur, Wt%	1.48	1.20	0.75	0.97	1.58	2.04	2.00	
Pyridine Sol., Wt%	88.32	85.24	81.29	70.34	48.92	77.94	77.33	

(1) Calculated on a moisture-free basis

TABLE 16 PART 2 CONT: EFFECT OF DEHYDRATION ON LIGNITE LIQUEFACTION

<u>Run No.</u>	442	445	530	532	531	525	526
6. <u>Filtrate</u>							
Ash, Wt%	0.00	0.19	0.08	0.03	0.03	0.00	0.06
Carbon, Wt%	--	--	86.75	89.35	86.75	86.31	87.43
Hydrogen, Wt%	--	--	7.67	6.09	7.67	7.73	7.87
Sulfur, Wt%	--	--	0.54	0.50	2.31	1.98	2.03
Specific Gravity 60/60	--	--	1.119	1.155	1.060	1.066	1.075
7. <u>Vacuum Btms</u>							
Ash, Wt%	0.05	0.18	0.32	0.42	0.09	0.20	0.31
Carbon, Wt%	--	--	86.51	86.39	86.06	84.95	85.08
Hydrogen, Wt%	--	--	5.59	5.33	6.24	6.09	6.29
Sulfur, Wt%	0.10	0.14	0.38	0.42	0.86	0.35	0.39
8. <u>Residue</u>							
Ash, Wt%	5.47	4.40	6.06	15.50	4.73	7.64	11.67
Carbon, Wt%	--	--	83.53	75.78	83.64	80.77	78.20
Hydrogen, Wt%	--	--	5.67	5.03	7.57	7.26	7.23
Sulfur, Wt%	0.53	0.57	0.87	1.21	2.19	2.13	2.35
Pyridine Sol., Wt%	99.99	99.28	90.49	89.40	90.14	88.91	89.95
9. <u>Light Oil</u>							
Carbon, Wt%	--	--	--	82.78	80.43	--	83.30
Hydrogen, Wt%	--	--	--	7.99	9.64	--	10.72
Sulfur, Wt%	--	--	--	0.73	0.01	0.59	0.69
10. <u>Product Solvent</u>							
Carbon, Wt%	--	--	89.93	90.90	87.96	87.88	88.27
Hydrogen, Wt%	--	--	6.19	6.22	8.30	8.15	8.13
Sulfur, Wt%	--	--	0.54	0.55	2.62	2.42	2.42
Specific Gravity 60/60	--	--	1.115	1.117	1.038	1.040	1.045
IR Ratio	0.88	0.91	1.23	1.32	0.19	0.20	0.20

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 17 PART 1: LIQUEFACTION OF MISCELLANEOUS LIGNITE SAMPLES

MATERIAL BALANCE AND YIELD DATA						
Run No.	469	476	512	487	489	492
<u>Test Conditions</u>						
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	755	755	755	750
Max. Press., psia	3310	3340	3210	3210	3150	3190
Gas Charged				CO/H ₂		
Solvent				UNDCAO-73		
Lignite		Denver Basin	THAI June 1973	72-1(-1/4")	72-1 (Dry) Phenol Treated	72-1 (Wet) Phenol Treated
<u>Material In, grams</u>						
Coal	300.6	298.1	257.9	324.0	228.3	304.2
H ₂ O	19.0	20.8	60.4	0.0	88.6	0.0
Solvent	400.1	399.3	399.8	399.9	400.2	400.8
Gas	144.3	145.4	147.2	143.9	146.8	144.0
Catalyst	0.0	0.0	0.0	0.0	0.0	0.0
Total	864.0	863.6	865.3	867.8	863.9	849.0
<u>Material Out, grams</u>						
Filtrate	474.8	456.1	465.5	433.8	450.9	469.7
Filter CAKE	47.5	60.5	46.1	57.3	43.2	34.6
Residues	35.4	29.7	25.3	42.8	24.6	20.5
Gas	207.2	214.4	206.4	230.2	227.9	204.5
H ₂ O	83.2	88.8	95.7	79.9	73.6	86.3
Light Oil	6.7	5.4	6.8	6.1	5.4	11.1
Total	854.8	854.8	845.8	850.1	835.6	826.6
<u>% Recovery</u>	98.9	99.0	97.7	98.0	96.7	97.4
<u>Yields, 100% Recovery Basis</u>						
<u>Products, grams</u>						
Light Oil	23.5	26.8	34.9	33.5	36.1	45.1
Solvent	382.3	365.8	374.6	376.9	373.2	390.4
Vac Btms	132.0	129.8	124.3	111.5	118.5	96.9
Total Liquid	537.8	522.4	533.8	521.9	527.8	532.4
Net Liquids	137.7	123.1	134.0	122.0	127.6	131.6
Net Gas	65.1	71.2	64.0	91.1	88.8	66.1
Net H ₂ O	-15.7	-9.9	-2.0	-19.9	-23.9	-25.7
Net Ash	0.7	0.8	-0.1	-2.6	-0.9	0.1
Unconverted Coal (MAF)	11.6	14.0	4.3	9.5	8.4	5.2
<u>Yields, Wt% MAF Coal</u>						
Net Gas	32.6	35.7	31.9	45.6	44.4	37.1
Net Liquid	69.0	61.8	67.0	61.0	63.8	73.9
Unconverted	5.9	7.0	2.1	4.7	4.2	2.9
Net H ₂ O + Ash	-7.5	-4.5	-1.0	-11.3	-12.4	-13.3
<u>Total Liquid Composition (Wt%)</u>						
Light Oil	4.4	5.1	6.5	6.4	6.8	8.5
Solvent	71.1	70.0	70.2	72.2	70.7	73.3
Vacuum Btms	24.5	24.9	23.3	21.4	22.5	18.1
<u>Net Liquid Composition (Wt%) MAF Coal</u>						
Net Light Oil	11.8	13.4	17.4	16.7	18.0	25.3
Net SRL	57.2	48.4	49.6	44.3	45.8	48.6
<u>Solvent Recovery</u>						
Wt%	94.5	90.7	91.6	92.3	90.2	94.8
Wt% (100% Rec. Basis)	95.5	91.6	93.7	94.2	93.2	97.4

TABLE 17 PART 2: LIQUEFACTION OF MISCELLANEOUS LIGNITE SAMPLES

ANALYTICAL DATA

<u>Run No.</u>	469	476	512	487	489	492
<u>Test Conditions</u>						
Time, hrs.	0.5	0.5	0.5	0.5	0.5	0.5
Avg. Temp., °F	750	750	755	755	755	750
Max. Press., psia	3310	3340	3210	3210	3150	3190
Gas Charged				CO/H ₂		
Solvent				UNDCAO-73		
Lignite		Denver Basin		THAI June 1973	72-1(-1/4")	72-1(Dry) Phenol treated
						72-1(Wet) Phenol treated
<u>Analytical Data</u>						
1. <u>Gas Analysis, Mol%</u>						
H ₂	44.5	40.7	39.5	41.2	42.6	43.5
CH ₄	1.5	1.6	1.8	1.9	2.2	1.7
CO	33.3	32.6	31.2	20.2	24.0	23.7
C ₂ H ₆	0.8	0.9	1.0	0.9	0.9	0.7
CO ₂	19.7	24.0	25.7	35.2	29.7	29.9
H ₂ S	0.2	0.2	0.8	0.6	0.6	0.5
Gas Specific Gravity @ RT	0.739	0.742	0.815	0.809	0.815	0.765
2. <u>Input Coal</u>						
Volatile Matter, Wt%	31.52	30.45	40.17	31.50	42.35	28.60
Ash, Wt%	6.50	6.54	7.06	6.92	7.35	4.06
Moisture, Wt%	26.89	26.44	15.30	31.33	4.99	37.34
Carbon, Wt% (1)	--	63.08	62.30	62.07	62.07	77.77
Hydrogen, Wt% (1)	--	4.43	4.43	5.02	5.02	8.51
Sulfur, Wt% (1)	1.07	0.79	1.98	0.66	0.53	0.56
3. <u>Input Solvent</u>						
Carbon, Wt%	90.76	90.76	90.71	91.36	90.58	89.88
Hydrogen, Wt%	5.98	5.98	5.94	6.00	6.06	5.91
Sulfur, Wt%	0.51	0.51	0.59	0.55	0.54	0.54
Specific Gravity 60/60	1.111	1.113	1.115	1.117	1.116	1.115
IR Ratio	1.58	1.58	1.66	1.65	1.57	1.64
4. <u>Coal-Solvent Slurry</u>						
5. <u>Cake</u>						
Ash, Wt%	33.38	28.74	35.66	28.69	27.67	31.43
Carbon, Wt%	48.37	58.10	53.69	60.25	62.11	52.41
Hydrogen, Wt%	3.00	3.71	4.21	4.08	3.99	3.77
Sulfur, Wt%	1.55	1.25	5.06	2.82	1.88	2.53
Pyridine Sol, Wt%	68.84	73.45	90.12	84.08	83.43	88.72

(1) Calculated on a moisture-free basis.

TABLE 17 PART 2 (CONT): LIQUEFACTION OF MISCELLANEOUS LIGNITE SAMPLES

Run No.	469	476	512	487	489	492
6. <u>Filtrate</u>						
Ash, Wt%	0.43	0.00	0.00	0.00	0.00	0.00
Carbon, Wt%	88.84	89.61	88.88	89.90	88.56	87.18
Hydrogen, Wt%	6.18	6.27	6.38	6.48	6.32	6.28
Sulfur, Wt%	0.36	0.41	0.50	0.41	0.40	0.40
Specific Gravity 60/60	1.152	1.151	1.125	1.125	1.130	1.113
7. <u>Vacuum Btms</u>						
Ash, Wt%	2.18	0.04	0.07	0.08	0.07	0.03
Carbon, Wt%	85.14	88.01	87.15	87.58	88.31	87.14
Hydrogen, Wt%	5.20	5.10	5.94	5.85	5.53	5.29
Sulfur, Wt%	0.34	0.24	0.47	0.23	0.29	0.29
8. <u>Residue</u>						
Ash, Wt%	9.88	18.33	6.76	12.33	2.69	16.41
Carbon, Wt%	80.86	72.38	82.09	76.96	87.91	74.63
Hydrogen, Wt%	5.75	5.06	6.21	5.63	6.33	5.54
Sulfur, Wt%	0.50	0.64	1.64	1.10	0.56	0.87
Pyridine Sol., Wt%	97.24	89.65	95.61	95.82	93.55	99.05
9. <u>Light Oil</u>						
Carbon, Wt%	86.39	86.15	84.67	85.03	85.13	81.15
Hydrogen, Wt%	9.08	8.97	9.30	9.17	8.18	8.37
Sulfur, Wt%	0.40	0.43	0.56	0.42	0.73	0.39
10. <u>Product Solvent</u>						
Carbon, Wt%	90.67	90.90	90.71	86.10	90.83	90.13
Hydrogen, Wt%	6.37	6.38	7.03	6.24	6.47	6.33
Sulfur, Wt%	0.36	0.47	0.41	0.43	0.43	0.42
Specific Gravity 60/60	1.107	1.105	1.105	1.105	1.106	1.115
IR Ratio	0.75	0.90	0.93	0.78	0.92	0.91

(1) Calculated on a moisture-free basis.

(2) Brookfield Viscosity measured at approximately 23°C.

TABLE 18 - PART 1: LIQUEFACTION PRODUCT WORK-UPS WITHOUT FILTRATION

MATERIAL BALANCE AND YIELD DATA				
Run No.	444	481	482	488
<u>Test Conditions</u>				
Time, hrs.	0.5	0.5	0.5	0.5
Avg. Temp., °F	752	752	752	745
Max. Press., psia	3115	3105	3045	3045
Gas Charged	CO/H ₂			
Starting Solvent	UNDRAO-72	UNDCAO-73		
Lignite	72-1(G)	72-1(K)	72-1(K)	72-1(M)
<u>Material In, grams</u>				
Coal	320.6	314.5	314.5	294.4
Water	1.5	8.1	8.1	28.5
Solvent	400.4	399.8	399.9	400.1
Gas	127.5	141.4	145.8	146.8
Total	850.0	863.8	868.3	869.8
<u>Material Out, grams</u>				
Unfiltered Coal Prod.	539.0	506.9	511.6	510.4
Residues	10.5	41.5	29.2	41.9
Gas	193.1	235.4	226.1	222.6
Water	60.3	74.2	71.1	75.0
Light Oil	9.2	7.1	11.6	7.1
Total	812.1	865.1	849.6	857.0
% Recovery	95.5	100.1	97.9	98.5
<u>Yields, 100% Rec. Basis,</u>				
<u>Products, grams</u>				
Light Oil	37.6	34.0	35.7	28.5
Solvent	338.4	379.5	382.5	390.7
Vac. Btms	176.6	87.6	93.6	90.2
Total Liquid	552.6	501.1	511.8	509.4
Net Liquids	152.2	101.3	111.9	109.3
Net Gas	74.6	93.7	85.3	79.1
Net Water	-36.8	-25.6	-27.1	-23.7
Net Ash	-1.0	2.0	1.9	-3.0
Unconverted Coal (MAF)	11.6	28.7	28.0	38.6
<u>Yields, Wt% MAF Coal</u>				
Net Gas	37.2	46.8	42.6	39.5
Net Liquid	75.9	50.6	56.0	54.5
Unconverted	5.8	14.4	14.0	19.3
Net H ₂ O & Ash	-18.9	-11.8	-12.6	-13.3
<u>Total Liquid Composition, (Wt%)</u>				
Light Oil	6.8	6.8	7.0	5.6
Solvent	61.2	75.7	74.7	76.7
Vac. Btms	32.0	17.5	18.3	17.7
<u>Net Liquid Yield, Wt% MAF Coal</u>				
Net Light Oil	18.7	17.0	17.8	14.2
Net SRL	57.2	33.6	38.2	40.3
<u>Solvent Recovery</u>				
Wt%	80.7	95.1	93.6	96
Wt% (100% Rec.)	84.5	94.9	95.6	97

TABLE 18 - PART 2: LIQUEFACTION PRODUCT WORK-UPS WITHOUT FILTRATION

<u>ANALYTICAL DATA</u>				
<u>Run No.</u>	444	481	482	488
<u>Test Conditions</u>				
Time, hrs.	0.5	0.5	0.5	0.5
Avg. Temp., °F	752	752	752	745
Max. Press., psia	3115	3105	3045	3045
Gas Charged	CO/H ₂			
Starting Solvent	UNDRAO-72	72-1(K)	UNDCAO-73	72-1(K)
Lignite	72-1(G)	72-1(K)	72-1(K)	72-1(M)
<u>Analytical Data</u>				
<u>1. Gas Analysis, Mol%</u>				
H ₂	39.7	41.7	39.5	42.1
CH ₄	1.2	2.1	3.3	1.9
CO	17.5	20.7	23.0	22.4
C ₂ H ₆	0.5	1.0	0.9	0.8
CO ₂	40.5	33.9	32.7	32.3
H ₂ S	0.6	0.6	0.6	0.5
Gas Specific Gravity @ RT	0.836	0.812	0.818	0.793
<u>2. Input Coal</u>				
Volatile Matter, Wt%	30.33	30.94	30.36	32.98
Ash, Wt%	6.80	7.24	7.24	7.75
Moisture, Wt%	30.68	29.14	29.15	24.23
Fixed Carbon, Wt%	32.19	32.68	33.25	35.04
Carbon, Wt% (1)	---	63.08	63.08	62.18
Hydrogen, Wt% (1)	---	4.43	4.43	7.70
Sulfur, Wt% (1)	1.21	1.10	1.16	1.02
<u>3. Input Solvent</u>				
Ash, Wt%	0.00	0.00	0.00	0.00
Carbon, Wt%	0.00	91.36	91.36	0.00
Hydrogen, Wt%	---	6.00	6.00	---
Sulfur, Wt%	---	0.55	0.55	0.57
IR Ratio	---	1.65	1.65	1.58
<u>4. Unfiltered Coal Product</u>				
Ash, Wt%	3.56	1.27	2.64	1.31
Carbon, Wt%	---	89.50	89.09	---
Hydrogen, Wt%	---	6.31	6.33	---
Sulfur, Wt%	---	0.44	0.38	0.47
Specific Gravity, 60/60°F	---	1.152	1.131	---
Pyridine Solubles, Wt% (2)	97.98	94.54	94.48	93.10
Distillation, Wt%				
Light Oils	4.9	4.9	4.3	3.8
Solvent	58.9	69.2	69.1	69.7
Vacuum Btms	36.2	25.9	26.6	26.5
<u>5. Vacuum Bottoms</u>				
Ash, Wt%	13.44	18.30	16.60	23.43
Carbon, Wt%	---	72.16	73.13	---
Hydrogen, Wt%	---	4.28	4.32	---
Sulfur, Wt%	0.95	1.55	1.43	---

TABLE 18 - PART 2 Cont: LIQUEFACTION PRODUCT WORK-UPS WITHOUT FILTRATION

ANALYTICAL DATA

<u>Run No.</u>	444	481	482	48
6. Residues				
Ash, Wt%	9.24	2.02	5.24	0.72
Carbon, Wt%	---	86.31	77.58	---
Hydrogen, Wt%	---	6.07	4.44	---
Sulfur, Wt%	---	0.20	0.49	0.68
Pyridine Solubles, Wt% (2)	95.27	93.98	98.64	90.14
7. Light Oil				
Carbon, Wt%	---	86.13	85.83	---
Hydrogen, Wt%	---	8.66	8.67	---
Sulfur, Wt%	---	0.34	0.34	---
8. Product Solvent				
Carbon, Wt%	---	89.72	90.60	---
Hydrogen, Wt%	---	6.37	6.43	---
Sulfur, Wt%	---	0.36	0.39	---
Specific Gravity, 60/60°F	---	1.106	1.100	---
IR Ratio	---	0.89	0.89	---

TABLE 19: EVALUATION OF LIQUEFACTION SOLVENTS

UND Designation (See Notes)	DISTILLATION DATA ON THE SOLVENTS							
	RAO	HRAO	CAO	HCAO	RCO	HRCO	CCO	HCCO
Vacuum Distillation @ 1.6 Torr								
IBP - 100°C, Wt%	6.2	7.0	3.9	6.0	26.8	16.3	20.5	16.3
100°-230°C, Wt%	85.4	83.6	84.7	85.6	68.5	78.6	72.9	78.3
230°+Bottoms, Wt%	8.4	9.4	11.4	8.4	4.7	5.1	6.6	5.4
ASTM D-246 Distillation								
IBP, °C	232		234		194		193	
IBP - 210°C, Wt%	0.0		0.0		0.6		0.7	
210°-235°C, Wt%	0.1		0.1		7.5		9.1	
235°-270°C, Wt%	2.2		2.6		27.9		27.9	
270°-315°C, Wt%	21.7		22.1		24.4		23.2	
315°-355°C, Wt%	35.7		36.6		22.7		18.0	
355°C+Bottoms, Wt%	40.3		38.6		16.9		21.1	
Atmospheric Distillation to 350°F								
IBP - 350°F, Wt%	0.6		0.7		1.2		1.0	
Topped 350°F + Cut, Wt%	99.4		99.3		98.8		97.0	
ASTM D-246 Distillation of Topped 350°F + Cut								
IBP, °C	251		254		---		---	
IBP - 210°C, Wt%	0.0		0.0		0.0		0.3	
210°-235°C, Wt%	0.0		0.0		5.0		6.5	
235°-270°C, Wt%	1.8		0.8		26.8		29.7	
270°-315°C, Wt%	10.7		13.1		24.6		24.5	
315°-355°C, Wt%	34.3		32.7		22.7		23.8	
355°C + Bottoms, Wt%	53.2		53.4		20.9		15.2	
Vacuum Distillation @ 1.6 Torr of Topped 350°F + Cut								
IBP - 100°C, Wt%	6.6		7.1		29.2		32.9	
100°-230°C, Wt%	86.8		84.0		66.2		60.5	
230°C + Bottoms, Wt%	6.5		8.9		4.6		6.6	
Used in Runs Nos								
As Received								
Topped to 350°F	511		510		463		464	
Standard 100°-230°C @ 1.6 Torr Cut	455	468	465,483	471,480	447,448	479	462	474
			484		491			

Notes: RAO, Raw Anthracene Oil; HRAO, Hydrogenated Raw Anthracene Oil; CAO, Chilled Anthracene Oil; HCAO, Hydrogenated Chilled Anthracene Oil; RCO, Raw Creosote Oil; HRCO, Hydrogenated Raw Creosote Oil; CCO, Chilled Creosote Oil; HCCO, Hydrogenated Chilled Creosote Oil.

RAO, CAO, RCO, and CCO from Reilly Tar and Chemical Co.

TABLE 19 Cont: EVALUATION OF LIQUEFACTION SOLVENTS

DISTILLATION DATA ON THE SOLVENTS								
UND Designation (See Notes)	LCO	MHCO	FS120	FO-6	FO-5	HAN	ATS-2	AC
Vacuum Distillation @ 1.6 Torr								
IBP - 100°C, Wt%			0.5	6.9	0.8	47.2	0.3	0.0
100°-230°C, Wt%			65.2	24.2	46.3	52.8	55.4	23.2
230°C + Bottoms, Wt%			34.3	66.8	52.9	0.0	44.3	76.8
ASTM D-246 Distillation								
IBP, °C	238	339	316	218	276	214	271	355
IBP - 210°C, Wt%	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
210°-235°C, Wt%	0.0	0.0	0.0	0.5	0.0	9.2	0.0	0.0
235°-270°C, Wt%	48.4	0.0	0.0	3.0	0.0	70.3	0.0	0.0
270°-315°C, Wt%	43.8	0.0	0.0	8.0	0.5	19.0	20.6	0.0
315°-355°C, Wt%	6.4	9.9	2.4	6.9	3.0	1.0	24.7	0.0
355°C + Bottoms, Wt%	1.4	90.1	97.6	81.6	96.5	0.5	54.7	100.0
Used in Runs Nos								
As Received	503	507,508	514		513	519	496,509	500,506
Topped to 350°F				485,497	473,478	486		
Standard 100°-230°C @ 1.6 Torr Cut							495,499	494,501

Notes: LCO Light Creosote Oil; MHCO, Middle Heavy Creosote Oil; FS120, FS120 Carbon Black Feed Stock; FO-6, No. 6 Fuel Oil; FO-5, No. 5 Fuel Oil; HAN, Heavy Aromatic Naphtha; ATS-2, Aromatic Tar S-2; AC, Aromatic Concentrate.

LCO and MHCO from United States Steel Co.

FS120 from Gulf Oil Co.

FO-5 from Standard Oil Company

FO-6, HAN, ATS-2, and AC from EXXON Company, USA.