Energy

F
O
S
S
I
L

233/84 J5 (2)
7-6

DR. 0179.1

DOE/PC/60048-2
(DE84011720)

ADVANCED DIRECT COAL LIQUEFACTION

Quarterly Technical Progress Report No. 2
for the Period December 1983—February 1984

By
A. S. Paranjape

April 30, 1984
Date Published

Work Performed Under Contract No. AC22-83PC60048

Kerr-McGee Corporation
Oklahoma City, Oklahoma

Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy

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.

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.

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A06
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts (ERA)*; *Government Reports Announcements and Index (GRA and I)*; *Scientific and Technical Abstract Reports (STAR)*; and publication NTIS-PR-360 available from NTIS at the above address.

ADVANCED DIRECT COAL LIQUEFACTION
QUARTERLY TECHNICAL PROGRESS REPORT NO. 2
PERIOD DECEMBER 1983 - FEBRUARY 1984

Prepared by
A. S. Paranjape

KERR-McGEE CORPORATION
Technology Division
Project Engineering Department
Oklahoma City, Oklahoma 73125

DATE PUBLISHED: April 30, 1984

Prepared for the United States
Department of Energy

Contract No. DE-AC22-83PC600048

TABLE OF CONTENTS

Section	<u>Page</u>
1 ABSTRACT	1-1
2 INTRODUCTION	2-1
3 SUMMARY	3-1
4 PROGRAM OUTLINE	4-1
5 BENCH-SCALE OPERATION	5-1
TASK 2 RUNS	
BENCH-SCALE RUN 35	5-1
BENCH-SCALE RUN 36	5-2
BENCH-SCALE RUN 37	5-2
TASK 3 RUNS	
BENCH-SCALE RUN 38	5-3
BENCH-SCALE RUN 39	5-4
6 BENCH-SCALE RESULTS	6-1
TASK 2 RUNS	
RESULTS - RUN 35	6-1
RESULTS - RUN 36	6-5
RESULTS - RUN 37	6-5
TASK 3 RUNS	
RESULTS - RUN 38 and 39	6-12
7 BATCH REACTOR TEST PROGRAM	7-1
BATCH REACTOR TESTS - TASK 2	
COKE PRECURSOR STUDIES	7-1
COAL LIQUEFACTION TESTS - SUBBITUMINOUS COAL	7-4
COAL LIQUEFACTION TESTS - BITUMINOUS COAL	7-9
BATCH REACTOR TESTS - TASK 3	7-10

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
5-1	Kerr-McGee Process Configuration No. 6	5-6
6-1	Summary of Operating Parameter Changes and Coal Conversion - Run 37	6-16
6-2	Volume Pore Size Distribution in Shell 324M Catalyst when used in Continuous Hydrotreatment of Full-Range Deashed Resid, Light Deashed Resid and Deashed Resid.	6-17
7-1	Volume Pore Size Distribution for Aged Catalyst after Hydro-processing Different Types of Resids	7-12
7-2	Volume Pore Size Distribution for Aged Catalyst after Hydro-processing Different Types of Resids	7-13
7-3	Volume Pore Size Distribution Comparison of Aged Catalyst "Oxide-Form" with and without Solvent-Conditioning	7-14
7-4	Volume Pore Size Distribution Comparison of Aged Catalyst "Presulfided-Form" with and without Solvent-Conditioning	7-15
7-5	Time-Temperature Profile for Batch Reactor Tests 83E6 and 83E7	7-16
7-6	Time-Temperature Profile for Batch Reactor Tests 83E3 and 83E8	7-17
7-7	Time-Temperature Profile for Batch Reactor Tests 84E21 and 84E22	7-18

TABLES

<u>Table</u>		<u>Page</u>
5-1	Summary of Operating Conditions - Run 35	5-7
5-2	Summary of Operating Conditions - Run 36	5-7
5-3	Summary of Operating Conditions - Run 37	5-8
5-4	Summary of Operating Conditions - Run 38	5-8
5-5	Summary of Operating Conditions - Run 39	5-9
6-1	Bench-Scale Runs - Overall Two-Stage Yields	6-18
6-2	Bench-Scale Runs - Thermal and Catalytic Stage Yields	6-19
6-3	Bench-Scale Runs - Summary of Overall Process Results	6-20
6-4	Summary of Process Results for Different Runs	6-21
6-5	Process Parameters used for Material Balances with Zero-Resid Production	6-22
6-6	Bench-Scale Runs - Comparison of the Composition of Liquefaction Streams in Different Runs	6-23
6-7	Bench-Scale Runs - Comparison of the Composition of Vacuum Bottoms in Different Runs	6-24
6-8	Bench-Scale Runs - Comparison of the Composition of CSD Products in Different Runs	6-25
6-9	Bench-Scale Runs - Comparison of the Composition of Hydrotreater Streams in Different Runs	6-26
6-10	List of Operating Parameters that could Possibly Affect Coal Conversion - Run 37	6-27
6-11	Comparison of Preheater plus Dissolver Volume for Different Runs	6-28
6-12	Vacuum-Oil Analyses Summary	6-29
6-13	Results of Evaluation of Run 37 Liquefaction Solvent	6-30
6-14	Summary of Spent Shell 324M Catalyst Analyses	6-31
7-1	Catalyst Aging Effect using LDAR	7-19
7-2	Catalyst Aging Effect using DAR	7-19
7-3	Comparison of Catalyst Aging Effect using Different Resids	7-20
7-4	Effect of Solvent Preconditioning on Oxide-Type Catalyst Feedstock DAR	7-21
7-5	Effect of Solvent Preconditioning on Presulfided-Type Catalyst Feedstock DAR	7-21
7-6	Summary of Batch Reactor Coal Liquefaction Tests - Task 2 - Subbituminous Coal	7-22

TABLES - contd

<u>Table</u>		<u>Page</u>
7-7	Summary of Batch Reactor Coal Liquefaction Tests - Task 2 - Bituminous Coal	7-23
7-8	Summary of Batch Reactor Coal Liquefaction Tests - Task 3	7-24
A-1	Daily Operating Log - Bench-Scale Runs	A-1
A-2	Coal Liquefaction Bench-Scale Operations On-Stream Factor	A-6
A-3	Bench-Scale Unit - Hydrotreater Catalyst Age Log	A-7
B-1	Operating Data - Thermal Stage	B-1
B-2	Operating Data - Catalytic Stage	B-4
C-1	Analytical Data - Sheet 1	C-1
C-2	Analytical Data - Sheet 2	C-4
C-3	Comparison of Analyses of Iron Oxide	C-7
D-1	Analysis of Starting Materials used in Batch Reactor Coke-Precursor Studies	D-1
D-2	Analysis of Starting Materials used in Batch Reactor Coal Liquefaction Studies	D-2
D-3	Analyses of Various Disposable Catalysts	D-3

SYMBOLS AND ABBREVIATIONS

A°	Angstrom
AC	Ash Concentrate
CSD	Critical Solvent Deashing
DAR	Deashed Resid
DAS	Deashing Solvent
DOE	Department of Energy
EPRI	Electric Power Research Institute
°F	Degrees Fahrenheit
FR-DAR	Full-Range Deashed Resid
H-DAR	Hydrotreated Deashed Resid
H-LDAR	Hydrotreated Light Deashed Resid
HTR	Hydrotreater
ID	Inside Diameter
LDAR	Light Deashed Resid
LFU	Liquefaction
MAF	Moisture and Ash-Free
MB	Material Balance
MF	Moisture-Free
Min	Minutes
NA	Not Available
No	Number
PA	Preasphaltenes
PT	Pressure Transmitter
R	Reactor
SRT	Short Reaction (Residence) Time
SS	Stainless Steel
THF	Tetrahydrofuran
UC	Unconverted Coal

as-is	uncorrected
avg	average
btms	bottoms
cat	catalyst
conc	concentrate, concentration
conv	conversion
cm ³ /gm	cubic centimeter per gram
cp	centipoise
cu ft	cubic feet
Dist, dist	distillables, distillate
Est, est	estimated
ft ²	square feet
ft ³	cubic feet
g	gram (s)
g/cm ³	grams per cubic centimeter
hr, hrs	hour (s)
lb, lbs	pound (s)
lb-cat	pound of catalyst
lb/hr	pounds per hour
M ² /g	square meters per gram

Preasph, preasph
psi
psia
psig
Pyr
r
sol
solv
Temp, temp
vac
vs
wt

preasphaltenes
pounds per square inch
pounds per square inch absolute
pounds per square inch gauge
Pyridine
radius
soluble
solvent
temperature
vacuum
versus
weight

%

percent

@

at

<--->

negative value

#

pounds, number

Section 1

ABSTRACT

During the December 1983 - February 1984 period, five Bench-Scale coal liquefaction runs were completed. Wyoming subbituminous coal was liquefied in a two-stage process scheme. In this process scheme, LDAR, the lighter fraction of ash-free resid, was fed to the catalytic stage prior to its recycle to the thermal stage, whereas DAR, the heavy fraction of the deashed resid, was directly recycled to the thermal stage without any intermediate processing step. Three Bench-Scale Runs (Nos. 35, 36 and 37) were completed under Task 2 and the remaining two Runs (Nos. 38 and 39) were completed under Task 3.

The results indicate that increasing coal space rate in the dissolver resulted in lower coal conversion and reduced distillate yield in this process configuration. The coal conversions decreased from 92 wt% to 89 wt% (MAF coal) and the distillate yield was reduced from 50 wt% to less than 40 wt% (MAF coal), as the coal space velocity increased. Attempts to duplicate the yields of Run 32, completed during previous quarter, at comparable process conditions in Runs 37 and 38, were unsuccessful. Several process parameters were investigated but failed to show why the yields of Run 32 could not be duplicated. Valuable process related information was gained as a result of process parameter studies completed during these runs. At comparable process conditions, coal conversions were lower by about 3-4 relative percent and were only in the 87 wt% (MAF coal) range. Similarly, the distillate yield was about 40 wt% (MAF coal) which is about 10 wt% lower than observed in Run 32. Although no exact cause for these results could be determined, it appeared that the H/C atomic ratio of the solvent and possibly the flow pattern (plug-flow versus back-mixed) could have affected the coal conversion and quantity of distillate product produced. Further evaluation is required to establish with a degree of certainty which particular process condition(s) affected these results.

A significant decrease in coal conversion of 4-5 wt% was observed when the disposable catalyst (iron oxide) was removed from the reaction mixture and therefore substantiates the need for a disposable catalyst in the liquefaction of Wyoming subbituminous coal.

This work is jointly sponsored by the Department of Energy, the Electric Power Research Institute and the Kerr-McGee Corporation.

Section 2

INTRODUCTION

Kerr-McGee Corporation has been actively involved in coal-related research over the last two decades. Earlier research led to the development of a proprietary process, Critical Solvent Deashing (CSD), for the selective and continuous removal of ash, mineral matter and unconverted coal from coal liquids. In 1977, a transportable CSD unit was built by Kerr-McGee Corporation, with the fabrication of the fractionation portion of the unit being funded by EPRI. The CSD unit is being provided on a no-fee basis by Kerr-McGee Corporation to the Advanced Coal Liquefaction Facility at Wilsonville, ALA, managed by Southern Company Services under DOE and EPRI contracts.

The CSD unit, being able to not only deash but also fractionate the resid, greatly increased the flexibility of options for coal liquefaction. New process concepts evolved incorporating resid fractionation and selective resid recycle in coal liquefaction. As a result of Kerr-McGee's and EPRI's interest in developing this technology, Kerr-McGee and EPRI jointly sponsored a multimillion dollar multi-year effort starting in August of 1979 for conducting research in Advanced Coal Liquefaction Technology. As a part of this effort, a new fifteen-pounds-per-hour coal-feed capacity Bench-Scale unit was built and has been operated at the Cimmaron Facility of the Kerr-McGee Corporation since 1980.

The current DOE contract was awarded to the Kerr-McGee Corporation on 2 September 1983 to perform exploratory research and development on process variations in two-stage liquefaction with emphasis on catalyst deactivation. This contract is jointly funded by DOE, EPRI and Kerr-McGee. Results derived from this program are expected to support and guide future Wilsonville coal liquefaction research and development.

This report covers work done during the December 1983 - February 1984 period on the Bench-Scale unit in testing one process option in two-stage liquefaction. Results from each of the five Bench-Scale runs completed as well as other related laboratory tests are reported.

Section 3

SUMMARY

Five bench-scale coal liquefaction runs were completed using Kerr-McGee Process Configuration No. 6. Wyoming subbituminous coal was liquefied under varying process conditions. Results indicate that increased coal space rate in the dissolver resulted in lower coal conversions and reduced distillate yields. Attempts to duplicate the results of Run 32, completed earlier, under similar process conditions were unsuccessful. The exact reason for this could not be readily ascertained. The need of a disposable catalyst conversion for improved coal conversion and distillate yield from Wyoming subbituminous coal was established.

Of the five bench-scale runs completed, the first three runs, Run Nos. 35, 36 and 37, were completed under Task 2. The remaining two bench-scale runs, Nos. 38 and 39, were completed under Task 3. All the bench-scale runs were made using the same two-stage process configuration. Wyoming subbituminous coal obtained from Kerr-McGee Clovis Point mine (upper seam) was liquefied using Kerr-McGee Process Configuration No. 6. In this particular process configuration, the overhead from the CSD first stage is fractionated in the CSD second stage into Deash Resid (DAR) and Light Deashed Resid (LDAR). The LDAR fraction is fed to the catalytic hydrotreater to produce a hydrotreater underflow containing hydrotreated LDAR (H-LDAR). This underflow containing H-LDAR is then mixed with DAR to provide the liquefaction solvent for the thermal stage.

Run 35, the first of three bench-scale runs completed during this quarter under Task 2, was started on 27 November. The primary objective of this run was to study the effect of higher coal space rate on the product distribution and yield. During this run, coal space rate in the dissolver was increased from 19 to 26 lb/hr-cu ft and the hydrotreater underflow was flashed at 310°F to increase its resid (+850°F fraction) concentration. Coal conversions in the 86-90 wt% (MAF coal) and a coal value rejection of 26-30 wt % (MAF coal) to ash concentrate were common during Run 35. After completion of two 24-hour material balances, Run 35 was ended on 13 December.

Based on the Run 35 material balances, a distillate yield of about 35 wt% (MAF coal) and a coal values rejection to the ash concentrate of about 29 wt % (MAF coal) was achieved. These results are not as favorable as those obtained in Run 32, and it

is likely that the poorer results are the direct result of the higher space rate used in Run 35.

Following Run 35, process conditions in CSD section were changed for Run 36 to alter the LDAR/DAR split. An attempt was made to increase the LDAR cut from 30 to 51 wt% of the total resid. Obviously, such a split would have meant that LDAR in Run 36 would contain heavier resid than the LDAR produced in Run 35. The hydrotreater catalyst from Run 35 was retained for Run 36 in view of its low age of 20.5 lbs-resid/lb-catalyst. Run 36 was started on 14 December without interruption following Run 35. However, steady integrated operation could not be established due to mechanical problems of plugged lines and letdown valves. Finally, Run 36 was discontinued for mechanical cleanout of CSD first stage vessel and associated piping.

Run 37 was the third bench-scale run completed during this quarter under Task 2 of the contract. This run was basically to be a duplicate of Run 32. Run 37 was made to determine if the high distillate yield achieved in Run 32 could be duplicated in a second run at a different time. Run 37 was started on 3 January. The hydrotreater catalyst used in Run 35 and 36 was retained in the unit because of its low age of 35 lbs-resid/lb-catalyst. Although good integrated operation was achieved, the run had to be extended well beyond the original planned duration of 10 days. This was because of the observed relatively low coal conversions in the 85-87 wt% (MAF coal) range compared to 91-93 wt% achieved routinely in Run 32. Several possible causes for the low coal conversions were investigated and several process changes were initiated to improve the coal conversions. Coal conversions comparable to Run 32 could be achieved by either increasing thermal severity or by decreasing the hydrotreater space rate. However, in Run 37, it was not possible to duplicate Run 32 results in terms of distillate yield and coal conversions at the same process parameters. After completion of two material balances, Run 37 was terminated on 6 February. Analysis of the spent catalyst removed after Run 37, which had a combined age of 117 lbs-resid/lb-catalyst from Runs 35, 36 and 37, did not appear to be significantly different from the spent catalyst from Run 32 and therefore, does not appear to be the cause for the difference between the two runs.

Following the completion of Task 2 runs, two additional bench-scale runs were completed. These runs were part of Task 3 work and were made to evaluate the use of a disposable catalyst at varying concentration to characterize the possible relationship of catalyst concentration with coal conversion, distillate yield and quality of products. Kerr-

McGee Process Configuration No. 6 was again used for these series of runs. The first of these Task 3 runs, Run 38, was made using 2 wt% iron oxide followed by Run 39 which did not use any iron oxide as a disposable catalyst. In this fashion, Run 38 also served as a second attempt to duplicate the results of Run 32. Run 38 was started on 10 February. During the start-up phase of operation, the entire solvent inventory was re-processed to remove the lighter components and decrease the H/C atomic ratio of the solvent material to the same level as Run 32. Following good integrated operation and two 24-hour material balances, Run 38 ended on 20 February. The results were encouraging but were not as favorable as those obtained in Run 32. Coal conversions were in the 90-91 wt % (MAF coal) range and the distillate yield of about 41 wt% (zero-resid basis) was obtained. This latter value is about 10% less than was achieved in Run 32.

Immediately following Run 38, process conditions were changed on 21 February to start Run 39. The coal feed was changed to one without any iron oxide. A very significant difference in coal conversions was observed as the iron oxide was removed from coal feed. The average coal conversions decreased from 90-91 wt% to 85-86 wt% range (MAF coal). This reduction of 4-5 % in coal conversion is believed to be a direct result of absence of iron oxide as a disposable catalyst. Similarly, the distillate yield decreased from 41 wt% to about 27 wt% (zero-resid basis) and coal value rejection to ash concentrate increased by about 10 wt% to approach 30 wt% (MAF coal) level. Run 39 was terminated earlier than planned in view of these poor results, and process conditions changed for Run 40. This particular run was just started as the quarter ended. In this run, the disposable catalyst concentration has been increased to 4 wt % (MF coal). Results of this run will be reported in the next quarterly report.

Section 4

PROJECT OUTLINE

The overall objectives of this project are to evaluate alternate two-stage process options involving selective recycle of fractionated resid and distillate to determine

- o which option or options provide improved product yields by maximizing the distillate products,
- o which process option or options result in reduced quantity of coke precursors in the resid feed to the catalytic stage,
- o the effectiveness of a disposable catalyst on improving the overall unit operability and the yield and quality of resultant liquid products,
- o the preferred process parameters for the secondary extraction step to evaluate CSD secondary extraction as a process option for further improvement of soluble-coal recovery.

Project studies for the above objectives are to be made using Wyoming subbituminous coal with additional comparative runs to be made using Illinois No. 6 bituminous coal. The research program is subdivided into various tasks. The specific objectives and outlines of each tasks are discussed in more detail in February 84 Quarterly Report.

Section 5

BENCH-SCALE OPERATION

TASK 2 RUNS

BENCH-SCALE RUN 35

Bench-scale Run No. 35 was the first run completed in this quarter. Run 35 was basically similar to an earlier run (Run 32) except that the coal space rate in the dissolver was increased from 19 (Run 32) to 26 lb/hr-cu ft. Furthermore, during Run 35, the hydrotreater underflow was flashed at about 310°F for removal of lighter components of the liquefaction solvent. This was done primarily to increase resid concentration in the liquefaction solvent. During this run, CSD deashed product split was intended to be 30 wt% of resid being removed as LDAR, the CSD third-stage product. However in actuality, on the basis of MB data, a 53 wt% of the resid was separated as LDAR.

Run 35 was started on 27 November. Fresh Shell 324M catalyst was presulfided using dimethyl-disulfide in No. 2 fuel oil. Presulfiding was completed on 29 November. Using process-derived vacuum bottoms from Run 34, the system resid inventory was built-up and the resid recycle from CSD section was started on 1 December. Coal feed was started on 2 December and by 4 December all three sections (LFU, HTR & CSD) were stabilized and were operating in an integrated mode. After a short integrated period of operation, intermittent plugging of coal slurry lines prevented smooth operation for several days. This was caused by small solid chunks of material having an appearance similar to coke. It was thought that this hard material came loose from the walls of process lines handling DAR from the CSD to LFU section. These process lines had been held dormant at 500°F for several weeks (during operation of Runs 33 and 34).

Integrated operation was re-established on 9 December. Following total integrated operation, two back-to-back 24-hour material balances were completed. Results of this combined MB are included in the Section 6. The process operating conditions for Run 35 are given in Table 5-1. Run 35 ended on 13 December, immediately after the Material Balances were completed.

The Daily Operating Log for this and other bench-scale runs is given in Table A-1, Appendix A and the on-stream times for the various bench-scale runs completed under this contract are summarized in Table A-2, Appendix A.

BENCH-SCALE RUN 36

Run 36 was the second run completed in this quarter using Kerr-McGee Process Configuration No. 6. This process configuration is same as used in the preceding run, Run 35. The only variation from Run 35 was that the intended split of LDAR as a fraction of the total CSD deashed product was altered. Instead of the 30 wt% of DAR intended to be removed as LDAR (CSD third-stage product) in Run 35, as much as 60 wt% of the DAR was intended to be removed as LDAR for hydrotreating prior to its use as liquefaction solvent in Run 36. However, as discussed earlier under Run 35 operation, 53 wt% of the total CSD product was actually separated as LDAR. Consequently, the intended split for Run 36 was not significantly different from Run 35.

The catalyst in hydrotreater at the end of Run 35 was retained for Run 36 in view of the relatively low catalyst age of 20.5 lbs-resid/lb-catalyst.

Run 36 was started on 14 December, immediately following Run 35. However, very little integration was achieved until 18 December. Most of the time was spent trying to establish steady-state operations at the program parameter conditions. Problems with plugging in the CSD letdown lines made it difficult to attain proper deashing and maintenance of required resid levels. In addition, high-ash DAR recycled from the CSD section to the LFU resulted in partial plugging and poor operability of the coal slurry feed pumps.

Attempts to obtain integrated operation continued through 21 December. However, it was not possible to attain steady-state operation due lack of proper deashing in the CSD first-stage and plugging of the letdown valves. Finally, Run 36 was discontinued without a material balance being made after it appeared that CSD first-stage vessel was possibly plugged. Table 5-2 summarizes the operating conditions during Run 36.

The CSD section was flushed with LDAR to insure a complete flush-out of the equipment before shutdown. After shutdown, the bottom of the CSD first stage was removed for inspection. Visual inspection showed that the upper portion of the vessel (above the level control) had about 1/8 inch of buildup. The lower portion (below the level control) had about 1/4 inch of buildup. There was no buildup in the letdown line. It had been nine months since the previous mechanical cleaning of the CSD first-stage.

BENCH-SCALE RUN 37

Run 37 was a planned duplicate of Run 32 using Kerr-McGee Process Configuration No. 6 (Figure 5-1). This run was made to determine if the high distillate yield achieved in Run 32 could be duplicated in a second run at a different time.

Because of the low catalyst age, the Shell 324M catalyst originally charged at the start of Run 35 was retained for Run 37. The cumulative catalyst age at the start of Run 37, from processing in Run 35 and 36, was only 35 lbs-resid/lb-catalyst.

Run 37 was started on 3 January with the establishment of solvent flows in the hydro-treater and CSD sections. Coal feed was started on 5 January. A number of minor problems, including line pluggage, prevented smooth operation from occurring in all sections for several days. The process-derived vacuum bottoms were very soluble in the deashing solvent and frequent adjustment of the deashing solvent composition was necessary in order to obtain proper deashing.

Integrated operation was established on 10 January. Smooth operation continued until 15 January when deashing problems developed because of continued increasing solubility of the vacuum bottoms. This problem was corrected by further adjustment in deashing solvent composition.

A major concern during this run was the low coal conversions. It appeared that coal conversions comparable to those obtained in Run 32 could be obtained only by either increasing the dissolver temperature from 825⁰F to 840⁰F or by decreasing the feed space rate in hydrotreater by approximately 22 % below the value used in Run 32.

Two 24-hour material balances were completed. After completion of the material balances, Run 37 was terminated on 6 February. The operating conditions for Run 37 are given in Table 5-3.

The daily operations log and the daily operating and analytical data for Runs 35, 36 and 37 are given in Appendix A, B and C, respectively. The percent on-stream time for individual sections as well as for the integrated unit are summarized in Table A-2, Appendix A. The daily catalyst age log for each of these runs is included in Table A-3, Appendix A.

TASK 3 RUNS

BENCH-SCALE RUN 38

Run 38 was the first bench-scale run completed under Task 3. In addition, the process conditions for Run 38 were selected so that this run could be used as a duplicate for Run 32. The iron oxide concentration was retained at 2 wt% (MF coal basis). The following runs, Runs 39 and 40, are to be made at 0 wt% and 4 wt% iron oxide concentration, respectively.

Run 38 was started on 10 February. Fresh Shell 324M catalyst was charged to the hydrotreater. Presulfiding of the catalyst using No. 2 fuel oil as solvent was completed on 11 February. After operating the hydrotreater for 29 hours on process solvent only, the hydrotreater feed was changed to include LDAR. While the presulfiding of catalyst was underway, the entire solvent inventory including process and flush solvent was processed through the unit for removal of the light fraction. As a result of this processing, the H/C atomic ratio of process solvent at the start of Run 38 decreased from 1.48 to 1.28.

Coal feed was started on 12 February. The LFU, CSD and HTR sections of the bench-scale unit operated in an integrated mode for the following seven days with a high (94%) on-time factor. Coal conversions were in the 90-92 wt% (MAF coal) range. Two 24-hour back-to-back material balances were made on 19 and 20 February. Run 38 ended following the completion of a second MB on 20 February. The operating conditions for Run 38 are given in Table 5-4.

BENCH-SCALE RUN 39

This was the second bench-scale run completed under Task 3 of this contract. Objective of this run was to establish a 'base-case' coal conversion for Wyoming subbituminous coal in absence of a disposable catalyst such as iron oxide.

Immediately following Run 38, process conditions were changed on 21 February to start Run 39. The coal feed was 'as-received' coal without iron oxide addition. Run 39 was primarily made as a 'base-case' run to establish the effect of iron oxide as a disposable catalyst on coal conversion and distillate yield under process configuration No. 6 (Figure 5-1).

Run 39 proceeded fairly smoothly to 26 February, except for minor CSD downtime because of a DAS solvent leak. The coal conversions as expected were appreciably lower and were primarily in the 86-87 wt% range.

A material balance was started at 2300 hours on 26 February but had to be discontinued after hydrotreater reactor temperature could not be maintained at the required level. The problem arose as a result of burned-out electrical heating elements in the hydrotreater preheater. In view of the consistently lower coal conversions obtained in absence of iron oxide, it was considered that the original goal of establishing a

'base-case' coal conversion value was achieved. Therefore, the decision was made to obtain and analyze special samples in lieu of an actual MB and proceed with the next run (Run 40) after repairs to the hydrotreater preheater electrical heaters were completed. The operating conditions for Run 39 are given in Table 5-5.

The daily operations log and the daily operating and analytical data for both of these runs (No. 38 and 39) are given in Appendix A, B and C, respectively. The percent on-stream time for individual sections as well as the integrated unit are summarized in Table A-2, Appendix A. The daily catalyst age log for each of the runs is also included in Table A-3, Appendix A.

KERR-McGEE PROCESS CONFIGURATION NO. 6

FIGURE 5-1

Table 5-1

Summary of Operating Conditions - Run 35

<u>Run Number</u>	<u>35</u>
Material balance no.	1 & 2
Date, 1983	Dec 12 & 13
Flowsheet number	6
<u>Coal Dissolver Conditions</u>	
Coal type	Wyoming Subbituminous (Clovis Point Mine)
MF coal rate, lb/hr	6.46
Temperature, °F	824
Pressure, psig	2350
MF coal space rate ¹ , lb/hr-ft ³	26.9
+850°F resid in solv., wt %	16.1
Ratio, liq.solv./MF coal	2.0

Hydrotreater Conditions

Temperature, °F	697
Pressure, psig	2290
Feed space rate, lb/hr-lb cat	1.1
+850°F resid in feed, wt %	11.2
Catalyst type	Shell 324M

¹ Based on net volume above 800°F

Table 5-2

Summary of Operating Conditions - Run 36

<u>Run Number</u>	<u>36</u>
Material balance no.	-
Date, 1983	Dec 17-19
Flowsheet number	6
<u>Coal Dissolver Conditions</u>	
Coal type	Wyoming Subbituminous (Clovis Point Mine)
MF coal rate, lb/hr	6.0
Temperature, °F	825
Pressure, psig	2360
MF coal space rate ¹ , lb/hr-ft ³	25.0
+850°F resid in solv., wt %	36.1
Ratio, liq.solv./MF coal	1.83

Hydrotreater Conditions

Temperature, °F	699
Pressure, psig	2380
Feed space rate, lb/hr-lb cat	1.05
+850°F resid in feed, wt %	17.7
Catalyst type	Shell 324M

¹ Based on net volume above 800°F

Table 5-3

Summary of Operating Conditions - Run 37

<u>Run Number</u>	<u>37</u>	
Material balance no.	1	2
Date, 1984	Jan 27	Feb 6
Flowsheet number	6	6
<u>Coal Dissolver Conditions</u>		
Coal type	Wyoming Subbituminous (Clovis Point Mine)	
MF coal rate, lb/hr	5.04	4.57
Temperature, °F	826	825
Pressure, psig	2340	2350
MF coal space rate ¹ , lb/hr-ft ³	14.8	13.4
+850°F resid in solv., wt %	16.8	15.1
Ratio, liq.solv./MF coal	1.91	1.89

Hydrotreater Conditions

Temperature, °F	700	702
Pressure, psig	2290	2160
Feed space rate, lb/hr-lb cat	0.86	0.90
+850°F resid in feed, wt %	13.9	14.3
Catalyst type	Shell 324M	

¹ Based on net volume above 800°F

Table 5-4

Summary of Operating Conditions - Run 38

<u>Run Number</u>	<u>38</u>
Material balance no.	1 & 2
Date, 1984	Feb 19 & 20
Flowsheet number	6
<u>Coal Dissolver Conditions</u>	
Coal type	Wyoming Subbituminous (Clovis Point Mine)
MF coal rate, lb/hr	7.43
Temperature, °F	824
Pressure, psig	2360
MF coal space rate ¹ , lb/hr-ft ³	21.86
+850°F resid in solv., wt %	25.9
Ratio, liq.solv./MF coal	1.76

Hydrotreater Conditions

Temperature, °F	700
Pressure, psig	2280
Feed space rate, lb/hr-lb cat	1.18
+850°F resid in feed, wt %	13.3
Catalyst type	Shell 324M

¹ Based on net volume above 800°F

Table 5-5
Summary of Operating Conditions - Run 39

<u>Run Number</u>	<u>39</u>
Material balance no.	1
Date, 1984	Feb 27
Flowsheet number	6
<u>Coal Dissolver Conditions</u>	
Coal type	Wyoming Subbituminous (Clovis Point Mine)
MF coal rate, lb/hr	7.82
Temperature, °F	824
Pressure, psig	2360
MF coal space rate ¹ , lb/hr-ft ³	23.0
+850°F resid in solv., wt %	24.45
Ratio, liq.solv./MF coal	1.94
<u>Hydrotreater Conditions</u>	
Temperature, °F	698
Pressure, psig	2260
Feed space rate, lb/hr-lb cat	1.08
+850°F resid in feed, wt %	15.15
Catalyst type	Shell 324M

¹ Based on net volume above 800°F

Section 6

BENCH SCALE RESULTS

TASK 2 RUNS

During the second quarter of this contract period, three Bench-Scale Runs (Nos 35, 36 and 37) were completed under Task 2 of this contract. Wyoming subbituminous coal (Clovis Point Mine), containing 2 wt% iron oxide (MF coal basis) as a disposable catalyst, was liquefied in all three runs using a two-stage liquefaction process scheme wherein the liquefaction solvent consisted of a mixture of DAR and hydrotreater underflow containing the H-LDAR. The overhead from the CSD first-stage was fractionated in the CSD second-stage to produce DAR which was recycled to the liquefaction section as a component of the liquefaction solvent. The balance of the resid was removed as Light Deashed Resid (LDAR) in the CSD third-stage and recycled to the hydrotreater along with vacuum gas oil from the liquefaction section to produce the hydrotreater underflow component of the liquefaction solvent.

All three runs utilized a common two-stage process configuration (Figure 5-1). In this process configuration LDAR is hydrogenated prior to its recycle to the thermal stage and DAR is recycled directly to the thermal stage without hydrotreating. The coal space velocity through the coal dissolver, however, were different in these three runs. In addition, the intended LDAR/DAR product split obtained in the CSD section was also different. The objective of this test period was to study the effect of increased coal space rate on coal conversion and overall yield. In addition, Run 37 was made primarily to determine if the high distillate yield achieved in Run 32 (completed during last quarter) could be duplicated in a second run at a different time.

RESULTS - RUN 35

Run 35 was similar to Run 32 except the coal space rate was increased from 19 to 26 lb/hr-cu ft. Furthermore, hydrotreater underflow was flashed at about 310°F to remove the lights and increase resid concentration in the liquefaction solvent. Two 24-hour material balances were made during Run 35. These two 24-hour balances were combined as one 48-hour balance. Results of combined MB along with MB's of other runs are given in Table 6-1 and 6-2.

Table 6-1 gives the overall two-stage yields whereas the yields for the individual stages, thermal and catalytic, are given in Table 6-2. Overall process results including coal conversion, resid conversions to distillate and gases plus water and CSD soluble-coal recovery are summarized in Table 6-3.

The increase in dissolver coal space rate from 19 (Run 32) to 26 lb/hr-cu ft for Run 35 resulted in lower coal conversions and reduced distillate yield. The calculated distillate (C₄ to -850°F) yield for Run 35 was 34.7 wt% (MAF coal) with a net deashed resid product yield of 7.8 wt%. The net hydrogen consumption was 4.5 wt% of MAF coal feed. Loss of coal values to the ash concentrate product was somewhat higher than in Run 32 and averaged 28.8 wt% of the MAF coal feed.

A comparison of the two-stage yields from Run 35 and 32 is given below,

Two-Stage Yields, wt % MAF Coal	Run 35	Run 32
	Avg(MB1 & 2)	Avg(MBs 2,4,5)
Hydrogen	<4.5>	<5.3>
Gases (H ₂ S, CO, CO ₂ , NH ₃)	10.5	8.6
Water	13.0	10.4
C ₁ -C ₃ hydrocarbons	9.7	13.2
C ₄ to -850°F liquids	34.7	54.4
+850°F resid	7.8	<2.7>
Coal values in ash conc	28.8	21.4
Net	100.0	100.0

In a truly balanced system with total distillate product slate, the resid product quantity is zero. Hence, the above results were recalculated using certain fixed parameters such as S/C ratio and the actual values of coal conversion, resid conversion to gases and water, coal value rejection to AC. Only the dependent variables, such as resid concentration in feed to dissolver and hydrotreater, were allowed to float for a resid balanced system. The summary of actual process parameters for Run 35 is given in Table 6-4 and those used in zero-resid MB calculation of Run 35 are given in Table 6-5. The changes made from actual values are also identified.

The results of actual and adjusted (zero-resid case) MB calculations are summarized below. The overall two-stage results show that Run 35 did not produce the same quantity of distillate as Run 32. It is obvious that among these two runs, Run 32 provided better distillate yield. Analyses of various product streams from Run 35 along with other runs are summarized in Tables 6-6 through 6-9. Based on the yields and product slate, it is concluded that the higher coal space rate used in Run 35 contributed to poorer results.

	Run 35 (MB 1 & 2)		Run 32 (MB 2,4,5)
	<u>Actual</u>	<u>Zero-Resid Adjusted</u>	<u>Zero-Resid Adjusted</u>
<u>Yields, wt % MAF Coal</u>			
<u>Thermal Stage</u>			
Hydrogen	<3.0>	<3.0>	<3.4>
CO,CO ₂ ,H ₂ S,NH ₃	10.0	10.2	8.8
Water	10.1	10.2	9.1
C ₁ -C ₃ hydrocarbons	9.3	9.5	13.5
C ₄ to -850°F liquids	37.7	40.7	45.2
+850°F resid	7.1	3.6	5.4
Coal values in ash conc	28.8	28.8	21.4
TOTAL	100.0	100.0	100.0
<u>Catalytic Stage</u>			
Hydrogen	<1.5>	<1.5>	<2.0>
CO,CO ₂ ,H ₂ S,NH ₃	0.5	0.7	0.3
Water	2.9	3.8	1.6
C ₁ -C ₃ hydrocarbons	0.4	0.6	0.4
C ₄ to -850°F liquids	<3.0>	0	5.1
+850°F resid	0.7	<3.6>	<5.4>
TOTAL	0.0	0.0	0.0
<u>Overall Two-Stage</u>			
Hydrogen	<4.5>	<4.5>	<5.4>
CO,CO ₂ ,H ₂ S,NH ₃	10.5	10.9	9.1
Water	13.0	14.0	10.7
C ₁ -C ₃ hydrocarbons	9.7	10.1	13.9
C ₄ to -850°F liquids	34.7	40.7	50.3
+850°F resid	7.8	0	0
Coal values in ash conc	28.8	28.8	21.4
TOTAL	100.0	100.0	100.0

Table 6-6 summarizes the composition changes across the liquefaction section for only the key process streams of incoming liquefaction solvent and outgoing vacuum oil product (spent solvent) and vacuum bottoms. The light oils and H₂ free vent-gas streams are the remaining two outgoing streams. But, these are generally very small and account for less than 10-15 wt % of the total mass. Hence, the performance of the thermal unit could be judged by comparison of the key process streams - liquefaction solvent, vacuum oil and vacuum bottoms. Analyses of Run 32 streams are included for comparison. As seen in Table 6-6, compared to Run 32, the resid concentration of liquefaction solvent in Run 35 was quite low at 16.1 wt%. Since liquefaction solvent consisted of DAR in this run, the low resid content will obviously affect the H/C

ratio of the liquefaction solvent and an appropriate allowance should be made for the reduced DAR content. However, the vacuum oil product from Run 35 also shows almost a step-change in its hydrogen content and the H/C atomic ratio. The hydrogen content and the H/C atomic ratios of the vacuum oil products for Runs 35 and 36 are comparable. However, these are higher than the values observed in Run 32. The exact cause of this increase in H/C ratio is not known and is possibly related to addition of hydrotreated solvent from Wilsonville to the process solvent inventory during and at the end of Runs 33 and 34 completed in the last quarter. Only near the end of Run 37 did the hydrogen content and H/C ratio of vacuum oil product and vacuum bottoms, approach values close to those in Run 32. Whether the lower coal conversions and distillate yields observed in Run 35 and 36 were a direct result of hydrogen content or H/C ratio is not known for certain, but will be investigated further in later runs.

The analyses and properties of various key streams from CSD section are summarized in Tables 6-7 and 6-8. Table 6-7 compares the differences in composition of vacuum bottoms obtained in the various runs. The viscosity of vacuum bottoms during Run 35 was 335 centipoise compared to an average of 177 centipoises in Run 32. In spite of higher H/C ratios, the preasphaltenes contents (on a soluble-coal basis) of vacuum bottoms from Runs 35 and 36 are higher than the average from Run 32 (Table 6-7). This perhaps is another indication of less effective coal dissolution during Runs 35 and 36. This is further supported by low coal conversion and high UC content of vacuum bottoms from those runs. During Run 35, the intended CSD product split of 30 wt% of the total resid being separated as LDAR (CSD third-stage product) was instead 53 wt%. Consequently, the CSD product characteristics and analyses for Run 35, shown in Table 6-8, do not show any major differences with products obtained in Run 36 which also had about the same LDAR/LDAR+DAR split at 50 wt% as LDAR. The preasphaltenes content of CSD-second stage DAR product is much lower in Run 35 than in Run 32. Since the preasphaltenes content was higher in the vacuum bottoms feed for Run 35 compared to Run 32, the difference in the preasphaltenes content in the DAR product is the result of greater rejection of preasphaltenes to the ash concentrate in Run 35.

On the basis of comparison of net changes across hydrotreater (Table 6-9), the hydrotreater section appears to have performed about the same during Runs 35 through 37 as in Run 32. The most notable differences are the higher H/C atomic ratios and lower resid concentration in the feeds in Runs 35 through 37 compared to Run 32.

RESULTS - RUN 36

Run 36 was completed using Kerr-McGee Process Configuration No. 6 (Figure 5-1), the same as used in Run 35. However, the split of Light DAR as a fraction of the total CSD products removed for hydrotreating was altered. As much as 60 wt % of the resid was intended to be removed as CSD third-stage product for hydrotreating prior to being used as a liquefaction solvent. Obviously, such a split would have meant that the LDAR in Run 36 should have been a little heavier than Run 35 DAR which had an intended LDAR to DAR split of 30:70.

Due to frequent plugging of CSD heavy-phase letdown lines, Run 36 was terminated without a material balance. Special samples of key process streams, in lieu of a material balance, were analyzed. These analyses, along with the analyses of samples from other Runs (32, 35 and 37), are summarized in Tables 6-6 through 6-9. These analyses show that there was no noticeable difference between Run 35 and 36. Compositions of liquefaction solvent, vacuum oil product and vac-bottoms were almost identical. So were the compositions of DAR and LDAR products. The hydrotreater feed and underflow streams were also almost identical in their composition as well as in their resid contents.

RESULTS - RUN 37

Run 37 was completed using Kerr-McGee Process Configuration No. 6 (Figure 5-1). Wyoming subbituminous coal was liquefied using 2 wt % (MF coal basis) iron oxide. Liquefaction solvent consisted of a mixture of hydrotreated Light Deashed Resid (H-LDAR) and DAR. This run, started on 3 January, was to be a duplicate of Run 32 and was made to determine if the high distillate yield achieved in Run 32 could be duplicated in a second run at a different time. On the basis of the results obtained, it was obvious that Run 37 results did not duplicate Run 32 results. There were several plausible reasons for why Run 32 results were not duplicated. In particular, the coal conversions were in the 85-87 wt% range in Run 37 compared to 91-93 wt% (MAF coal) achieved in Run 32. Various operating parameters were investigated as a possible cause for these low coal conversions. These included a wide range of process condition changes as well as equipment changes. The various changes investigated are summarized in Table 6-10. Higher dissolver temperature, lower space rates in dissolver and hydrotreater, increased iron content in the coal feed were tested to attempt to increase the coal conversions. Many of these changes were overlapped. As a result, it was difficult in some cases to ascertain the exact cause-effect relationship of these changes on coal conversions. The various operating parameter changes made during Run 37 and their resultant effect on daily coal conversion are shown in Figure 6-1.

The salt-bath preheaters (in liquefaction) were bypassed starting with Run 35. Both Run Nos. 35 and 36 exhibited low coal conversions in the 87-89 wt% range. However at that time, these low coal conversions were believed to be the result of increased dissolver coal space rate used for those runs. After experiencing consistently low conversions in the early part of Run 37, one salt-bath heater was re-installed in the liquefaction circuit on 17 January. There was no appreciable increase in the coal conversions. However, the coal conversions did finally increase to the 91-94 wt% range after several additional process changes were also made. The approximate coke-free volumes of the various preheater sections and the coal dissolver are listed below.

Unit	Type of Heat	Unit Volume ft ³	Volume of Preheater section as a % of Dissolver Volume %
Preheater A	Therminol	0.020	5.2
B	Dowtherm	0.035	9.0
C	Molten Salt	0.055	14.2
D	Molten Salt	0.055	14.2
New Design (Double pipe)	Dowtherm	0.013	3.4
Dissolver		0.387	

The flow arrangement for various runs along with the increase in volume (residence time) over and above dissolver with the inclusion of preheaters is summarized in Table 6-11.

It is evident that during Run 37, even after including one molten salt heated preheater, the total volume (including preheater D) did not quite approach the total volume used in Run 32. The preheaters have a very small cross section and the flow regime through preheaters is different and probably approaches a plug-flow pattern. It is possible that inadequate residence time in the appropriate flow regime may have been one of the prime variables affecting the coal conversions in recent runs. Of course, other process variables such as hydrogen donor quality of the solvent, and the thermal severity will also affect coal conversions. However, based on the total preheater volumes and the associated coal conversion in these runs, it appears that to get higher coal conversion it may be necessary to have a greater percentage of the total reactor volume in the plug flow regimes such as in preheaters.

The results of a batch reactor test made in parallel with the Bench-Scale Run 37 added credence to the above theory. This particular batch reactor test (84E12) was made

using a sample of coal slurry from the Bench-Scale Unit during Run 37. The particular coal slurry sample was obtained from Coal Slurry Tank (D-2103) on 12 January when coal conversion in the Bench-Scale Unit were about 86 wt %. This batch test, completed at conditions of 825⁰F, 50 minutes and 1400 psig (cold) hydrogen pressure, resulted in coal conversion of 96.7 wt%, again indicating that the liquefaction solvent quality was satisfactory. Results of this batch test are summarized below.

Test Number	84E12
Solvent/MF coal ratio	1.91
+850 ⁰ F conc in solvent, wt %	19.97
Coal conversion, wt % MAF coal	97.27
Resid conversion	
to gases and water, rel wt %	16.12
to distillate, rel wt %	23.96
Product Yield, wt % MAF coal	
Hydrogen	<1.91>
Gases (H ₂ S, CO, CO ₂ , NH ₃)	6.96
Water	11.50
C ₁ -C ₃ hydrocarbons	3.97
C ₄ to -850 ⁰ F liquids	35.25
+850 ⁰ F resid	40.03
Unconverted coal	4.20
Net	<hr/> 100.0

The thermal stage distillation yields obtained in bench-scale Run 37 (to be discussed later) are lower than the distillate yield obtained in this batch reactor test.

The dissolver agitator steady bearing configuration and its location were altered prior to the start of Run 35. This change was made with the expectation of reducing the bearing wear which indeed was observed. It was thought that the low conversions in Run 37 may have been the result of the changes in configuration and location of the agitator steady bearing. These changes may have resulted in a change in solids accumulation and gas hold-up in the dissolver. The old-style steady bearing may have increased hold-up of coal ash and iron oxide solids in the bottom section of the dissolver and thereby resulted in a beneficial (catalytic) effect on coal conversion. As a test of this solids hold-up theory, a short segment of increased iron oxide content (4 wt% MF coal basis) was conducted from 15-20 January. Although the coal conversions increased by about 3-4 rel % as a result of increased iron oxide concentration, they did not reach the 91-93 wt% range routinely obtained in Run 32 at 2 wt% iron oxide level. Moreover, as Run 37 progressed and other process variables changed, coal

conversions did eventually reach the 91-93 wt% range at 2 wt% (not 4 wt%) iron oxide level thus indicating that neither iron oxide level in coal feed by itself or the suspected change in solids or gas hold-up as a result of agitator bearing change, were responsible for the low conversions obtained earlier.

During Runs 32 through 34, a higher than normal gas yield containing increased quantities of C₁-C₅ hydrocarbons was obtained in the thermal stage. It was felt that these gases were possibly being generated in the dissolver by either cracking or reactions involving lighter materials (with higher H/C ratios) in feed to the dissolver. Hence in order to reduce the hydrocarbon gas make, starting with Run 35 the hydrotreater underflow was subjected to an atmospheric flash to reduce the lighter components of the liquefaction solvent stream. It was thought that this change may have caused the low conversion in Run 37. The atmospheric flashing of hydrotreater underflow was discontinued to see if the coal conversion would improve. However, this change by itself failed to show any noticeable improvements in coal conversion.

The iron analyses of coal feed for Run 37 was checked and compared with prior runs for its variation and was found to have only minor differences which could not be directly correlated to have any appreciable effect on coal conversion. The range of iron content as iron oxide (MF coal basis) for the various runs was as follows:

<u>Run No.</u>	<u>Range of Wt % Iron as Fe₂O₃(MF Coal Basis)</u>
32	2.4 - 2.6
33	2.3 - 3.1
34	2.2 - 2.7
35	2.2 - 2.6
36	2.2 - 2.4
37	2.4 - 2.8
	(3.8 - 4.9) during high oxide test period.

The composition and analyses of the iron oxide materials were also compared (Table C-3, Appendix C) but did not reveal any noticeable differences.

A decrease in solvent quality of the liquefaction solvent could also be a major cause for low coal conversions. Comparison of the vacuum-oil analyses (see Table 6-12) produced in liquefaction section and used as hydrotreater process solvent, showed a noticeable increase in the hydrogen content and the H/C atomic ratio for this material between Run 34 and 35. External solvent from Wilsonville was added at the start of

Run 35 to maintain the required system solvent level. It is believed that process solvent composition may have been significantly altered by the addition of hydro-treater solvent from Wilsonville. Hydrotreating of this solvent may have resulted in a lower quality donor solvent possibly due to over-hydrogenation. This may have, in turn, resulted in the lower coal conversions and distillate yields in Runs 35 and 36. Even during Run 37, the initial H/C atomic ratio of vacuum-oil samples was high (>1.4) and only towards the end of the run, as the coal conversion improved this ratio dropped to 1.25, the same range as observed in Runs 32 through 34.

In order to further evaluate this theory, additional microautoclave tests were made using the liquefaction solvent samples obtained from the bench-scale unit. These microautoclave tests results are summarized in Table 6-13. The test conditions in Sets 2 and 3 were intentionally varied to cause noticeable differences in microautoclave results and obtain data which was closer to the bench-scale observed values. All solvents, with the exception of the sample of 1 February, followed the expected pattern. The last two Sets of tests (No. 4 and 5) were intended to study possible degrading of reaction products in the bench-scale unit, after having attained initial high coal conversions. It was thought that the solvent quality may have been such that with initial high coal conversions the vacuum bottoms possibly being more reactive and unstable could retrograde in the bench-scale unit, and thereby resulting in higher UC content and show lower coal conversion. However, the results of the microautoclave tests show that the reaction products were very stable and did not retrograde at 750°F for 60 minutes even in absence of hydrogen atmosphere. All of these tests, did confirm that the above microautoclave tests could not be used to detect any major differences in the quality of liquefaction solvent and did result in high (92-94 wt%) coal conversion as is normally indicative of a good quality solvent. It is still likely that changes in the process solvent quality was the primary reason for the change in coal conversion even though it could not be detected using the microautoclave tests described here.

Following the first material balance on 27 January, the feed space rate in hydrotreater was increased from 0.72 to 1.09 lb/hr-lb catalyst while maintaining the MF coal space rate in the dissolver at 15 lb/hr-cu ft. This was done in an attempt to decouple these two variables in terms of their effect on coal conversion. After two days, the coal conversions decreased over a five-day period from 93 to 86 wt%. When the hydrotreater feed space rate was again decreased from 1.09 to 0.72 lb/hr-lb catalyst, the coal conversions increased back up to 92 wt%. These results were somewhat surprising in comparison to results obtained in the earlier runs and suggested that the catalyst might

be fouled. At the end of Run 37, the catalyst was removed. The recovered catalyst weight of 12.8 pounds corresponded closely to the quantity of catalyst removed in prior runs and indicated negligible loss of catalyst from the reactor. The spent catalyst was analyzed for elemental as well as pore volume and surface area determinations to see how it compared with spent catalyst from Run 32.

A comparison of the spent catalyst properties from Run 37 as well as other prior runs is given in Table 6-14. The volume pore size distribution for these spent catalysts is given in Figure 6-2. These results appear to indicate a little more severe pore blockage for Run 37 catalyst but the loss of available pore volume and surface area for Run 37 catalyst does not appear to be much different. Even the coke deposition and deposition of other heavy metals appears to be comparable to catalyst from Run 32, thus ruling out excessively fouled catalyst as a cause of poorer results in Run 37. There appears to be a somewhat larger difference in the sulfur content of the spent catalyst. Whether the 1.3 wt% difference in the sulfur content is appreciable with regard to catalyst activity is not known.

Since the coal conversions did improve at the lower space rate, it may be that bypassing was occurring in the hydrotreater. This would have resulted in a space rate that was effectively higher than calculated. This will be evaluated in a future run.

The results of Run 37, MB-1 are summarized below.

<u>Yields, wt % MAF Coal</u>	Run 37 MB-1		Run 32 MB Avg(2,4,5)
	<u>Actual</u>	<u>Zero-Resid Adjusted</u>	<u>Zero-Resid Adjusted</u>
<u>Thermal Stage</u>			
Hydrogen	<2.8>	<2.8>	<3.4>
CO, CO ₂ , H ₂ S, NH ₃	10.0	11.6	8.8
Water	7.8	9.0	9.1
C ₁ -C ₃ hydrocarbons	14.8	17.0	13.5
C ₄ to -850°F liquids	29.0	34.3	45.2
+850°F resid	19.0	8.7	5.4
Coal values in ash conc	22.2	22.2	21.4
TOTAL	100.0	100.0	100.0

<u>Yields, wt % MAF Coal</u>	Run 37 MB-1		Run 32 MB Avg(2,4,5)
	<u>Actual</u>	<u>Zero-Resid Adjusted</u>	<u>Zero-Resid Adjusted</u>
<u>Catalytic Stage</u>			
Hydrogen	<2.1>	<2.1>	<2.0>
CO,CO ₂ ,H ₂ S,NH ₃	0.4	0.8	0.3
Water	1.9	4.0	1.6
C ₁ -C ₃ hydrocarbons	0.3	0.7	0.4
C ₄ to -850 ⁰ F liquids	2.9	5.3	5.1
+850 ⁰ F resid	<3.4>	<8.7>	<5.4>
	<hr/>	<hr/>	<hr/>
TOTAL	0.0	0.0	0.0
<u>Overall Two-Stage</u>			
Hydrogen	<4.9>	<4.9>	<5.4>
CO,CO ₂ ,H ₂ S,NH ₃	10.4	12.4	9.1
Water	9.7	13.0	10.7
C ₁ -C ₃ hydrocarbons	15.1	17.7	13.9
C ₄ to -850 ⁰ F liquids	31.9	39.6	50.3
+850 ⁰ F resid	15.6	0.0	0
Coal values in ash conc	22.2	22.2	21.4
	<hr/>	<hr/>	<hr/>
TOTAL	100.0	100.0	100.0

The zero-resid adjusted results were obtained by recalculation of the MB under the constraints of S/C ratio of 1.8 and zero-resid product using the similar values of resid conversion as were actually observed in the run. The actual process parameters and those used in the estimated zero-resid product case are given in Tables 6-4 and 6-5. More gases and water and less distillate yield were observed in Run 37 than in Run 32.

In summary, Run 37 results did not completely duplicate Run 32 results. Coal conversions comparable to those obtained in Run 32 could be obtained by either increasing dissolver temperature from 825⁰F to 840⁰F or by decreasing the feed space rate in hydro-treater to 0.79 lbs/hr-lb catalyst which was approximately 22% below the feed space rate used in Run 32. On a zero-resid product basis, the overall yield of gas plus water was 43.1 wt% MAF coal which is about ten percent higher than in Run 32. Correspondingly, the distillate yield for Run 37 was 39.6 wt% which is about ten percent lower in comparison to 50.3 wt % (MAF coal) for Run 32. Evaluation of spent catalyst properties from Run 37 did not show any appreciable differences in catalyst deactivation which could be directly attributed to the low coal conversion and distillate yields. However, the high gas yield coupled with low distillate yield raised a strong probability that the high hydrogen content and H/C atomic ratio of solvents during Run 37 may be the

primary cause of these lower results. Further evaluation is required to establish with certainty which particular factors were responsible for the differences in results between Run 37 and Run 32.

TASK 3 RUNS

The primary objective of Task 3 Bench-Scale runs is to evaluate the use of disposable catalysts in the liquefaction of Wyoming subbituminous coal in the thermal stage to,

- a) decrease the coke precursor concentration in the resid produced,
- b) increase distillate yield, and
- c) improve product quality.

Preliminary development work including the work completed in Task 2 has already established that about 2 wt% iron oxide (MF coal basis) will increase the coal conversion of the subbituminous coal from 84-86 wt% range to 91-95 wt% range (pyridine-soluble coal, MAF basis). The plan in Task 3 is to test the use of iron oxide at several concentrations. For the three bench-scale runs planned in Task 3, only one process configuration was to be selected on the basis of Task 2 results and only one type of coal was to be used. The primary variable was to be the quantity of disposable catalyst to be added. In addition to the bench-scale runs, a batch reactor study was planned to test alternate iron oxide materials as disposable catalyst. The results of batch reactor tests are given in Section 7.

During the second quarter of this contract period, two bench-scale runs (Nos. 38 and 39) were completed. Wyoming subbituminous coal (Clovis Point mine) was liquefied using a two-stage process scheme (Figure 5-1) wherein the liquefaction solvent consisted of a mixture of DAR and hydrotreater underflow containing H-LDAR. Mobile iron oxide was used as the disposable catalyst.

RESULTS - RUN 38 AND 39

Run 38 was the first run and was made with 2 wt% iron oxide (MF coal basis) followed by Run 39 which was made with no disposable catalyst being added.

A very significant difference in coal conversion was noted as the iron oxide was removed from the coal feed. The average coal conversions decreased from 90-91 wt% for Run 38 to 85-86 wt% range for Run 39. This reduction of 4-5 wt% in coal conversion is

considered to be a direct result of not using any iron oxide in Run 39, since no other process parameters were changed. The actual daily coal conversions for the two runs were as follows:

<u>Date</u>	<u>Coal Conv., wt% MAF Coal</u>	
	<u>Run 38</u>	<u>Run 39</u>
Feb 13	80.9	
14	87.1	
15	90.1	
16	90.0	
17	91.8	
18	89.8	
19	92.3	
20	91.1	
21		92.6
22		86.5
23		86.2
24		87.0
25		85.4
26		87.0
27		86.1

The low conversion of 80.9 wt% and 87.1 wt% observed on 13 and 14 February are typical of the start-up operation. Similarly, the high conversion of 92.6 wt% observed on 21 February is typical of the time-delay effect and is a result of iron oxide still in the process system.

Two 24-hour back-to-back material balances were completed in Run 38 which were combined into a single 48-hour material balance for calculations. The results of this material balance are discussed below. Run 39 was completed without an actual material balance. However, special samples were taken to permit a simulated material balance to be made. Results of this material balance are also discussed below.

The product yields for the individual and two-stage unit for Runs 38 and 39 are given below. Except for the use of disposable catalyst in Run 38, the operating conditions for Run 39 were very similar to Run 38 (see Tables 5-4 and 5-5).

<u>Yields, wt % MAF Coal</u>	<u>Run 39</u>	<u>Run 38</u>
<u>Thermal Stage</u>		
Hydrogen	<1.4>	<3.0>
CO, CO ₂ , H ₂ S, NH ₃	12.6	12.9
Water	11.8	7.9
C ₁ -C ₃ hydrocarbons	15.4	13.4
C ₄ to -850°F liquids	16.2	25.5
+850°F resid	12.6	18.5
Coal values in ash conc	32.8	24.8
Total	<u>100.0</u>	<u>100.0</u>

<u>Catalytic Stage</u>	<u>Run 39</u>	<u>Run 38</u>
Hydrogen	<3.1>	<2.9>
CO, CO ₂ , H ₂ S, NH ₃	0.6	0.4
Water	1.4	1.8
C ₁ -C ₃ hydrocarbons	0.3	1.0
C ₄ to -850 ⁰ F liquids	3.7	4.0
+850 ⁰ F resid	<2.9>	<4.3>
Total	0.0	0.0
<u>Overall Two-Stage</u>		
Hydrogen	<4.5>	<5.9>
CO, CO ₂ , H ₂ S, NH ₃	13.2	13.3
Water	13.2	9.7
C ₁ -C ₃ hydrocarbons	15.7	14.4
C ₄ to -850 ⁰ F liquids	19.9	29.5
+850 ⁰ F resid	9.7	14.2
Coal values in ash conc	32.8	24.8
Total	100.0	100.0

The distillate yield for Run 39 was quite low (19.9 wt% MAF coal) and significantly lower than Run 38 (29.5 wt% MAF coal). It is believed that this yield was low because of the following reasons.

- 1) The rejection of the soluble-coal to the ash concentrate was quite high at 32.8 wt% (MAF coal). This is 10 wt% higher than for Run 38.
- 2) The hydrogen consumption in the dissolver for Run 39 was very low (1.3 wt%) and the conversion of the resid to distillate in the dissolver was also low.

A zero-resid adjusted material balance was also calculated for Run 38 and 39. The actual process parameters are given in Table 6-4 and the process parameters used for the zero-resid balance are given in Table 6-5. For this balance, the liquefaction solvent to MF coal ratio was changed from the actual value to 1.80; the liquefaction resid conversion to distillate was calculated based on the results of the zero-resid material balance; and the wt% resid in the liquefaction solvent and the hydrotreater feed were also calculated based on the results of the zero-resid material balance.

The yields for this zero-resid case for Run 38 and 39 are given below. These results show that for base-case run without iron oxide in a resid balanced system, the distillate yield was 27.6 wt% (MAF coal basis). The distillate yield increased by almost 13 wt% with the use of 2 wt% iron oxide. Also, the coal conversions improved from 86 to 90 wt% range as discussed earlier.

	<u>Run 39</u> <u>Zero-Resid</u> <u>Adjusted</u>	<u>Run 38</u> <u>Zero-Resid</u> <u>Adjusted</u>
<u>Yields, wt % MAF Coal</u>		
<u>Thermal Stage</u>		
Hydrogen	<1.4>	<3.0>
CO, CO ₂ , H ₂ S, NH ₃	13.3	13.9
Water	12.5	8.4
C ₁ -C ₃ hydrocarbons	15.8	14.4
C ₄ to -850°F liquids	23.5	37.1
+850°F resid	3.5	4.4
Coal values in ash conc	<u>32.8</u>	<u>24.8</u>
Total	100.0	100.0
<u>Catalytic Stage</u>		
Hydrogen	<3.1>	<2.9>
CO, CO ₂ , H ₂ S, NH ₃	0.6	0.4
Water	1.6	1.8
C ₁ -C ₃ hydrocarbons	0.3	1.1
C ₄ to -850°F liquids	4.1	4.0
+850°F resid	<u><3.5></u>	<u><4.4></u>
Total	0.0	0.0
<u>Overall Two-Stage</u>		
Hydrogen	<4.5>	<5.9>
CO, CO ₂ , H ₂ S, NH ₃	13.9	14.3
Water	14.1	10.2
C ₁ -C ₃ hydrocarbons	16.1	15.5
C ₄ to -850°F liquids	27.6	41.1
+850°F resid	0.0	0.0
Coal values in ash conc	<u>32.8</u>	<u>24.8</u>
Total	100.0	100.0

Figure 6-1

SUMMARY OF OPERATING PARAMETER CHANGES AND COAL CONVERSION - RUN 37

Curve No.	Catalyst Type	Pore Volume, cm ³ /gram	Catalyst Age lbs-resid/lb-cat	Resid Feed Type
1	Fresh	0.374	-	-
2	Spent-Run 32	0.235	187	LDAR
3	Spent-Run 33	0.236	105	FR-DAR
4	Spent-Run 34	0.258	51	DAR
5	Spent-Run 20	0.168	225	FR-DAR
6	Spent-Run 29	0.138	517	LDAR & FR-DAR
7	Spent-Runs 35-37	0.227	117	LDAR

Figure 6-2. Volume Pore Size Distribution in Shell 324M Catalyst (1/32" extrudate) when used in Continuous Hydrotreatment of Full-Range Deashed Resid, Light Deashed Resid and Deashed Resid.

Table 6-1
BENCH-SCALE RUNS
OVERALL TWO-STAGE YIELDS

<u>Run Number</u>	35	37	38	39
Material Balance No.	1&2	1	1&2	1
<u>Wt %, MAF Coal Equivalent</u>				
Hydrogen	<4.5>	<4.9>	<5.9>	<4.5>
Gases (H ₂ S, CO, CO ₂ , H ₂)	10.5	10.4	13.3	13.2
Water	13.0	9.7	9.7	13.2
C ₁ -C ₃ hydrocarbons	9.7	15.1	14.4	15.7
C ₄ to -850°F liquids	34.7	31.9	29.5	19.9
+850°F resid	7.8	15.6	14.2	9.7
Coal values in ash conc	28.8	22.2	24.8	32.8
Net	100.0	100.0	100.0	100.0

Table 6-2
BENCH-SCALE RUNS
THERMAL AND CATALYTIC STAGE YIELDS

<u>Run Number</u>	35	37	38	39
Material Balance No.	1&2	1	1&2	1
<u>Thermal Stage Yields, wt% MAF Coal</u>				
Liquefaction plus CSD				
Gases (CO,CO ₂ ,H ₂ S,NH ₃)	10.0	10.0	12.9	12.6
Water	10.1	7.8	7.9	11.7
C ₁ -C ₃ hydrocarbons	9.3	14.8	13.4	15.4
C ₄ to -850°F liquids	37.7	29.1	25.5	16.2
+850°F resid	7.1	18.9	18.5	12.6
Coal values in ash conc	28.8	22.2	24.8	32.8
Sub-Total	103.0	102.8	103.0	101.3
<u>Catalytic Stage Yields, wt% MAF Coal</u>				
Hydrotreater Unit				
Gases (CO,CO ₂ ,H ₂ S,NH ₃)	0.5	0.4	0.4	0.6
Water	2.9	1.9	1.8	1.5
C ₁ -C ₃ hydrocarbons	0.4	0.3	1.0	0.3
C ₄ to -850°F liquids	<3.0>	2.8	4.0	3.7
+850°F resid	0.7	<3.3>	<4.3>	<2.9>
Sub-Total	1.5	2.1	2.9	3.2
Total Yield, wt% MAF Coal	104.5	104.9	105.9	104.5
Less Net Total Input	4.5	4.9	5.9	4.5
NET	100.0	100.0	100.0	100.0

Table 6-3
BENCH-SCALE RUNS
SUMMARY OF OVERALL PROCESS RESULTS

Run Number	32	35	36	37	38	39
Process Configuration	6	6	6	6	6	6
Material Balance No.	2,4,5	1&2	-	1	1&2	1
LIQUEFACTION SECTION						
Coal conversion, wt% MAF Coal	92.1	89.0	82.4	93.5	92.1	86.8
Resid conversion ¹ , wt %						
to gases and water ²	18.4	20.4	-	25.6	23.9	28.7
to distillate ³	30.7	27.0	-	23.1	18.4	12.4
CSD SECTION						
Soluble-coal recovery, wt%	87.1	81.8	81.4	80.4	81.2	79.4
Coal value rejection to ash conc, wt % MAF coal	21.4	28.8	-	21.4	24.8	32.8
HYDROTREATER SECTION						
Resid conversion ¹ , wt %						
to gases and water ²	6.4	16.4	-	13.3	12.3	8.2
to distillate ³	13.4	<14.8>	-	12.8	15.0	13.1

¹ - Based on total resid. Includes pyridine-soluble coal and resid in solvent.

² - Includes heterogases as well as C₁-C₃ hydrocarbons and water.

³ - C₄ to -850°F fraction.

Table 6-4

SUMMARY OF PROCESS RESULTS FOR DIFFERENT RUNS

A. Run No.	32	35	37	38	39
E. LIQUEFACTION UNIT					
1. Iron oxide in coal, wt % MF coal	2.4	2.0	2.8	2.0	0.0
2. Coal conversion, wt % MAF coal	92.1	89.0	93.5	92.1	86.8
3. Hydrogen consumption, wt % MAF coal	3.4	3.0	2.8	3.0	1.3
4. Hetro. gases/H ₂ O/C ₁ -C ₃	0.28/0.29/0.43	0.34/0.34/0.32	0.31/0.24/0.45	0.38/0.23/0.39	0.32/0.30/0.38
5. Liquefaction solvent/MF coal ratio	2.04	2.0	1.91	1.76	1.94
6. Resid in liq. solvent, wt %	45.9	16.1	16.8	25.9	24.5
7. Resid conversion, wt % ¹					
a. to gases and water	18.4	20.4	25.6	23.9	28.6
b. to distillate	<u>30.7</u>	<u>27.0</u>	<u>23.1</u>	<u>18.4</u>	<u>12.4</u>
TOTAL	49.1	47.4	48.7	42.3	41.0
8. -850°F distillables in vacuum bottoms, wt%	6.9	4.0	2.6	6.7	12.3
C. CSD UNIT					
1. MAF coal rejection to ash concentrate, wt%	21.4	28.8	22.2	24.8	32.8
2. Soluble-coal recovery in CSD, wt %	87.1	78.2	80.4	81.2	79.4
3. -850°F distillables in ash concentrate, wt%	4.2	2.8	0.6	1.9	2.3
4. Split of LDAR/DAR + LDAR	0.47	0.53	0.68	0.44	0.53
5. Split of -850°F distillables LDAR/DAR + LDAR	0.73	0.79	0.95	0.76	0.64
D. HYDROTREATER					
1. Hydrogen consumption	2.0	1.5	2.1	2.9	3.1
2. Hetro. gases/H ₂ O/C ₁ -C ₃	0.14/0.69/0.17	0.14/0.74/0.12	0.15/0.73/0.12	0.13/0.56/0.31	0.26/0.63/0.11
3. Resid in feed, wt %	32.3	11.2	14.1	13.3	15.2
4. Resid conversion, wt %					
a. to gases and water	6.2	16.4	13.3	12.3	8.2
b. to distillate	<u>13.4</u>	<u><14.9></u>	<u>12.8</u>	<u>15.0</u>	<u>13.1</u>
TOTAL	19.6	1.5	26.1	27.3	21.3

¹ - Based on total resid. Includes pyridine-soluble coal and resid in solvent.

Table 6-5.
PROCESS PARAMETERS USED FOR MATERIAL BALANCES
WITH ZERO-RESID PRODUCTION²

A. Run No.	32	35	37	38	39
B. <u>LIQUEFACTION UNIT</u>					
1. Iron oxide in coal, wt % MF coal	2.4	2.0	2.8	2.0	0
2. Coal conversion, wt % MAF coal	92.1	89.0	93.5	92.1	86.8
3. Hydrogen consumption, wt % MAF coal	3.4	3.0	2.8	3.0	1.3
4. Hetro. gases/H ₂ O/C ₁ -C ₃	0.29/0.29/0.43	0.34/0.34/0.32	0.31/0.24/0.45	0.38/0.23/0.39	0.32/0.30/0.38
5. Liquefaction solvent/MF coal ratio	<u>1.80</u>	<u>1.80</u>	<u>1.80</u>	<u>1.80</u>	<u>1.80</u>
6. Resid in liq. solvent, wt %	<u>39.5</u> ³	<u>28.7</u> ³	<u>25.6</u> ³	<u>29.6</u> ³	<u>29.0</u> ³
7. Resid conversion ¹ , wt %					
a. to gases and water	18.4	20.4	25.6	23.9	28.6
b. to distillate	<u>26.5</u>	<u>28.6</u>	<u>23.5</u>	<u>24.2</u>	<u>16.9</u>
TOTAL	<u>44.9</u>	<u>49.0</u>	<u>49.1</u>	<u>48.1</u>	<u>45.5</u>
8. -850°F distillables in vacuum bottoms, wt%	6.9	4.0	2.6	6.7	12.3
C. <u>CSD UNIT</u>					
1. MAF coal rejection to ash concentrate, wt%	21.4	28.8	22.2	24.8	32.8
2. Soluble-coal recovery in CSD, wt %	87.1	78.2	80.4	81.2	79.4
3. -850°F distillables in ash concentrate, wt%	4.2	2.8	0.6	1.9	2.3
4. Split of LDAR/DAR + LDAR	0.47	0.53	0.68	0.44	0.53
5. Split of -850°F distillables LDAR/DAR + LDAR	0.73	0.79	0.95	0.76	0.64
D. <u>HYDROTREATER</u>					
1. Hydrogen consumption	2.0	1.5	2.1	2.9	3.1
2. Hetro. gases/H ₂ O/C ₁ -C ₃	0.14/0.69/0.17	0.14/0.74/0.12	0.15/0.73/0.12	0.13/0.56/0.31	0.26/0.63/0.11
3. Resid in feed, wt %	<u>24.1</u> ³	<u>18.2</u> ³	<u>21.2</u> ³	<u>15.7</u> ³	<u>18.5</u> ³
4. Resid conversion ¹ , wt %					
a. to gases and water	6.2	16.4	13.3	12.3	8.2
b. to distillate	<u>13.4</u>	<u>0</u>	<u>12.8</u>	<u>15.0</u>	<u>13.1</u>
TOTAL	19.6	<u>16.4</u>	26.1	27.3	21.3

¹- Based on total resid. Includes pyridine-soluble coal and resid in solvent.

²- Values in boxes are only parameters changed from actual material balance process results.

³- Result of other parameters.

Table 6-6
BENCH-SCALE RUNS
COMPARISON OF THE COMPOSITION OF LIQUEFACTION STREAMS
IN DIFFERENT RUNS

CLOVIS POINT COAL
PROCESS CONFIGURATION NO. 6

Run Number	32	35	36	37
Avgerate of MB No.	2,4,5	1&2	12/17-19/83	1
<u>Liquefaction Solvent</u>				
Elemental Analysis, wt%				
Hydrogen	8.28	10.04	10.04	10.08
Carbon	89.55	89.05	88.50	88.80
Nitrogen	0.52	0.29	0.27	0.21
Sulfur	0.04	0.03	0.06	0.01
Oxygen (by diff)	1.61	0.59	1.13	0.90
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	1.10	1.37	1.35	1.36
+850°F Resid, wt%	46.2	16.1	20.3	19.9
<u>Vacuum Oil Product (Process Solvent)</u>				
Elemental Analysis, wt %				
Hydrogen	9.22	10.38	10.41	9.36
Carbon	88.53	87.94	88.50	89.20
Nitrogen	0.37	0.22	0.19	0.27
Sulfur	0.01	0.02	0.08	0.01
Oxygen (by diff)	1.87	1.44	0.82	1.16
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	1.24	1.41	1.40	1.26
+850°F Resid, wt %	5.2	5.3	5.3	6.1
<u>Vacuum Bottoms (Ash-Free)</u>				
Elemental Analysis, wt %				
Hydrogen	5.97	6.17	6.32	5.68
Carbon	89.90	88.46	88.48	89.66
Nitrogen	1.21	1.15	1.17	1.06
Sulfur	0.74	0.95	1.03	1.90
Oxygen (by diff)	2.18	3.27	3.00	1.70
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	0.79	0.83	0.85	0.76

Table 6-7
BENCH-SCALE RUNS
COMPARISON OF THE COMPOSITION OF VACUUM BOTTOMS IN DIFFERENT RUNS
CLOVIS POINT COAL
PROCESS CONFIGURATION NO. 6

Run Number	32	35	36	37
Average of MB No.	2,4,5	1&2	12/17-19/83	1
Pyridine-Soluble, wt%	85.8	77.2	75.0	72.4
Unconverted Coal, wt%	6.2	11.4	12.7	7.9
Ash, wt %	8.0	11.4	12.3	19.7
TOTAL	100.0	100.0	100.0	100.0
-850°F Distillable, wt%	6.4	3.6	6.2	2.4
Viscosity at 500°F, cp	177	355	390	875
Composition of Soluble-coal Fraction, wt%				
Oils	15.6	13.1	12.5	7.9
Asphaltenes	67.5	65.7	67.8	69.2
Preasphaltenes	16.9	21.2	19.7	22.9
TOTAL	100.0	100.0	100.0	100.0
Elemental Analysis, wt % (ash-free basis)				
Hydrogen	5.97	6.17	6.32	5.68
Carbon	89.90	88.46	88.48	89.66
Nitrogen	1.21	1.15	1.17	1.06
Sulfur	0.74	0.95	1.03	1.90
Oxygen (by diff)	2.18	3.27	3.00	1.70
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	0.79	0.83	0.85	0.76

Table 6-8

BENCH-SCALE RUNS
COMPARISON OF THE COMPOSITION OF CSD PRODUCTS IN DIFFERENT RUNS
CLOVIS POINT COAL

PROCESS CONFIGURATION NO. 6

Run Number	32	35	36	37
Average of MB No.	2,4,5	1&2	12/17-19/83	1
<u>Second Stage DAR Product</u>				
Elemental Analysis, wt %				
Hydrogen	5.78	6.07	6.11	5.34
Carbon	90.37	89.71	89.93	91.24
Nitrogen	1.29	1.16	1.13	1.09
Sulfur	0.20	0.32	0.27	0.12
Oxygen (by diff)	2.36	2.74	2.56	2.21
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	0.76	0.81	0.81	0.70
-850°F Distillable, wt%	4.2	3.1	4.0	1.4
Viscosity at 500°F, cp	718	510	-	1,365
Composition of Soluble-coal, wt %				
Oils	5.6	5.6	6.9	3.4
Asphaltenes	63.1	77.9	78.3	59.8
Preasphaltenes	31.3	16.5	14.8	36.8
TOTAL	100.0	100.0	100.0	100.0
<u>Third Stage LDAR Product</u>				
Elemental Analysis, wt %				
Hydrogen	6.65	7.17	7.10	6.29
Carbon	90.07	89.59	89.65	90.83
Nitrogen	0.99	0.84	0.79	0.77
Sulfur	0.16	0.18	0.16	0.10
Oxygen (by diff)	2.13	2.22	2.30	2.01
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	0.88	0.95	0.94	0.83
-850°F Distillable, wt %	12.3	11.4	15.1	9.4
Viscosity at 350°F, cp	-	63	-	135
400°F, cp	55	-	-	-

Table 6-9
BENCH-SCALE RUNS
COMPARISON OF THE COMPOSITION OF HYDROTREATER STREAMS IN DIFFERENT RUNS
CLOVIS POINT COAL
PROCESS CONFIGURATION NO. 6

Run No.	32	35	36	37
Average of MB No.	2,4,5	1&2	12/17-19/83	1
Average Feed Space Rate, lb/hr lb-cat	1.04	1.12	1.14	0.86
<u>Hydrotreater Feed</u>				
Elemental Analysis, wt %				
Hydrogen	8.43	10.00	10.27	9.02
Carbon	88.96	88.16	88.57	89.10
Nitrogen	0.55	0.29	0.24	0.33
Sulfur	0.07	0.06	0.10	0.03
Oxygen (by diff)	1.99	1.49	0.82	1.52
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	1.13	1.35	1.38	1.21
+850°F Resid, wt %	32.5	13.9	13.3	13.9
<u>Hydrotreater Underflow</u>				
Elemental Analysis, wt %				
Hydrogen	9.49	10.95	11.16	10.14
Carbon	88.90	88.64	88.70	89.50
Nitrogen	0.25	0.14	0.05	0.10
Sulfur	<0.01	0.18	0.03	0.02
Oxygen (by diff)	1.35	0.09	0.06	0.24
TOTAL	100.00	100.00	100.00	100.00
H/C Atomic Ratio	1.26	1.47	1.50	1.36
+850°F Resid, wt %	28.2	12.8	11.5	12.6
<u>Net Change Across Hydrotreater</u>				
Elemental Analysis, wt %				
Hydrogen	1.06	0.95	0.89	1.12
Carbon	<0.06>	0.48	0.13	0.40
Nitrogen	<0.30>	<0.15>	<0.19>	<0.23>
Sulfur	<0.06>	0.12	<0.07>	<0.01>
Oxygen (by diff)	<0.64>	<1.40>	<0.76>	<1.28>
TOTAL	0.00	0.00	0.00	0.00
H/C Atomic Ratio	0.13	0.12	0.12	0.15
+850°F Resid, wt %	<4.3>	<1.1>	<1.8>	<1.3>

Table 6-10

LIST OF OPERATING PARAMETERS INVESTIGATED THAT COULD
POSSIBLY AFFECT COAL CONVERSIONS IN RUN 37

Equipment Changes

LFU preheater arrangement and design

Dissolver steady-bearing and mixer-
design and location

Process Conditions

HTU underflow flashing

LFU temperature - preheater

Dissolver temperature

Iron oxide level in coal

Solvent quality

Catalyst variation with batch

Catalyst deactivation level

Hydrotreater space rate (WHSV)

Table 6-11

COMPARISON OF PREHEATER PLUS DISSOLVER VOLUMES FOR DIFFERENT RUNS

Run No.	Preheater to Dissolver Flow Route	Increased volume As a percent of full Dissolver Volume, %			Coal Conversions wt % MAF Coal	
		above 500°F	above 600°F	above 700°F	Range	Avg
32 ¹	I → A → B → C → D → Full Dissolver	42.6	37.4	28.4	90.3-95.9	93.1
	II → A → B → C → → Full Dissolver	23.4	23.2	14.2	90.9-93.2	92.0
33	→ A → B → C → Full Dissolver	23.4	23.2	14.2	91.6-95.5	93.9
34	→ A → B → C → Full Dissolver	23.4	23.2	14.2	90.0-95.8	93.5
35 ²	→ A → B → C → Partial Dissolver (0.29 ft ³)	3.4	<1.8>	<10.9>	85.2-91.5	87.8
36	→ A → B → C → Partial Dissolver (0.29 ft ³)	3.4	<1.8>	<10.9>	87.3-91.1	88.4
37	I → New → → → Full Dissolver	3.4	<3.4>	0	81.9-87.6	85.6
	II ³ → New → D → → Full Dissolver	17.6	17.6	14.2	85.5-94.2 ⁴	91.9 ⁴

NOTES:

- ¹ Preheater D was by-passed on 8 October after 30 days into the run.
- ² Dissolver agitator steady bearing design and location changed at the start of Run 35.
- ³ Preheater D included in the circuit on 16 January till the end of run.
- ⁴ Result of several other parameter changes including, dissolver temperature, reactor space rates and iron oxide concentration.

Table 6-12

VACUUM OIL ANALYSIS SUMMARY
(USED FOR HYDROTREATER FEED)

Run No.	32			33			34		
	Date	H/C	H	Date	H/C	H	Date	H/C	H
	9/20/83	1.43	10.57	10/27/83	1.32	9.78	11/16/83	1.31	9.72
	9/27/83	1.28	9.47	10/31/83	1.26	9.34	11/18/83	1.26	9.30
	10/4/83	1.18	8.78	11/2/83	1.23	9.14	11/20/83	1.27	9.39
	10/10/83	1.26	9.36	11/4/83	1.21	9.04	11/21/83	1.26	9.30
	10/12/83	1.26	9.42	11/6/83	1.23	9.18			
	10/15/83	1.20	8.88	11/8/83	1.26	9.37			
	10/19/83	1.25	9.20						
	10/20/83	1.17	8.77						
	Avg.	1.25	9.31		1.25	9.31		1.28	9.43

Run No.	35			36			37		
	Date	H/C	H	Date	H/C	H	Date	H/C	H
	12/1/83	1.43	10.58	12/15/83	1.40	10.42	1/5/84	1.48	11.01
	12/12/83	1.38	10.15	12/18/83	1.40	10.40	1/9/84	1.42	10.52
	12/13/83	1.41	10.40				1/12/84	1.40	10.34
							1/16/84	1.30	9.67
							1/19/84	1.36	10.05
							1/26/84	1.26	9.40
							1/27/84	1.25	9.30
							2/6/84	1.25	9.31
	Avg.	1.41	10.38		1.40	10.41		1.34	9.95

Table 6-13

RESULTS OF EVALUATION OF RUN 37 LIQUEFACTION SOLVENT

<u>Date</u>	Bench-Scale Unit MAF Coal Conversions ² wt % Pyridine-soluble	MICROAUTOCLAVE RESULTS ¹				
		MAF Coal Conv. wt % THF Soluble		<u>Set #3</u>	<u>Set #4</u>	<u>Set #5</u>
		<u>Set #1</u>	<u>Set #2</u>			
1/13/84	84.2	93.5	88.3	74.5	93.2	-
1/14/84	86.3	92.7	89.7	78.0	-	94.1
1/25/84	92.9	94.0	91.5	82.9	94.0	-
1/26/84	93.8	94.4	90.5	81.4	-	93.2
2/1/84	85.8	94.7	91.2	83.0	94.6	-
2/2/84	88.8	93.6	88.3	76.0	-	94.5
2/5/84	93.6	92.2	89.9	80.1	92.7	-
2/6/84	-	94.1	84.5	84.5	-	94.2
	$\bar{x}$	93.7	90.0	80.1	93.6	94.0
	σ	0.8	1.1	3.4	0.7	0.5

¹ - Tubing-Bomb Conditions

	<u>Set #1</u>	<u>Set #2</u>	<u>Set #3</u>	<u>Set #4</u>	<u>Set #5</u>
Temperature, °F	825	800	800	825	825
Time, min	30	30	15	30	30
H ₂ pressure (cold), psig	1,400	1,400	1,400	1,400	1,400
Fe ₂ O ₃ , wt % MF coal	2.0	0	0	2.0	2.0
Post-treatment					
Temperature, °F	-	-	-	700	750
Time, min	-	-	-	60	60
H ₂ pressure (cold), psig	-	-	-	0	0

² - One day later than date given to more nearly match period when liquefaction solvent was used.

Table 6-14

SUMMARY OF SPENT SHELL 324M CATALYST ANALYSES

Catalyst	Fresh ¹	Spent Run 32	Spent Run 37	Spent Run 33	Spent Run 34	Spent Run 20	Spent Run 29
Resid type processed		LDAR	LDAR	FR-DAR	DAR	FR-DAR	FR-DAR & LDAR
Age, lbs-resid/lb-catalyst	0	187	117	105	51	226	517
Surface area ² , M ² /g	146	116	107.7	117	129	93	89
Pore volume @60,000 psi ² , cm ³ /g	0.374	0.235	0.227	0.236	0.258	0.168	0.138
Bulk density of dried catalyst, g/cm ³	0.784	1.000	0.990	0.950	0.964	1.026	1.050
Apparent density of dried catalyst, g/cm ³	3.48	2.89	2.83	2.58	2.67	2.70	2.64
Carbon, wt %	-	8.9	10.16	10.5	9.82	9.40	14.9
Hydrogen, wt %	-	1.14	1.40	1.17	1.24	1.21	1.68
H/C atomic ratio	-	1.54	1.65	1.34	1.52	1.54	1.35
Sulfur, wt %	-	8.32	7.02	7.04	6.93	7.27	6.87
Iron, wt %	0.013	0.33	0.28	0.085	0.071	0.47	0.34
Silicon, wt %	0.077	0.19	0.16	0.067	0.051	0.11	0.87
Titanium, wt %	0.037	0.21	0.076	0.046	0.038	0.47	0.35

¹ - As-produced catalyst in the oxide form. Catalyst was presulfided for each of the bench-scale runs.

² - Based on mercury porosimeter test.

Section 7

BATCH REACTOR TEST PROGRAM

BATCH REACTOR TESTS - TASK 2

COKE PRECURSOR STUDIES

The objective of the batch tests completed under Task 2 of this contact is to evaluate different coal-derived resids and catalysts in terms of catalyst deactivation. The data obtained from such batch tests identify the preferred resids and catalysts for testing in continuous units.

Tests with several resids at varying catalyst age were completed during this quarter. The detailed information concerning the experimental set-up and analytical procedures used for the coke-precursor batch reactor tests are covered in the February 84 quarterly report. The analyses of various raw materials used in these tests are given in Appendix D.

Effect of Resid Type

In earlier tests, reported in February 84 quarterly report, three different resid types (H-LDAR, LDAR and FR-DAR) were evaluated in a single-cycle test. During this quarter, this testing was extended to include three-cycle and a five-cycle tests using LDAR and DAR resids as feed material. Tests 83A9 and 83A10 were 3-cycle tests and tests 83A7 (or 84A16) and 83A8 were 5-cycle tests. The three- and five-cycle tests correspond to a catalyst age of 120 and 200 lbs-resid/lb-catalyst, respectively. Results of these tests are given below.

Resid type processed	-	LDAR	DAR
Test number	-	83A7 84A16	83A8

Shell 324M Catalyst Properties

Type	Fresh Oxide	Aged	Aged
Age, lbs-resid/lb-cat	-	200	200
Pore volume, cm ³ /g	0.374	0.254	0.227
Surface area, M ² /g	146	141	136

These results indicate that the differences exhibited in the single-cycle test continued to be observed in multi-cycle tests as the overall catalyst age increased. Processing of heavier resid (DAR) resulted in increased pore plugging causing a greater loss of available pore volume and surface area. Detailed results from these tests are summarized in Tables 7-1, 7-2 and 7-3. The surface area and Ti deposition values reported for Test 83A9 are somewhat questionable. The volume pore size distribution for these different resid treated aged catalyst are shown in Figure 7-1.

Effect of Process Configuration

In another set of batch tests (Nos. 83A9, 84A13, 84A18), the type of resid feed was the same as those obtained in Bench-Scale Runs 32, 33 and 34, using different process configurations. This set of batch tests was completed at similar catalyst age to permit relative evaluation of the effect on catalyst properties of different resid types obtained from varying process configurations. Although similar information was obtained from the bench-scale runs, these results could not be directly compared because of varying catalyst age. The spent catalysts from the bench-scale runs had a catalyst age ranging from 187 lbs-resid/lb-catalyst for Run 32 (LDAR as resid feed) to 104 for Run 33 (FR-DAR) and 54 for Run 34 (DAR). On the other hand, the catalyst age obtained from Tests 83A9, 83A13 and 84A18 was 120, 110 and 104 lbs-resid/lb-catalyst. The properties of aged catalysts from these tests are summarized below.

Resid type processed	-	LDAR	FR-DAR	DAR
Test number	-	83A9	83A13	84A18
<u>Shell 324M Catalyst Properties</u>				
Type	Fresh Oxide	Aged	Aged	Aged
Age, lbs-resid/lb-cat	-	120	110	104
Pore volume, cm ³ /g	0.374	0.281	0.256	0.245
Surface area, M ² /g	146	158	145	141

These results again show that loss of pore volume by pore plugging was more severe as the resid became more heavier. Hydroprocessing of LDAR produced the least pore plugging and DAR produced the most pore plugging. Detailed analyses and properties of the aged catalysts are given in Table 7-3. The volume pore size distribution for the aged catalyst are shown in Figure 7-2.

Effect of Initial Conditioning of Catalyst

In the petroleum refining industry, researchers have always maintained that the start-up or initial processing conditions utilized can substantially alter the hydroprocessing catalyst performance during its life cycle. The hydroprocessing catalysts used in coal liquefaction are probably no exception. In order to evaluate how the overall catalyst performance was affected by the initial treating period, two series of tests were made. In the first series of tests (MM-5, E-15B) reported in the February 84 quarterly report, both the oxide and pre-sulfided forms of catalyst, used in hydroprocessing of DAR, were exposed to resid without any appreciable exposure to any other material. In a second series of tests, (84A14 and 84A15) an identical batch of catalysts was first "conditioned" by hydroprocessing of essentially distillate type coal-derived solvent for an initial catalyst age of 35 lbs-solvent/lb-catalyst plus 5 lbs-resid/lb-catalyst. Following this initial conditioning with solvent, the aged catalyst was used in Tests 84A19 and 84A20 for hydroprocessing of DAR as was done in the first series of tests. The properties of fresh, solvent "preconditioned", and aged catalysts from the various tests are summarized below for both the oxide as well as sulfide forms of catalyst.

Resid type processed	-	DAR	DAR
Test number	-	MM-5	84A19
<u>Shell 324M Catalyst Properties</u>			
Type	Fresh Oxide	Aged	Aged (solvent-conditioned)
Age, lbs-resid/lb-catalyst	-	120	125
Pore volume, cm ³ /g	0.374	0.165	0.205
Surface area, M ² /g	146	92	135

Resid type processed	-	DAR	DAR
Test number	-	E-15B	84A20
<u>Shell 324M Catalyst Properties</u>			
Type	Sulfided	Aged	Aged (solvent-conditioned)
Age, lbs-resid/lb-cat	-	120	125
Pore volume, cm ³ /g	0.278	0.094	0.152
Surface area, M ² /g	131	70	96

These results show a definite improvement in catalyst properties by initial conditioning with solvent. The percent reduction in available surface area and pore volume is much smaller for the distillate solvent-conditioned catalyst than non-solvent treated catalyst. This is true for oxide as well as sulfided form of catalyst. These results are very encouraging and may provide a method of improving overall catalyst performance over the current level and help attain a 2000 lbs-resid/lb-catalyst catalyst age which is so highly desired in commercial applications. Additional testing is necessary in order to establish the optimum duration of initial solvent conditioning as well as the type of solvent more suited for a particular type of catalyst. More detailed analyses and properties of the aged catalysts are given in Tables 7-5 and 7-6. The reported higher surface area for solvent-conditioned catalyst cannot be readily explained. The volume pore size distribution for the aged catalyst are shown in Figures 7-3 and 7-4.

COAL LIQUEFACTION TESTS - SUBBITUMINOUS COAL

During Bench-Scale Runs 32 and 34, process-derived samples were obtained for the purpose of conducting batch reactor tests. Some of these tests, conducted during this reporting period, were comparison tests with the thermal stage of the Bench-Scale Runs. Results for some of these batch tests are discussed here. The overall results from all of the batch reactor liquefaction tests with subbituminous coal are given in Table 7-6. Analyses of the raw coal along with other raw materials are given in Appendix D.

Test 83E3 was completed using a mixture of DAR and Run 32 hydrotreater underflow containing H-LDAR. The results were to be compared with Run 32 MB results for product yields. This test, although originally planned at 1.8:1 solvent-to-MF coal (S/C) ratio, was run at S/C ratio of 1.52. This lower S/F ratio was a result of some distillate solvent loss (about 30 gm) during the solvent-drying step. Similarly, Test 83E5, using Run 32 hydro-underflow and DAR (similar to 83E3), was planned at a S/C ratio of 2.60 to simulate Run 32 MB-6. However, in actuality due to distillate loss in solvent-drying step, the test was completed at a S/C ratio of 2.13.

Of the several material balances completed during Run 32, MB's 2, 4 and 5 were completed at an average S/C ratio of 2.04. The results of the above two tests were compared with Run 32 Avg (of MB-2, 4 and 5) and are shown below,

	Batch Tests		Bench-Scale Run 32 MB
	<u>83E3</u>	<u>83E5</u>	<u>Avg (2,4,5)</u>
Liquefaction solvent type	H-LDAR**	H-LDAR**	H-LDAR+DAR
	DAR	DAR	
Ratio, Solvent/MF coal	1.52	2.13	2.04
+850°F conc. in Solv, wt%	35.8	33.8	45.9
Ratio, recycle resid/MAF coal	0.59	0.84	1.84
Coal conv., wt% MAF coal	96.4	96.0	92.1
Resid conversion			
To gases & water, wt%	16.1	12.1	19.9
To distillate, wt %	28.8	31.1	29.2
Thermal Stage Product Yield, wt % MAF Coal			
Hydrogen	<2.47>	<2.22>	<3.4>
Gases (H ₂ S, CO, CO ₂ , NH ₃)	6.61	6.25	8.2
Water	13.42	10.01	8.5
C ₁ -C ₃ hydrocarbons	4.84	5.34	12.7
C ₄ to -850°F liquids	46.94	58.26	49.3
+850°F resid	27.07	18.32	16.8
UC	<u>3.59</u>	<u>4.04</u>	<u>7.9</u>
Net	100.00	100.00	100.00

* Equivalent to atmospheric-flash material.

On the basis of these results, the following observations can be made:

- o As the S/C ratio increased from 1.52 to 2.13 for tests 83E3 and 83E5, the resid conversions to distillate increased which resulted in an increase in the overall distillate yield from 46.94 to 58.26 wt %. Of this total increase in distillate, 4.5 % increase was a direct result of improved resid conversion, (from 28.8 % to 31.3 %) and the balance of 6.8 % increase was the result of the increased S/C ratio. Obviously, process economics of reduced throughput at higher S/C ratio will have to be balanced against the improved distillate yield obtained at a higher S/C ratio.
- o In a similar manner, the distillate yield in Test 83E5 can be re-calculated to match the recycle resid/MAF coal ratio of 1.04 used in Run 32. At this resid level, the distillate yield in Test 83E5 will increase by about 6 wt% to 64 wt% (MAF coal basis). The batch reactor approaches a plug-flow system and appears to result in a much

higher coal conversion and produces more distillate and less gases than a continuous-flowing stirred-reactor system. The primary difference appears to be in the net make of C₁-C₃ hydro-carbon gases.

Test 83E6 was completed using H-LDAR from Run 32. No DAR was added as was done in Test 83E3. The purpose of this test was to evaluate the effect of addition of DAR as a component of the liquefaction solvent on the coal conversion and product yields. The results are summarized below.

Test Number	83E3	83E6
Liquefaction solvent type	H-LDAR+DAR	H-LDAR
Ratio, Solvent/MF coal	1.52	1.50
+850 ⁰ F conc. in solvent, wt%	35.8	20.0
Ratio, recycle resid/MAF coal	0.59	0.34
Coal conversion, wt % MAF coal	96.4	97.4
Resid Conversion		
To gases plus water, wt %	16.1	16.1
To distillate, wt %	28.8	34.5
Product Yield, wt % MAF Coal		
Hydrogen	<2.47>	<2.47>
Gases (H ₂ S,CO,CO ₂)	6.61	6.50
Water	13.42	10.15
C ₁ -C ₃ hydrocarbons	4.84	4.50
C ₄ to -850 ⁰ F liquids	46.94	47.68
+850 ⁰ F resid	27.07	31.04
UC	<u>3.59</u>	<u>2.60</u>
Net	100.00	100.00

Comparison of these results indicates the following:

- o In presence of H-LDAR, addition of a DAR did not appreciably affect coal conversion.
- o The resid conversion to distillate was lower for the H-LDAR + DAR mixture than for H-LDAR. The distillate yields are about the same for both tests because of the higher recycle resid/MAF coal in Test 83E3 compared to Test 83E6.

Test 83E8 was completed using H-DAR obtained in Bench-Scale Run 34. This test was a comparison test with the Bench-Scale Run 34. There were a total of three MB periods during Run 34. All of these had higher S/C ratios than Test 83E7. Of these three material balances, MB-2 and MB-3 had comparable resid levels and the results of these are compared with Test 83E8 as shown below.

	<u>Batch Test</u> <u>83E8</u>	<u>Bench-Scale Run 34 MB</u> <u>Avg (2 & 3)</u>
Liquefaction solvent type	H-DAR*+LDAR	H-DAR+LDAR
Ratio, solvent/MF coal	1.62	1.83
+850°F conc in solvent, wt%	41.87	22.0
Ratio, recycle resid/MAF coal	0.76	0.46
Coal conversion, wt% MAF coal	96.1	93.4
Resid Conversion		
To gases and water, wt %	14.11	23.1
To distillate, wt. %	21.9	25.6
Thermal Stage Product Yield, wt% MAF Coal		
Hydrogen	<1.75>	<2.0>
Gases (H ₂ S, CO, CO ₂ , NH ₃)	7.41	11.9
Water	12.06	7.3
C ₁ -C ₃ hydrocarbons	4.88	11.2
C ₄ to -850°F liquids	39.53	35.9
+850°F resid	33.96	29.2
UC	<u>3.91</u>	<u>6.6</u>
Net	100.00	100.0

* Equivalent to atmospheric flash material.

These results appear to be somewhat different. However, the differences are primarily the result of difference in resid concentration in liquefaction solvent. The coal conversion obtained in a batch reactor (plug-flow) was higher than obtained in continuous-flowing stirred-reactor the bench-scale unit. Moreover, loss of lights during solvent drying of coal in the batch reactor test favored a greater fraction of resid being converted to distillate as compared to gases and water. Another major difference was in S/C ratio. The bench-scale Run 34 was conducted at a somewhat higher solvent-to-MF coal ratio.

Another interesting comparison, shown below, was made between Tests 83E6 and 83E7. Although the reaction severity (time at temperature) was somewhat different the actual differences were quite small (Figure 7-5). Both tests were completed at almost identical S/C ratios using comparable resid concentrations. The major difference was in the type of solvent used. Test 83E6 used only H-LDAR underflow, obtained from Run 32, whereas Test 83E7 used only H-DAR underflow, obtained from Run 34. The hydrotreating temperature was identical for both Bench-Scale runs. The differences in product yields and resid conversion levels reveal the true effect of the quality of solvent being used.

Test Number	83E6	83E7
Liquefaction solvent type	H-LDAR	H-DAR
Ratio, solvent/MF coal	1.50	1.56
+850°F conc. in solvent, wt %	20.0	26.6
Ratio, recycle resid/MAF coal	0.34	0.46
Coal conversion, wt % MAF coal	97.4	85.6
Resid Conversion		
To gases plus water, wt %	16.1	17.2
To distillate, wt %	34.5	29.0
Product Yield, wt % MAF coal		
Hydrogen	<2.47>	<0.84>
Gases (H ₂ S, CO, CO ₂)	6.50	7.79
Water	10.15	10.06
C ₁ -C ₃ hydrocarbons	4.50	4.92
C ₄ to -850°F liquids	47.68	39.21
+850°F resid	31.04	24.50
UC	2.60	14.36
Net	<u>100.00</u>	<u>100.00</u>

Even at a somewhat lower resid level, use of H-LDAR as solvent resulted in a higher resid conversion to distillate and higher distillate yields, thus showing the superiority of this solvent over H-DAR. If the actual distillate yield in Test 83E7 is corrected for the recycle resid/MAF coal ratio to match a value of 0.34 of Test 83E6, the distillate yield in Test 83E7 would decrease by 4.5 wt % to an adjusted value of 34.7 wt %, which is 10 wt % lower than obtained with H-LDAR as a solvent.

The effectiveness of LDAR hydrotreating is further exemplified by reviewing the results of another pair of Tests, 83E3 and 83E8. In Test 83E8, the liquefaction solvent consisted of Run 34 LDAR mixed with Run 34 H-DAR underflow. This is exactly the reverse of Test 83E3 where Run 32 H-LDAR underflow was mixed with Run 32 DAR. A comparison of results is shown below.

Test Number	83E3	83E8
Liquefaction solvent type	H-LDAR+DAR	H-DAR+LDAR
Ratio, solvent/MF coal	1.52	1.62
+850°F conc. in solvent, wt %	35.78	41.87
Ratio, recycle resid/MAF coal	0.59	0.76
Coal conversion, wt % MAF coal	96.4	96.1
Resid conversion		
To gases plus water, wt %	16.10	14.11
To distillate, wt %	28.79	21.90
Product Yield, wt % MAF Coal		
Hydrogen	<2.47>	<1.75>
Gases (H ₂ S, CO, CO ₂)	6.61	7.41
Water	13.42	12.06
C ₁ -C ₃ hydrocarbons	4.84	4.88
C ₄ to -850°F liquids	46.94	39.53
+850°F resid	27.07	33.96
UC	3.59	3.91
Net	<u>100.00</u>	<u>100.00</u>

Test 83E8 has a higher recycle resid/MAF coal ratio (0.76 vs 0.59). If the actual distillate yield in Test 83E8 is adjusted, using the value of 21.9 wt % resid conversion to distillate, to match the lower recycle resid ratio of 0.59 in Test 83E3, the distillate yield in Test 83E8 would decrease by about 4.4 wt % to an adjusted distillate yield of 35.1 wt %. This adjusted distillate yield of 35.1 wt % is 11.8 wt% lower than obtained in Test 83E3.

This decrease in distillate yield was obviously the result of decreased resid conversion to distillate. The decreased resid conversion may be partially due to poor quality of the liquefaction solvent and the result of somewhat lower reaction temperature in Test 83E8. Test 83E8 shows a noticeable and severe endotherm after about 10 minutes into the test time (see Figure 7-6). However, high coal conversions (above 96 wt %) were obtained in both tests and appear not to be affected by the differences in the time-temperature profile. The distillate yield, on the other hand, was much less for Test 83E8 in comparison to Test 83E3. This raises a possible question as to whether the endothermic behavior is indicative of inadequate or improper level of hydrogenation of the starting solvent. Perhaps, during this endothermic behavior, the solvent is undergoing further conditioning before it is able to participate in a proper manner in the hydrogen donation reactions. And given adequate reaction time after this pre-conditioning phase is completed, both of these solvents may have resulted in similar product yields. Comparison of the results of these tests do, however, suggest the superiority of H-LDAR as a liquefaction solvent in terms of producing higher distillate yield over either LDAR or H-DAR.

COAL LIQUEFACTION TESTS - BITUMINOUS COAL

Three batch coal liquefaction tests using Illinois No. 6 bituminous coal were completed during this quarter. These batch tests were made to investigate the possible use of a disposable catalyst to improve distillate yields obtained from bituminous coal. On the basis of results obtained from the batch reactor tests, two bench-scale runs are planned in the next quarter using Illinois No. 6 coal.

The process conditions and results obtained from these three batch reactor tests are summarized in Table 7-7. All three tests utilized H-LDAR obtained from an earlier bench-scale run. The analyses of this material along with Illinois coal and other raw materials used are given in Appendix D.

Test 84E21 was made without any iron oxide. This was a base-test made to evaluate the effectiveness of iron oxide to be used in subsequent tests. However, during this

test, the actual average reaction temperature was 785°F instead of the intended value of 824°F. This sudden drop in reaction temperature was the result of endothermic reactions that are observed when a liner is used in the batch reactor. The test was continued in the expectation of regaining the required temperature level. However, this resulted in increasing the total reaction time (above 800°F) to 110 minutes rather than the intended 50 minutes. To eliminate the effect of the endothermic reactions, this test was repeated (84E22) without the use of a liner. During this test, the endothermic effect was minimal and reaction temperature was maintained near the intended value.

A comparison test (84E23) using 4 wt % iron oxide was also completed without the use of a liner. No difficulty was encountered in maintaining the intended reaction temperature. The operating conditions and detailed results of these three tests are summarized in Table 7-7. Comparison of results from Tests 84E22 and 84E23 indicate an improvement in distillate yield with 4 wt % iron oxide.

When results of test 84E21, made using an autoclave liner and no iron oxide, are compared with Test 84E22, made without liner, several observations can be made. The tests, 84E21 and 84E22, had appreciably different temperature profiles during the test period (see Figure 7-7). The time-averaged temperature for Test 84E21 was only 785°F compared to 827°F for Test 84E22 and 822°F for Test 84E23. However, in spite of this low temperature, the coal conversion and distillate yields for Test 84E21 were higher than those obtained in Test 84E22. It would appear that the longer reaction time in Test 84E21 (110 mins) more than made up for the lower temperature. The MAF coal conversion for Tests 84E21 and 84E22 were 95.7 wt% and 94.5 wt%, respectively, and the distillate yield was 46.68 wt% and 40.65 wt%, respectively. These results suggest that there may be a favorable effect from endothermic reactions (resulting from the use of a liner) and require further testing to ascertain if the distillate yields could be further improved by taking advantage of such endothermic reaction kinetics which appear to favor formation of distillate products at lower severity while maintaining high coal conversions.

BATCH REACTOR TESTS - TASK 3

In support of the bench-scale runs completed under Task 3, several batch reactor tests were completed during this quarter. Objective of these tests was to evaluate the effectiveness of various disposable catalysts in liquefaction of Wyoming subbituminous coal. Samples of various materials as a possible source of iron were obtained and analyzed. These included iron pyrite as well as various iron ores, mill scale, flue

dust, etc. All of these samples were analyzed for density, surface area and chemical analyses. The analyses of these materials including the total iron content as well as Fe^{+2} contents are given in Appendix D, Table D-3. Two sources of iron have been investigated as possible substitute for K-M Mobile Plant iron oxide. These were a) Iron ore pellets obtained near Pioneer and Eagle Mills in Wyoming and b) Mill Scale obtained from Paul Blum Company, Inc. of Buffalo, N.Y.

Results of the three tests 84E15, 84E16 and 84E17 are summarized below. Test 84E15 is a 'base-case' test without use of any disposable catalyst. Test 84E16 and 84E17 were completed using 2 wt % (MF coal basis) of Wyoming iron ore and mill-scale, respectively. All other process conditions including solvent were intended to be identical. An earlier test 83E3 is a comparable test using Mobile iron oxide at 2 wt % of MF coal. The results of these tests indicate that Wyoming iron ore performed somewhat comparable to Mobile iron oxide and resulted in higher coal conversion. The mill-scale, on the other hand, did not improve the coal conversion. The distillate yields for both tests, however, were not as good as those obtained with Mobile iron oxide.

Test Number	84E15	83E3	84E16	84E17
Disposable catalyst type	None	Iron Oxide	Iron Ore	Mill Scale
Catalyst quantity, wt% MF coal	0	2.0	2.0	2.0
Coal conversion, wt% MAF Coal	91.4	96.4	96.4	91.5
C ₄ to -850°F liquid yield, wt% MAF coal	36.7	46.9	33.6	34.9

Detail results for the above tests are given in Table 7-8.

Ref. Test No.	Shell 324M	Resid Type
	Catalyst Age	
	lbs-resid/lb-cat	
83A7	200	LDAR
83A8	200	DAR
83A9	120	LDAR
83A10	120	DAR

Figure 7-1. Volume Pore Size Distribution For Aged Catalyst After Hydroprocessing Different Types of Resids.

Figure 7-2. Volume Pore Size Distribution for Aged Catalyst After Hydroprocessing Different Types of Resids.

	Shell 324M
	Catalyst Age
<u>Ref. Test No.</u>	<u>lbs-resid/lb-cat</u>
MM-5	120
84A19	125

Figure 7-3. Volume Pore Size Distribution Comparison of Aged Catalyst "Oxide-Form" With and Without Solvent-Conditioning.

Figure 7-4. Volume Pore Size Distribution Comparison of Aged Catalyst "Presulfided-Form" with and without Solvent-Conditioning.

Figure 7-5. Time-Temperature Profile for Batch Reactor
Test 83E6 and 83E7

Figure 7-6. Time-Temperature Profile for Batch Reactor Tests 83E3 and 83E8

Figure 7-7. Time-Temperature Profile for Batch Reactor Tests 84E21 and 84E22

Table 7-1

Catalyst Aging Effect using LDAR¹

<u>Shell 324M Catalyst</u>	Fresh	Aged	Aged
Age (lbs-resid/lb-cat)	-	120	200
<u>Test No.</u>	-	83A9	83A7 84A16
<u>Properties</u>			
Pore volume (cm ³ /g)	0.374	0.281	0.254
% Reduction	-	25	32
Surface area (M ² /g)	146	158	141
% Reduction	-	-	5
<u>Composition, wt %</u>			
Carbon	-	12.6	13.2 ²
Hydrogen	-	1.5	1.4
H/C atomic ratio	-	1.42	1.27
Sulfur	-	2.36	2.90
Titanium	-	0.20	0.045
<u>Avg +850⁰F Resid Conv, wt %</u>	-	<u>43</u>	<u>33</u>
1 cycle	-	47	37
3 cycle	-	40	-
5 cycle	-	-	30

¹- For analysis, see Table D-1, Appendix D.

²- Reported composition is average of two tests.

Table 7-2

Catalyst Aging Effect using DAR¹

<u>Shell 324M Catalyst</u>	Fresh	Aged	Aged
Age (lbs-resid/lb-cat)	-	120	200
<u>Test No.</u>	-	83A10	83A8
<u>Properties</u>			
Pore volume (cm ³ /g)	0.374	0.251	0.227
% Reduction	-	33	39
Surface area (M ² /g)	146	147	136
% Reduction	-	-	7
<u>Composition, wt %</u>			
Carbon	-	10.88	11.43
Hydrogen	-	1.26	1.42
H/C atomic ratio	-	1.39	1.49
Sulfur	-	2.42	2.69
Titanium	-	0.14	0.15
<u>Avg +850⁰F Resid Conv, wt %</u>	-	<u>8</u>	<u>12</u>
1 cycle	-	7	12
3 cycle	-	9	11
5 cycle	-	-	13

¹- For analysis, see Table D-1, Appendix D.

Table 7-3

Comparison of Catalyst Aging Effect using Different Resids¹

<u>Resid Type in Feed</u>	-	LDAR	FR-DAR	DAR	LDAR	DAR
<u>Shell 324M Catalyst</u>	Fresh	Aged	Aged ²	Aged ³	Aged	Aged
Age (lbs-resid/lb-cat)	-	120	110	104	200	200
<u>Test No.</u>	-	83A9	83A13	84A18	83A7 & 84A16	83A8
<u>Properties</u>						
Pore volume (cm ³ /g)	0.374	0.281	0.256	0.245	0.254	0.227
% Reduction	-	25	32	35	32	39
Surface area (M ² /g)	146	158	145	141	141	136
% Reduction	-	-	1	3	5	7
<u>Composition, wt %</u>						
Carbon	-	12.60	9.72	9.65	13.2 ⁴	11.43
Hydrogen	-	1.50	1.36	1.22	1.4	1.42
H/C atomic ratio	-	1.42	1.70	1.52	1.27	1.49
Sulfur	-	2.36	1.55	1.82	2.90	2.69
Titanium	-	0.20	0.69	0.06	0.045	0.15
<u>Avg +850°F Resid Conv, wt %</u>	-	<u>41</u>	<u>36</u>	<u>25</u>	<u>33</u>	<u>12</u>
1 cycle	-	47	36	22	37	12
2 or 3 cycle	-	36	36	23	-	11
4 or 5 cycle	-	40	37	30	30	13

¹ - For analysis, see Table D-1, Appendix D.² - 4-cycle test.³ - 5-cycle test.⁴ - Average of two tests.

Table 7- 4

Effect of Solvent Preconditioning on Oxide Type Catalyst

Feedstock¹ DAR

Shell 324M Catalyst	Fresh	Aged	Solvent Preconditioned	
			Fresh	Aged
Age (lbs-resid/lb-cat)	-	120	5	125
Test No.	-	MM-5	83A14	84A19
<u>Properties</u>				
Pore volume (cm ³ /g)	0.374	0.165	0.346	0.205
% Reduction	-	56	7	45
Surface area (M ² /g)	146	92	171 ²	135
% Reduction	-	37	-	8
<u>Composition, wt %</u>				
Carbon	-	14.50	4.71	11.01
Hydrogen	-	1.74	1.02	1.21
H/C atomic ratio	-	1.44	2.60	1.32
Sulfur	-	4.26	1.14	3.76
Titanium	-	0.39	0.042	0.21
Avg +850°F Resid Conv, wt %	-	<u>14</u>	<u>36</u>	<u>14</u>
1 cycle		16	35	12
2 cycle		11	36	15
3 cycle		15	38	-

¹-For analysis, see Table D-1, Appendix D.²-Possibly an outlier value.

Table 7- 5

Effect of Solvent Preconditioning on Presulfided Catalyst

Feedstock¹ DAR

Shell 324M Catalyst	Fresh	Fresh	Aged	Solvent Preconditioned	
				Fresh	Aged
Chemical Form	Oxide	Presulfided			
Age (lbs-resid/lb-cat)	-		120	5	125
Test No.	-		E-15B	83A15	84A20
<u>Properties</u>					
Pore volume (cm ³ /g)	0.374	0.278	0.094	0.311	0.152
% Reduction	-	26	75	17	59
Surface area (M ² /g)	146	131	70	129	96
% Reduction	-	10	52	12	34
<u>Composition, wt %</u>					
Carbon	-	4.94	19.40	4.54	13.63
Hydrogen	-	1.40	2.02	1.04	1.28
H/C atomic ratio	-	3.40	1.25	2.75	1.13
Sulfur	-	5.66	6.16	8.29	6.53
Titanium	-	0.06	0.16	0.06	0.12
Avg +850°F Resid Conv, wt %	-		<u>26</u>	-	<u>21</u>
1 cycle			38	-	24
2 cycle			22	-	20
3 cycle			19	-	18

¹-For analysis, see Table D-1, Appendix D.

Table 7-6

SUMMARY OF BATCH REACTOR COAL LIQUEFACTION TESTS
DOE/EPRI/KERR-McGEE SPONSORED - 1983/84
Subbituminous Coal - TASK 2

TEST NO.	83E3	83E5	83E6	83E7	83E8	84E12
<u>Process Conditions</u>						
Coal Type	← Wyoming Subbituminous - Clovis Point Mine →					
Moisture, wt%	0 ⁵	0 ⁵	0 ⁵	0 ⁵	0 ⁵	0 ⁴
Ash (MF Basis), wt%	11.28	11.28	11.28	11.28	11.28	12.12
Solvent to Coal Ratio	1.52	2.13	1.50	1.56	1.62	1.91
Solvent Type ¹	H-LDAR + DAR	H-LDAR + DAR	H-LDAR	H-DAR	H-DAR + LDAR	H-LDAR+DAR (Run 3 ⁷)
	2.0 wt % iron oxide					
+850°F Conc. in Solv., wt%	35.78	33.77	20.03	26.55	41.87	19.97
Ratio, Recycle Resid/MAF Coal	0.59	0.84	0.34	0.46	0.76	0.38
Avg Reaction Temperature, °F	804	811	804	789	775	793
Reaction Pressure(Max), psig	2800	2570	2850	2880	2940	2640
Reaction Time ⁵ , minutes	50	50	50	50	50	50
<u>Material Recovery, wt%</u>	95.9	95.8	98.4	100.2	97.6	97.27
<u>Coal Conversion, wt% MAF Coal</u>	96.4	95.96	97.4	85.64	96.1	95.80
<u>Resid Conversion ²</u>						
To gases and water, rel wt %	16.10	12.06	16.13	17.22	14.11	16.12
To distillate, rel wt %	28.79	31.29	34.46	29.03	21.50	23.96
<u>Product Yield, wt % MAF Coal</u>						
Hydrogen	<2.47>	<2.22>	<2.47>	<0.84>	<1.75>	<1.91>
Gases (H ₂ S, CO, CO ₂)	6.51	6.25	6.50	7.79	7.41	6.96
Water	13.42	10.01	10.15	10.06	12.06	11.50
C ₁ to C ₃ hydrocarbons	4.34	5.34	4.50	4.92	4.88	3.97
C ₄ to +850°F liquids	46.34	58.26	47.68	39.21	39.53	35.25
+850°F Resid	27.07	18.32	31.04	24.50	33.56	40.03
Unconverted Coal	3.59	4.04	2.60	14.36	3.51	4.20
Net	100.0	100.0	100.0	100.0	100.0	100.0

NOTES¹ PS is process solvent, HDAR is hydrogenated DAR, HLDAR is hydrogenated Light DAR.² Conversion based on total resid weight. Includes resid equivalent of converted coal plus resid content of incoming solvent.³ Results are considered questionable due to large errors because of low material recovery.⁴ Solvent-dried coal slurry from Bench-Scale Run.⁵ Solvent-dried at 300°F for 1.0 hours.⁶ Counted after reaching initial temperature of 750°F.

Table 7-7
SUMMARY OF BATCH REACTOR COAL LIQUEFACTION TESTS

Bituminous Coal - TASK 2

TEST NO.	84E21	84E22 ⁴	84E23 ⁴
Iron Oxide Conc, wt % MF Coal	0	0	4.0
<u>Process Conditions</u>			
Coal Type	← Bituminous - Illinois No. 6 →		
Moisture, wt%	4.93	4.93	4.93
Ash (MF Basis), wt%	10.99	10.99	10.99
Solvent to Coal Ratio	1.80	1.91	1.90
Solvent Type ¹	H-LDAR	H-LDAR	H-LDAR
+850°F Conc. in Solv., wt%	37.2	37.2	37.2
Ratio, Recycle Resid/MAF Coal	0.67	0.71	0.71
Avg Reaction Temperature, °F	785	827	822
Reaction Pressure(Max), psig	2730	2650	2590
Reaction Time ³ , minutes	110	50	45
<u>Material Recovery, wt%</u>	97.89	98.86	98.87
<u>Coal Conversion, wt% MAF Coal</u>	95.73	94.47	93.54
<u>Resid Conversion</u> ²			
To gases and water, rel wt %	11.45	13.61	12.56
To distillate, rel wt %	25.52	22.73	24.83
<u>Product Yield, wt % MAF Coal</u>			
Hydrogen	<3.02>	<1.03>	<2.06>
Gases (H ₂ S, CO, CO ₂)	2.56	3.39	2.52
Water	9.70	11.00	11.42
C ₁ to C ₃ hydrocarbons	7.33	9.32	7.80
C ₄ to -850°F liquids	46.68	40.65	45.04
+850°F Resid	32.48	31.14	28.82
Unconverted Coal	4.27	5.53	6.46
Net	100.0	100.0	100.0

NOTES

- ¹ PS is process solvent, H-DAR is hydrogenated DAR, H-LDAR is hydrogenated LDAR.
- ² Conversion based on total resid weight. Includes resid equivalent of converted coal plus resid content of incoming solvent.
- ³ Counted after reaching initial temperature of 800°F.
- ⁴ Tests without autoclave liner.

Table 7-8
SUMMARY OF BATCH REACTOR COAL LIQUEFACTION TESTS

Subbituminous Coal - TASK 3

TEST NO.	84E15	83E3	84E16	84E17
Disposable Catalyst, Type	None	Iron Oxide	Iron Ore	Mill Scale
Disposable Catalyst, wt% MF Coal	-	2.0	2.0	2.0
<u>Process Conditions</u>				
Coal Type	Wyoming Subbituminous - Clovis Point Mine			
Moisture, wt%	0 ³	0 ³	0 ³	0 ³
Ash (MF Basis), wt %	12.2	11.28	12.7	12.2
Solvent to Coal Ratio	1.66	1.52	1.71	1.63
Solvent Type ¹	Run 32 H-LDAR + DAR			
+850 ⁰ F Conc in Solv, wt%	39.0	35.8	38.6	39.1
Ratio, Recycle Resid/MAF Coal	0.64	0.59	0.66	0.64
Avg Reaction Temp, ⁰ F	771	804	773	775
Reaction Press(Max), psig	2870	2800	2820	2780
Reaction Time ⁴ , min	50	50	50	50
<u>Material Recovery, wt%</u>	98.65	95.9	98.42	98.17
<u>Coal Conversion, wt% MAF Coal</u>	91.42	96.4	96.37	91.53
<u>Resid Conversion ²</u>				
To gases and water, rel wt %	15.15	16.10	14.95	15.87
To distillate, rel wt %	21.94	28.79	18.99	20.25
<u>Product Yield, wt % MAF Coal</u>				
Hydrogen	<0.72>	<2.47>	<1.19>	<1.78>
Gases (H ₂ S, CO, CO ₂)	7.05	6.61	5.78	6.50
Water	13.05	13.42	16.06	14.56
C ₁ to C ₃ hydrocarbons	4.76	4.84	3.65	4.87
C ₄ to -850 ⁰ F liquids	36.70	46.94	33.57	34.87
+850 ⁰ F Resid	30.57	27.07	38.50	32.51
Unconverted Coal	8.59	3.59	3.63	8.47
Net	100.0	100.0	100.0	100.0

NOTES

- ¹ PS is process solvent, H-DAR is hydrogenated DAR, H-LDAR is hydrogenated LDAR.
- ² Conversion based on total resid weight. Includes resid equivalent of converted coal plus resid content of incoming solvent.
- ³ Solvent-dried coal at 300⁰F for 1.0 hours.
- ⁴ Counted after reaching initial temperature of 750⁰F.

Section 8

FUTURE PROGRAM

A revision to the original work statement has been submitted. In accordance with this revision, during the next period the following work is planned.

Task 3

Bench-scale Run 40, the last of the Task 3 runs will be completed using 4 wt% (MF coal) iron oxide as a disposable catalyst. This will complete the Task 3 bench-scale runs.

Task 2

Two bench-scale runs under Task 2 to investigate the use of a disposable catalyst to improve distillate yield from Illinois No. 6 bituminous coal will be completed during the next quarter. With completion of these two bench-scale runs, requirements of Task 2 bench-scale runs will be complete.

Task 4

All Bench-Scale Runs under Task 4, to establish the preferred process parameters for carrying out secondary extraction for enhanced soluble-coal recoveries, will be completed during this quarter. Vacuum bottoms feed obtained from Wilsonville facility will be processed during these runs.

Table A-1

DAILY OPERATING LOG - BENCH SCALE UNIT

Run 35 (cont'd)

- Dec 1 Resid feed to the hydrotreater and liquefaction sections started at 1800 hours. Stopped external vac-bottoms feed to CSD at 0800 and repaired CSD feed pump.
- Dec 2 Restarted external vac-bottom feed to CSD but still experiencing minor feed pump problem possibly due to chunks of unmelted material. Coal feed started at 1200 hours.
- Dec 3 Experiencing some pump problems in all three sections.
- Dec 4 Unit on program conditions starting at 1045 hours. Minor plugging problems in LFU process solvent suction line and CSD first-stage letdown line.
- Dec 5 Increased ebullation recycle rate to decrease temperature spread in hydrotreater reactor.
- Dec 6 In order to help decrease gas make in the dissolver, restored heat on the hydrotreater underflow lines and started atmospheric flash of this material for removal of lighter components as a product. Heavy resid feed to LFU probably caused some feed pump plugging and resulted in interruption of coal feed to LFU section.
- Dec 7 Cleaned the solvent lines in LFU section. Hydrotreater and CSD on program conditions. External vac-bottoms being used in CSD to build-up the required resid levels.
- Dec 8 Continuing to experience plugging in the liquefaction solvent and coal slurry feed system.
- Dec 9 Changed to Run Period #3 conditions. Hydrotreater feed pump showing erratic feed rates.
- Dec 10 Repaired positioner and hydraulics on hydrotreater feed pump.
- Dec 11 Unit back on program conditions and started 35MB-1 at 2300 hours.
- Dec 12 Unit on 35MB-1 but experiencing some plugging in HTR and LFU solvent lines. Adjusted DAS composition. Ended MB-1 and started 35MB-2 at 2300 hours.
- Dec 13 Liquefaction solvent flow erratic due to line plugging and both hot separator letdown valves either malfunctioning or plugged. CSD on DAR recycle for 3 hours due to ash carryover. Ended 35MB-2 and Run 35 at 2300 hours.

Table A-1 - cont'd

DAILY OPERATING LOG - BENCH SCALE UNIT

Run 36

- Dec 14-15 Cleaned solvent lines in LFU section. Hydrotreater and liquefaction on solvent awaiting resid from CSD. Erratic and improper deashing in CSD in spite of changes in the DAS composition.
- Dec 16 Shutdown CSD to repair leak. Drained material from CSD feed tanks and charged Wilsonville Run 244 vac-bottoms. Other two sections operating on solvent.
- Dec 17 Hydrotreater on resid feed only for half the period. Started process vacuum bottoms to CSD. First stage heavy phase line plugs cleared with DAR recycle.
- Dec 18 One ebullation pump-head off-line because of excessive noise. CSD down for repair of a leak in the second-stage overhead line.
- Dec 19-21 Adjusting DAS composition did not correct ash carryover problems in CSD. Requiring DAR flushing often to cleanout process lines. Experiencing leak/plugging problems at several of the pumps in LFU and HTR areas. Decision made to terminate Run 36 and flushout CSD using LDAR before shutdown.
- Dec 22 Hydrotreater and LFU section shutdown after solvent flush for major maintenance items. CSD section shutdown after extended LDAR flushout. First stage vessel (bottom) removed for inspection/cleaning.
- Dec 23-26 Unit shutdown for holidays.
- Dec 27-31 Worked on major repair items in LFU and HTR sections. CSD unit after repair/cleaning was restarted in preparation of a short test for KM client testing.

Run 37

- Jan 3 Established solvent flows in the hydrotreater and CSD sections. Started melting Wilsonville Run 244 vac-bottoms for CSD feed. Replaced rupture disc on Dowtherm heater.
- Jan 4 Started vac-bottoms feed to CSD. Worked on minor process and heat-media leaks in liquefaction section.
- Jan 5 Started resid feed to hydrotreater.
- Jan 6-8 Buffer oil feed lines in hydrotreater required frequent cleaning. Otherwise hydrotreater operating at program conditions. Adjusting vacuum flash conditions to control vac-bottom viscosity. Problems in proper deashing required frequent DAR flushes. Lack of adequate resid flows resulted in hydrotreater operation on straight solvent.

Table A-1 - cont'd

DAILY OPERATING LOG - BENCH SCALE UNIT

Run 37 - cont'd

Jan 9-11	Adjusted DAS composition and started addition of vac-bottom from Runs 36 and 37 to build-up required system resid levels. Installed a knock-out pot in suction of buffer oil pumps and corrected ebullation pump control problem causing irregular reactor temperature.
Jan 12	The unit was on resid feed operating under planned conditions.
Jan 13	Started coal feed containing 4 wt % (MF basis) iron oxide.
Jan 14-15	Experienced minor problems with hydrotreater feed and ebullation pumps. Balance of the unit operating smoothly. Occasional problem with proper deashing in the CSD unit.
Jan 16	Adjusted DAS composition. Hydrotreater feed pump packing leak resulted in interruption of resid feed to hydrotreater.
Jan 17	Dissolver was flushed out and liquefaction section shutdown to include one salt-bath preheater in the process loop. Repaired hydrotreater feed pump and established resid feed.
Jan 18	Unit operating on program conditions.
Jan 19-21	Raised dissolver temperature first to 835°F and then to 840°F in order to improve coal conversions. Adjusted DAS composition.
Jan 22	Adjusted unit to modified conditions and reduced the coal rate.
Jan 23	Lowered coal dissolver temperature to 825°F at 0900 hours. Other sections operating on program conditions.
Jan 24-25	Unit operated on program conditions. CSD feed was off for 3.5 hours to replace feed pump packing.
Jan 26	Started 37MB-1 at 2300 hours.
Jan 27	Plugged line in liquefaction required batching process solvent. Ended 37MB-1 at 2300 hours.
Jan 28-29	Continued batching process solvent in liquefaction. Added Wilsonville Run 244 vac-bottoms as needed to build-up resid levels in the unit.
Jan 30-31	Continued batching process solvent in liquefaction. Other units operating at program conditions.

Table A-1 - cont'd

DAILY OPERATING LOG - BENCH SCALE UNIT

Run 37 - cont'd

Feb 1 Continued batching process solvent in liquefaction. Melted LDAR from Wilsonville Run 244 for use in LFU section. CSD off feed for a total of four hours for equipment repair. Added two drums of Wilsonville solvent to process solvent storage vessel.

Feb 2-3 Unit operating on program conditions. Continued to add LDAR from Wilsonville Run 244.

Feb 4 Discontinued external LDAR feed to hydrotreater and started DAR feed to liquefaction section.

Feb 5 Started 37MB-2 at 2300 hours.

Feb 6 Ended 37MB-2 and Run 37 at 2300 hours.

Feb 7-8 Flushed LFU and HTR sections and cooled down. Removed catalyst from hydrotreater reactor.

Run 38

Feb 9 Fresh Shell 324M catalyst charged to hydrotreater and for Run 38.

Feb 10-12 Finished pre-sulfiding of the fresh catalyst. Processed entire inventory of process solvent (including flush solvent) through liquefaction section for removal of lighter fractions and started coal feed. CSD being operated in DAR recycle mode.

Feb 13 Started feeding hydro-underflow and DAR directly to coal slurry make tank.

Feb 14-15 To maintain proper deashing, adjusted DAS composition and first stage operating pressure-temperature. Hydrotreater and liquefaction operating with minor plugging and pump problems.

Feb 16 Liquefaction off coal feed for several hours while letdown valve repaired for broken stem.

Feb 17 Unit operating smoothly.

Feb 18-19 Completed two 24-hour material balances and ended Run 38.

Table A-1 - cont'd

DAILY OPERATING LOG - BENCH SCALE UNIT

Run 39

Feb 20 Switched to Run 39 conditions and started coal feed without iron oxide.

Feb 21 Solvent leak in CSD section. Hydrotreater and liquefaction sections being operated on resid from inventory with balance of flows coming from make-up solvent.

Feb 22 Restarted CSD feed after repair of system leak.

Feb 23 Prepared CSD secondary extractor for testing.

Feb 24 Hydrotreater and liquefaction operating at Run Conditions. Adjusted DAS composition in CSD.

Feb 25 Shut off chilled glycol to vac-overhead cooler after it plugged.

Feb 26 Started 39MB-1 at 2300 hours.

Feb 27 MB had to be terminated due to loss of hydrotreater reactor temperature resulting from burned out heating elements in the preheater. Because of the, consistantly lower coal conversions in absence of iron oxide, a decision was made to terminate run. Special samples obtained in lieu of an actual material balance.

Run 40

Feb 28-29 After replacement of electric heating elements in the hydrotreater preheater, switched to coal feed containing 4 wt % (MF coal basis) iron oxide for Run 40. Cleaned and prepared for testing the CSD secondary extractor.

TABLE A-2
COAL LIQUEFACTION BENCH-SCALE OPERATION ON-STREAM FACTOR
Sept 1983 - Feb 1984

Operation Under DOE Contract No. DE-AC22-83FC60048

Run No.	Run Duration (days)	ON-STREAM TIME - DAYS						
		<u>Integrated Resid & Coal</u>	<u>Liquefaction</u>		<u>CSD</u>		<u>Hydrotreater</u>	
			<u>Solvent</u>	<u>Coal</u>	<u>Solvent</u>	<u>Resid</u>	<u>Solvent</u>	<u>Resid</u>
32	44	20.0	42.7	29.1	41.0	27.5	36.8	32.1
33	15	12.4	15.0	13.3	15.0	13.2	15.0	13.9
34	12	8.4	11.9	10.3	11.6	9.7	12.0	9.8
35	17	8.4	16.0	9.4	15.9	11.7	16.5	10.8
36	9	3.0	7.6	5.8	8.4	4.2	8.3	3.8
37	35	25.7	33.0	31.2	34.9	30.2	35.0	29.3
38	11	7.4	10.8	7.7	10.7	8.3	11.0	8.9
39	7	4.8	7.0	7.0	6.5	5.7	7.0	5.4
Total		90.1	144.0	113.8	144.0	110.5	141.6	114.0
% On-Stream Time ¹		(150) 60.0	(150) 96.0	(150) 75.9	(150) 96.0	(150) 73.7	(150) 94.4	(150) 76.0

NOTES:

¹ Numbers in paranthesis are the total operating days used in calculation of percent on-stream time.

Table A-3

BENCH SCALE UNIT - HYDROTREATER CATALYST AGE LOG

Catalyst Type - Shell 324M
 Weight - 11.48 lb
 Date Charged - November 23, 1983
 Run Number - 35

Date	Hydrotreater Feed Time, hrs		D-5108 Feed		Catalyst Age	
	Total	850°F + Resid	Rate lb/hr	wt % 850°F + Resid	Daily	lb+850°F lb-cat Cumm
12/1	24	6	10.1	4.6	0.2	0.2
12/2	24	23	7.2	12.0	1.7	1.9
12/3	24	20.8	6.5	14.3	1.7	3.6
12/4	24	24	4.8	18.6	1.9	5.5
12/5	24	24	4.5	22.0	2.1	7.6
12/6	24	24	4.6	13.4	1.3	8.9
12/7	24	24	5.1	14.0	1.5	10.4
12/8	24	24	5.8	11.8	1.4	11.8
12/9	24	24	6.0	26.2	3.3	15.1
12/10	24	14	6.9	8.1	0.7	15.8
12/11	24	24	5.4	14.8	1.7	17.5
12/12	24	24	6.2	12.4	1.6	19.1
12/13	24	24	5.7	12.0	1.4	20.5

12/14/83 Run 35 was terminated and catalyst was not removed.

Catalyst Type - Shell 324M
 Weight - 11.48 lb
 Date Charged - November 23, 1983
 Run Number - 36

Date	Hydrotreater Feed Time, hrs		D-5108 Feed		Catalyst Age	
	Total	850°F + Resid	Rate lb/hr	wt % 850°F + Resid	Daily	lb+850°F lb-cat Cumm
12/14	24	24	9.0	6.8	8.7	29.2
12/15	24	2	9.1	11.9	0.2	29.4
12/16	24	5.5	6.1	11.8	0.3	29.7
12/17	24	9	6.7	8.8	0.5	30.2
12/18	24	21	5.0	11.5	1.1	31.3
12/19	24	22	5.8	15.2	1.7	33.0
12/20	24	8	11.6	20.7	1.7	34.7
12/21	24	0	22.8	-	-	34.7

12/22/83 Run 36 was terminated and catalyst was not removed.

12-23/31-83 Maintenance period.

Table A-3 (contd)

BENCH SCALE UNIT - HYDROTREATER CATALYST AGE LOG

Catalyst Type - Shell 324M
 Weight - 11.48 lb
 Date Charged - November 23, 1983
 Run Number - 37

Date	Hydrotreater Feed Time, hrs		D-5108 Feed		Catalyst lb+850°F	
	Total	850°F + Resid	Rate lb/hr	wt % 850°F + Resid	Age Daily	lb-cat Cumm
1/3	-	-	-	-	-	34.7
1/4	24	-	13.0	-	-	34.7
1/5	24	22	10.5	0.4	0.1	34.8
1/6	24	24	8.7	46.5	8.5	43.3
1/7	24	20	6.4	44.0	4.9	48.2
1/8	24	8	7.5	19.7	1.0	49.2
1/9	24	2.5	8.3	2.0	-	49.2
1/10	24	1	6.9	6.2	-	49.2
1/11	24	24	7.2	32.2	4.8	54.0
1/12	24	24	8.3	12.3	2.1	56.1
1/13	24	24	7.6	14.3	2.3	58.4
1/14	24	24	10.5	16.5	3.6	62.0
1/15	24	22	7.1	42.3	5.8	67.8
1/16	24	21.8	6.5	20.8	2.6	70.4
1/17	24	11	7.0	2.2	0.2	70.6
1/18	24	16	7.3	12.1	1.2	71.8
1/19	24	24	7.8	40.0	6.5	78.3
1/20	24	24	8.4	30.7	5.4	83.7
1/21	24	24	7.3	23.6	3.6	87.3
1/22	24	24	5.1	16.6	1.8	89.1
1/23	24	24	3.2	21.4	1.4	90.5
1/24	24	24	2.7	21.0	1.2	91.7
1/25	24	24	3.4	35.2	2.5	94.2
1/26	24	24	3.5	15.9	1.2	95.4
1/27	24	24	3.4	15.1	1.1	96.5
1/28	24	24	7.3	17.7	2.7	99.2
1/29	24	24	7.5	25.9	4.1	103.3
1/30	24	24	7.0	11.2	1.6	104.9
1/31	24	24	7.2	14.6	2.2	107.1
2/1	24	24	7.5	21.3	3.3	110.4
2/2	24	24	5.6	13.2	1.5	111.9
2/3	24	24	4.2	13.0	1.1	113.0
2/4	24	24	3.4	20.7	1.5	114.5
2/5	24	24	3.0	19.2	1.2	115.7
2/6	24	21.4	3.4	16.9	1.1	116.8

2/7/84 Run 37 was terminated and catalyst was removed.

Table A-3 (contd)

BENCH SCALE UNIT - HYDROTREATER CATALYST AGE LOG

Catalyst Type - Shell 324M
 Weight - 11.48 lb
 Date Charged - February 9, 1984
 Run Number - 38

Date	Hydrotreater Feed Time, hrs		D-5108 Feed		Catalyst Age	
	Total	850°F + Resid	Rate lb/hr	wt% 850°F + Resid	Daily	lb+850°F lb-cat Cumm
2-12	24	14	5.3	-	-	0
2-13	24	24	5.6	39.5	4.6	4.6
2-14	24	24	7.3	48.9	7.5	12.1
2-15	24	24	6.4	45.6	6.1	18.2
2-16	24	24	7.4	45.9	7.1	25.3
2-17	24	24	5.9	24.2	3.0	28.3
2-18	24	24	6.6	27.2	3.8	32.1
2-19	24	24	6.5	18.7	2.5	34.6
2-20	24	24	6.6	22.2	3.1	37.7

End of Run 38. Catalyst was not removed.

Catalyst Type - Shell 324M
 Weight - 11.48 lb
 Date Charged - February 9, 1984
 Run Number - 39

Date	Hydrotreater Feed Time, hrs		D-5108 Feed		Catalyst Age	
	Total	850°F + Resid	Rate lb/hr	wt% 850°F + Resid	Daily	lb+850°F lb-cat Cumm
2-21	24	16	7.0	16.8	1.6	39.3
2-22	24	16	5.4	17.3	1.3	40.6
2-23	24	24	6.9	35.3	5.1	45.7
2-24	24	24	6.4	30.3	4.1	49.8
2-25	24	21.2	6.9	19.7	2.5	52.3
2-26	24	24	6.1	30.6	3.9	56.2
2-27	24	10	7.2	26.3	1.6	57.8

End of Run 39. Catalyst was not removed.

Table B-1
OPERATING DATA
Thermal Stage

Date 1983	Coal Feed Rate lb/hr	Coal Slurry		Liq Solv +850 wt%	Preheater Outlet Temp °F	Dissolver Temp		Dissolver Pressure psig	CSD Feed Rate lb/hr	CSD Soluble Coal Recovery %
		Conc.MF Coal wt%	Feed Temp °F			Bottom °F	Top °F			
Run 35										
DEC										
1	10.1	0.7	301	4.8	787	823	808	2351	12.2	81.7
2	7.2	0.1	313	18.8	786	823	810	2351	14.8	77.1
3	6.5	6.8	313	23.2	784	823	805	2359	14.6	77.8
4	4.8	28.2	314	42.5	786	825	799	2361	9.9	79.2
5	4.5	21.2	310	31.5	785	824	800	2359	7.5	79.1
6	4.6	26.2	298	31.7	787	823	815	2357	6.7	80.3
7	5.1	1.1	288	24.9	790	823	822	2350	6.8	78.6
8	5.8	23.9	322	20.9	784	826	824	2355	9.9	78.4
9	6.0	22.8	323	33.3	777	824	823	2374	11.0	85.0
10	6.9	29.7	325	30.7	785	825	823	2371	7.5	80.9
11	5.4	36.6	322	28.5	785	824	823	2354	7.4	77.0
12	6.2	36.2	320	28.2	785	824	823	2351	7.5	76.8
13	5.7	25.6	329	27.8	787	823	822	2343	7.1	72.5
Run 36										
DEC										
14	11.4	38.6	324	16.8	787	820	818	2339	6.0	66.4
15	10.7	38.2	325	8.8	789	824	823	2354	-	63.9
16	10.9	30.5	308	5.4	788	824	822	2350	-	72.6
17	11.0	32.3	323	27.1	792	825	823	2356	14.0	-
18	11.0	35.7	324	24.7	787	823	822	2360	9.4	70.1
19	11.1	35.6	317	22.5	784	826	824	2360	7.6	76.7
20	11.2	36.3	318	21.4	784	826	825	2357	7.9	-
21	-	-	294	-	722	676	674	2359	10.0	-

B-1

Table B-1 (contd)
OPERATING DATA
Thermal Stage

Date 1984	Coal Feed Rate lb/hr	Coal Slurry		Liq Solv +850 wt%	Preheater Outlet Temp °F	Dissolver Temp		Dissolver Pressure psig	CSD Feed Rate lb/hr	CSD Soluble Coal Recovery %
		Conc. °F Coal wt%	Feed Temp °F			Bottom °F	Top °F			
Run 37										
JAN										
11	10.8	35.8	308	21.8	-	824	826	2357	9.6	79.1
12	11.1	25.7	304	16.6	-	824	826	2353	8.4	66.8
13	11.3	35.3	306	22.1	-	824	825	2359	8.0	70.6
14	11.2	37.7	306	17.4	-	824	825	2358	7.5	74.4
15	11.2	37.4	367	20.8	-	822	823	2352	7.5	72.6
16	11.3	44.0	375	13.5	-	823	824	2358	7.2	69.2
17	10.4	31.4	380	8.3	-	824	825	2358	9.6	69.9
18	10.6	35.7	350	9.6	776	823	823	2360	10.1	70.3
19	11.1	33.1	300	14.4	788	831	831	2361	9.9	76.0
20	11.1	35.1	289	22.4	781	835	835	2368	7.2	77.8
21	11.0	34.4	300	20.0	787	840	840	2356	6.8	79.0
22	8.7	37.0	295	22.2	787	838	838	2351	5.8	75.5
23	7.3	43.1	298	19.7	790	824	824	2342	5.4	69.9
24	7.4	38.4	318	21.2	789	823	823	2351	5.0	74.9
25	7.2	35.1	328	15.4	783	822	822	2352	4.8	78.0
26	8.3	37.5	330	13.8	786	826	826	2353	4.4	74.5
27	7.1	38.4	321	18.8	787	826	826	2344	4.3	77.1
28	7.3	31.7	292	11.2	786	824	825	2357	9.9	84.5
29	7.2	36.6	292	19.6	784	822	822	2361	9.7	80.0
30	7.1	31.4	289	22.5	785	821	821	2360	7.1	82.8
31	7.3	30.3	294	13.7	784	822	823	2359	5.3	83.0
FEB										
1	7.2	13.8	301	-	785	822	823	2359	5.3	81.1
2	7.3	27.5	336	-	788	823	823	2353	5.3	74.8
3	7.4	25.9	313	32.3	784	821	822	2362	4.9	65.7
4	7.3	30.4	311	37.2	786	823	823	2364	5.4	74.5
5	7.4	33.6	322	24.9	786	823	824	2355	5.3	71.0
6	7.3	40.4	336	12.6	787	825	825	2346	5.4	76.3

Table B-1 (contd)

OPERATING DATA
Thermal Stage

Date 1984	Coal Feed Rate lb/hr	Coal Slurry		Liq Solv +850 wt%	Preheater Outlet Temp °F	Dissolver Temp		Dissolver Pressure psig	CSD Feed Rate lb/hr	CSD Soluble Coal Recovery %
		Conc.MF Coal wt%	Feed Temp °F			Bottom °F	Top °F			
Run 38										
FEB										
13	10.9	12.8	334	-	784	826	827	2358	14.1	85.5
14	11.0	28.0	328	-	783	826	826	2360	12.8	85.3
15	11.2	16.5	323	-	776	825	824	2358	7.1	85.0
16	11.1	39.1	326	-	784	825	825	2366	7.2	85.2
17	11.1	35.9	329	-	784	825	824	2358	7.2	84.0
18	10.8	32.6	328	31.5	787	825	825	2359	7.1	81.2
19	11.2	38.2	339	32.4	784	823	823	2361	7.1	82.1
20	11.2	40.1	350	25.6	787	824	825	2355	7.2	80.3
Run 39										
FEB										
21	11.1	38.7	370	22.9	785	824	824	2354	8.3	69.9
22	11.1	40.6	359	29.8	787	825	826	2350	13.1	79.5
23	11.5	36.1	350	34.5	785	824	825	2355	9.8	73.9
24	11.5	36.2	339	33.0	785	825	826	2352	7.2	72.8
25	10.9	39.4	-	30.8	782	-	-	-	7.2	67.5
26	11.1	39.7	358	34.9	783	823	824	2359	8.1	68.8
27	10.9	36.8	360	34.4	784	823	825	2359	7.2	59.9

Table B-2
OPERATING DATA
Catalytic Stage

Date 1983	D-5108 Feed Rate lb/hr	+850°F Conc wt%	Buffer Oil Rate lb/hr	Reactor Avg Temp °F	Outlet Pressure psig	Hydro Underflow +850°F Conc wt%	Catalyst Age #+850°F/# Cat Cumm
Run 35 DEC							
1	10.1	4.6	6.1	694	2281	4.0	0.2
2	7.2	12.0	5.6	694	2292	11.0	1.9
3	6.5	14.3	5.2	697	2289	6.4	3.6
4	4.8	18.6	5.0	695	2290	9.2	5.5
5	4.5	22.0	4.9	696	2299	7.3	7.6
6	4.6	13.4	5.2	703	2296	4.8	8.9
7	5.1	14.0	5.0	702	2282	3.4	10.4
8	5.8	11.8	4.7	700	2278	5.9	11.8
9	6.0	26.2	6.1	698	2287	6.1	15.1
10	6.9	8.1	6.1	699	2277	3.0	15.8
11	5.4	14.8	5.2	698	2290	7.3	17.5
12	6.2	12.4	6.0	697	2289	6.8	19.1
13	5.7	12.0	5.9	698	2286	5.0	20.5
Run 36 DEC							
14	9.0	6.8	6.3	693	2291	3.2	29.2
15	9.1	11.9	6.9	697	2288	3.9	29.4
16	6.1	11.8	5.8	701	2275	3.5	29.7
17	6.7	8.8	4.4	697	2280	4.2	30.2
18	5.0	11.5	6.1	700	2374	4.9	31.3
19	5.8	15.2	6.3	699	2379	6.5	33.0
20	11.6	20.7	5.4	705	2380	11.6	34.7
21	-	-	6.8	697	2380	-	34.7

Table B-2 (contd)
OPERATING DATA
Catalytic Stage

Date 1984	Rate	D-5108 Feed +850°F Conc wt%	Buffer Oil Rate lb/hr	Avg Temp °F	Reactor Outlet Pressure psig	Hydro Underflow +850°F Conc wt%	Catalyst Age #+850°F/# Cat Cumm
Run 37							
JAN							
11	7.2	32.2	6.0	702	2297	17.8	54.0
12	8.3	12.3	5.8	700	2290	5.3	56.1
13	7.6	14.3	5.1	699	2296	9.6	58.4
14	10.5	16.5	6.2	698	2294	12.8	62.0
15	7.1	42.3	5.3	700	2294	26.6	67.8
16	6.5	20.8	7.5	698	2292	7.2	70.4
17	7.0	2.2	7.8	691	2291	12.6	70.6
18	7.3	12.1	5.4	697	2293	11.1	71.8
19	7.8	40.0	5.4	699	2288	16.8	78.3
20	8.4	30.7	5.8	697	2299	15.5	83.7
21	7.3	23.6	5.6	698	2290	10.0	87.3
22	5.1	16.6	5.2	700	2294	7.6	89.1
23	3.2	21.4	5.2	701	2293	8.5	90.5
24	2.7	21.0	5.6	699	2289	7.6	91.7
25	3.4	35.2	4.8	693	2289	12.7	94.2
26	3.5	15.9	4.7	700	2288	7.2	95.4
27	3.4	15.1	5.4	700	2290	7.2	96.5
28	7.3	17.7	5.8	696	2290	9.9	99.2
29	7.5	25.9	5.5	693	2291	12.6	103.3
30	7.0	11.2	5.5	696	2291	8.9	104.9
31	7.2	14.6	6.5	698	2286	9.4	107.1
FEB							
1	7.5	21.3	5.4	697	2287	9.9	110.4
2	5.6	13.2	5.4	689	2286	4.3	111.9
3	3.0	13.0	5.0	693	2287	4.5	113.0
4	3.4	20.7	5.1	696	2295	5.5	114.5
5	4.2	19.2	5.6	694	2288	7.6	115.7
6	3.4	16.9	5.2	700	2280	7.0	116.8

Table B-2 (contd)

OPERATING DATA
Catalytic Stage

Date 1984	D-5108 Feed Rate lb/hr	+850°F Conc wt%	Buffer Oil Rate lb/hr	Reactor Avg Temp °F	Outlet Pressure psig	Hydro Underflow +850°F Conc wt%	Catalyst Age #+850°F/# Cat Cumm
Run 38 FEB							
13	5.6	39.5	5.8	700	2273	21.1	4.6
14	7.3	48.9	5.6	699	2280	28.3	12.1
15	6.4	45.6	5.6	697	2273	30.1	18.2
16	7.4	45.9	5.5	699	2276	22.4	25.3
17	5.6	24.2	5.5	700	2277	16.8	28.3
18	6.6	27.2	5.6	698	2271	18.8	32.1
19	6.5	18.7	5.3	699	2281	17.5	34.6
20	6.6	22.2	5.3	701	2278	17.3	37.7
Run 39 FEB							
21	7.0	16.8	7.3	700	2272	12.8	39.3
22	5.4	17.3	8.2	702	2265	20.1	40.6
23	6.9	35.3	6.4	699	2267	19.0	45.7
24	6.4	30.3	5.9	700	2265	18.2	49.8
25	6.9	19.7	5.4	700	-	14.3	52.3
26	6.1	30.6	5.6	698	2263	18.5	56.2
27	7.2	26.3	9.5	-	2271	17.0	57.8

Table C-1
ANALYTICAL DATA (Sheet 1)

Date 1983	Liquefaction						Hydrotreater			
	Coal Feed		Soln +850°F wt%	Vent Gas		Coal Conversion wt%	Feed +850°F wt%	Underflow +850°F wt%	Vent Gas	
	Moist wt%	Ash wt%		H ₂ S, CO, CO ₂ wt%	C ₁ -C ₅ wt%				H ₂ S wt%	C ₁ -C ₅ wt%
Run 35 DEC										
1	-	-	4.8	0.19	14.6	-	3.2	4.0	0.35	2.86
2	-	-	18.8	0.31	20.5	-	6.4	11.0	0.19	2.54
3	27.3	9.66	23.2	8.19	17.2	86.7	4.5	6.4	-	1.37
4	27.3	9.66	42.5	16.1	45.2	86.2	9.2	9.2	0.42	1.45
5	28.6	9.98	31.5	30.2	22.5	87.5	10.8	7.3	0.38	1.40
6	28.6	9.98	31.7	15.9	29.1	92.8	5.6	4.8	0.83	2.45
7	28.6	9.98	24.9	8.98	33.7	86.5	5.8	3.4	0.38	4.41
8	28.6	10.09	20.9	28.2	21.7	90.1	6.0	5.9	1.64	3.30
9	28.6	10.09	33.3	19.6	47.8	84.2	10.8	6.1	0.43	3.46
10	28.6	10.09	30.7	33.1	31.2	85.7	-	3.0	0.66	5.25
11	26.8	9.90	28.5	35.4	26.3	87.3	7.8	7.3	0.36	3.98
12	26.8	9.90	28.2	31.0	33.4	90.1	6.6	6.8	0.41	3.92
13	27.5	9.53	27.8	25.1	28.7	86.8	5.5	5.0	0.38	3.63
Run 36 DEC										
14	-	-	16.8	26.3	26.8	-	4.1	3.2	0.17	3.21
15	27.5	9.5	8.8	14.3	24.5	86.9	5.7	3.9	0.17	2.63
16	29.2	9.4	5.4	21.4	24.3	90.6	6.8	3.5	0.71	3.33
17	29.2	9.4	27.1	20.2	27.3	87.6	5.1	4.2	1.14	3.59
18	28.5	9.9	24.7	39.3	26.0	87.9	7.2	4.9	0.92	21.1
19	28.5	9.9	22.5	27.3	23.8	87.1	8.8	6.5	0.21	3.51
20	27.4	9.9	21.4	30.3	29.6	87.7	16.9	11.6	0.64	7.49
21	27.4	9.9	-	-	-	-	-	-	-	-

C-1

Table C-1 (contd)

ANALYTICAL DATA (Sheet 1)

Date 1984	Liquefaction					Hydrotreater				
	Coal Feed		Solv +850°F wt%	Vent Gas		Coal Conversion wt%	Feed +850°F wt%	Underflow +850°F wt%	Vent Gas	
	Moist wt%	Ash wt%		H ₂ S, CO, CO ₂ wt%	C ₁ -C ₅ wt%				H ₂ S wt%	C ₁ -C ₅ wt%
Run 37										
JAN										
11	27.4	10.06	21.8	17.68	42.2	85.8	18.0	17.8	-	11.9
12	27.4	10.06	16.6	25.84	35.6	85.8	6.4	5.3	-	6.7
13	29.2	10.06	22.1	28.23	34.8	85.7	8.7	9.6	-	9.4
14	24.0	8.24	17.4	24.37	37.5	84.2	9.8	12.8	-	8.8
15	24.0	8.24	20.8	35.69	28.2	86.3	24.4	26.6	-	3.9
16	22.4	7.36	13.5	31.13	31.0	87.7	11.2	7.2	-	3.0
17	22.4	7.36	8.3	18.56	28.3	89.8	1.1	12.6	-	-
18	22.4	7.36	9.6	40.04	18.8	65.0	7.1	11.1	-	3.6
19	29.2	10.06	14.4	14.08	38.1	89.2	22.0	16.8	-	3.8
20	27.3	10.94	22.4	33.65	35.5	86.4	17.7	15.5	-	6.0
21	27.0	10.79	20.0	27.37	42.2	92.1	12.5	10.0	-	6.7
22	27.0	10.79	22.2	31.16	43.0	94.2	9.9	7.6	-	7.8
23	28.0	10.78	19.7	20.12	46.4	93.1	8.9	8.5	-	7.8
24	26.5	9.70	21.2	29.02	38.2	93.9	9.3	7.6	0.19	7.7
25	26.5	9.70	15.4	25.64	35.4	93.9	14.2	12.7	0.23	5.5
26	27.4	11.02	13.8	23.04	38.1	92.9	6.4	7.2	-	5.9
27	29.2	11.54	18.8	22.80	39.6	93.8	6.1	7.2	0.19	5.1
28	29.2	11.54	11.2	19.73	38.5	93.7	9.2	9.9	0.18	7.3
29	30.0	11.74	19.6	29.58	34.3	90.7	14.7	12.6	0.43	6.2
30	29.9	11.11	22.5	26.70	36.1	91.7	6.4	8.9	0.59	6.3
31	29.9	11.11	13.7	31.60	35.8	89.4	8.3	9.4	0.97	5.9
FEB										
1	29.9	11.11	-	26.5	30.6	85.5	12.3	9.9	0.40	6.3
2	29.9	11.11	-	24.0	33.9	85.8	7.0	4.3	0.19	10.9
3	29.3	11.52	32.3	18.9	37.8	88.8	5.3	4.5	0.24	3.6
4	29.3	11.52	37.2	25.1	41.0	91.2	7.9	5.5	0.23	5.9
5	29.3	11.52	24.9	25.9	35.9	91.1	9.5	7.6	0.73	6.4
6	29.3	11.52	12.6	23.2	42.4	93.6	7.3	7.0	0.32	6.2

Table C-1 (contd)
ANALYTICAL DATA (Sheet 1)

Date 1984	Liquefaction						Hydrotreater			
	Coal Feed		Solv +850°F wt%	Vent Gas		Coal Conversion wt%	Feed +850°F wt%	Underflow +850°F wt%	Vent Gas	
	Moist wt%	Asn wt%		H ₂ S, CO, CO ₂ wt%	C ₁ -C ₅ wt%				H ₂ S wt%	C ₁ -C ₅ wt%
Run 38 FEB										
13	28.6	10.51	-	15.7	27.7	80.9	19.9	21.1	0.55	10.50
14	28.8	11.05	-	31.6	37.3	87.1	26.0	28.3	1.16	13.76
15	28.8	11.05	-	12.0	36.6	90.1	24.6	30.1	0.46	12.03
16	28.7	10.81	-	26.1	37.5	90.0	24.2	22.4	0.37	12.47
17	28.7	10.81	-	33.3	35.4	91.8	10.7	16.8	0.39	16.52
18	29.9	11.09	31.5	28.1	37.9	89.8	15.5	18.8	0.21	14.19
19	28.8	11.58	32.4	27.4	36.0	92.3	10.5	17.5	0.28	9.99
20	28.8	11.58	25.6	32.2	38.8	91.1	12.5	17.3	0.42	10.18
Run 39 FEB										
21	27.6	11.62	22.9	21.7	37.5	92.6	9.5	12.8	0.74	7.45
22	27.6	11.62	29.8	31.6	39.5	86.5	3.7	20.1	0.54	11.30
23	27.6	11.62	34.5	30.9	37.6	86.2	19.8	19.0	1.77	14.25
24	27.8	10.41	33.0	22.2	39.1	87.0	15.0	18.2	0.84	10.71
25	28.1	10.52	30.8	29.8	36.4	85.4	11.0	14.3	0.75	11.47
26	28.1	10.52	34.9	39.5	36.3	87.0	17.7	18.5	0.70	10.98
27	28.1	10.52	34.4	25.9	36.1	86.1	13.2	17.0	0.85	2.89

Table C-2
ANALYTICAL DATA (Sheet 2)

Date	CSD							
	Vacuum Bottoms Feed					Ash Conc	DAR	LDAR
	Ash	Pyr Sol	UC	Viscosity	-850°F	Ash	Ash	Ash
	wt%	wt%	wt%	@500°F cp	Dist wt%	wt%	wt%	wt%
Run 35								
DEC								
1	-	-	-	-	-	32.9	0.5	0.02
2	3.33	94.1	2.57	90	2.6	26.2	5.7	0.02
3	5.71	87.6	6.69	175	3.5	21.8	0.1	0.03
4	7.30	83.9	8.30	280	5.4	29.1	0.03	0.02
5	8.97	83.3	7.73	95	14.2	27.0	<0.01	<0.01
6	12.23	80.1	7.67	170	13.2	29.4	0.01	<0.01
7	11.19	78.4	10.41	205	10.9	33.5	0.07	<0.01
8	12.27	77.2	10.53	385	1.0	33.8	0.53	<0.01
9	11.29	76.5	12.11	400	2.8	27.2	9.03	0.01
10	10.70	78.4	10.90	425	2.9	29.4	0.09	0.01
11	11.93	78.3	9.77	300	6.9	32.9	0.05	<0.01
12	12.51	78.5	8.89	340	5.1	32.8	0.07	0.03
13	11.26	77.2	11.54	350	6.8	29.1	7.75	0.05
Run 36								
DEC								
14	13.31	75.2	11.5	450	5.4	25.4	0.74	<0.01
15	14.04	72.8	13.2	405	6.3	34.2	14.98	<0.01
16	18.25	69.1	12.7	435	15.2	51.3	3.56	0.02
17	14.36	72.5	13.1	440	10.9	26.1	1.35	0.03
18	11.43	77.8	10.8	375	7.9	45.3	0.02	0.02
19	12.11	76.5	11.4	355	9.4	25.7	0.26	<0.01
20	12.20	77.3	10.5	285	8.3	26.2	-	<0.01

C-4

Table C-2 (contd)
ANALYTICAL DATA (Sheet 2)

Date 1984	CSD					Ash Conc Ash	DAR Ash	LDAR Ash
	Vacuum Bottoms Feed							
	Ash	Pyr Sol	UC	Viscosity @500°F	-850°F Dist			
	wt%	wt%	wt%	cp	wt%			
Run 37								
JAN								
11	11.7	75.9	12.4	215	15.8	29.8	0.07	0.03
12	13.2	72.9	13.9	330	11.0	30.0	0.06	<0.01
13	13.3	73.8	12.9	375	8.2	28.5	0.05	0.02
14	12.3	73.5	14.2	360	6.6	28.4	0.69	0.01
15	12.5	74.6	12.9	210	8.0	29.0	<0.01	<0.01
16	13.3	73.7	13.0	280	7.4	30.5	0.60	<0.01
17	14.1	73.5	12.4	290	10.6	33.5	0.61	0.07
18	9.3	64.3	26.4	635	6.1	26.4	0.09	0.06
19	15.2	72.6	12.2	370	12.2	30.3	<0.01	0.02
20	15.3	71.4	13.3	540	8.4	34.1	0.05	<0.01
21	13.9	78.5	7.6	340	7.4	36.6	0.21	<0.01
22	13.8	80.5	5.7	260	10.9	37.4	0.06	<0.01
23	14.1	79.3	6.6	305	20.6	37.5	0.04	<0.01
24	15.3	77.9	6.8	560	7.2	40.2	0.05	0.01
25	13.9	79.7	6.4	390	7.7	38.8	0.06	0.02
26	14.7	78.4	6.9	720	3.8	37.2	0.02	0.01
27	21.3	70.1	8.6	1300	1.4	42.0	0.06	0.04
28	18.2	74.3	7.5	630	6.4	35.1	0.68	0.06
29	16.1	74.4	9.5	430	10.1	30.2	0.16	<0.01
30	15.7	75.5	8.8	390	10.0	35.4	0.11	0.04
31	14.1	75.7	10.2	400	8.8	33.8	0.05	<0.01
FEB								
1	12.0	76.3	11.7	190	13.5	33.6	0.06	<0.01
2	14.4	71.8	13.8	320	7.7	32.1	0.06	<0.01
3	17.9	69.2	12.9	1060	5.4	33.4	0.06	<0.01
4	11.5	81.9	6.6	425	11.6	33.7	<0.01	<0.01
5	10.2	84.4	5.4	370	14.1	30.9	0.11	<0.01
6	15.8	77.0	7.2	335	13.4	34.1	0.03	<0.01

Table C-2 (contd)
ANALYTICAL DATA (Sheet 2)

Date 1984	CSD					Ash Conc Ash wt%	DAR Ash wt%	LDAR Ash wt%
	Vacuum Bottoms Feed							
	Ash wt%	Pyr Sol wt%	UC wt%	Viscosity @500°F cp	-850°F Dist wt%			
Run 38 FEB								
13	3.39	92.0	4.61	25	15.3	28.5	0.03	<0.01
14	9.55	82.1	8.35	95	9.0	32.2	0.05	<0.01
15	14.66	75.5	9.84	285	3.2	33.6	0.65	0.04
16	14.97	75.2	9.83	420	3.4	37.8	8.70	<0.01
17	13.58	78.7	7.72	375	4.4	37.0	0.13	<0.01
18	12.03	79.5	8.37	255	4.6	35.5	0.09	0.01
19	12.13	81.3	6.07	220	6.7	36.3	0.03	<0.01
20	12.38	80.5	7.12	385	4.8	36.5	0.01	0.03
Run 39 FEB								
21	13.22	79.3	7.48	445	5.9	36.8	0.02	0.04
22	10.23	79.8	9.97	500	5.7	32.6	-	-
23	10.59	78.3	11.11	625	5.7	28.1	0.07	0.07
24	9.84	79.4	10.76	490	5.4	25.0	0.07	0.09
25	9.67	78.9	11.43	660	3.4	23.0	0.02	0.08
26	9.46	80.1	10.44	290	6.7	24.0	<0.01	0.01
27	10.22	78.1	11.68	560	5.2	24.2	<0.01	0.03

Table C-3

COMPARISON OF ANALYSES OF IRON OXIDE

RUN	<u>32</u>	<u>37</u>
<u>ANALYSIS</u>		
Moisture, wt %	0.6	2.9
Surface Area, M ² /g	4.5	4.8
Chloride, wt %	0.9	0.7
Iron (Total)	67.3	62.2
Fe ⁺⁺	2.4	2.4
Fe ⁺⁺⁺	64.9	59.8
Other Elements		
Al	0.50	0.55
Ca	0.22	0.07
Cr	0.02	0.04
Mg	0.36	0.33
Pb	0.04	0.03
Ti	0.99	1.10
V	0.07	0.07
Zn	0.06	0.07
Dry Screen (after milling)		
+ 60	0.0	0.8
- 60+100	3.1	3.2
-100+200	84.9*	10.4
-200+325	7.1	67.1
-325	4.9	18.5
TOTAL	100.0	100.0

* Value may be biased due to possible "balling" of the material.

APPENDIX D

Table D-1

ANALYSIS OF STARTING MATERIALS USED IN
BATCH REACTOR COKE-PRECURSOR STUDIES

Material Type	Process Solvent	Process Solvent	Hydrotreater Feed	Hydrotreater Feed	Hydrotreater Feed	Hydrotreater Feed	LDAR	DAR	DAR
Sample Identification	D-3106 3/17/83	#8833 Run 32	#8832 Run 32	#8838 Run 32	#8989 Run 33	#9128 Run 34	#8845 Run 32	#8334 Run 32	Drum M-462 WLV Run 240
Analysis, wt %									
Carbon	88.8	88.6	89.3	89.4	88.1	89.0	90.1	90.2	86.5
Hydrogen	9.27	9.32	7.77	8.56	8.46	8.50	6.42	5.86	5.76
Nitrogen	0.28	0.42	0.68	0.53	0.54	0.65	0.99	1.32	1.82
Sulfur	<0.01	<0.01	0.06	0.02	0.04	0.01	0.14	0.26	0.81
Oxygen (by difference)	1.65	1.66	2.19	1.39	2.02	1.64	2.35	2.30	5.04
Ash	-	-	<0.01	0.1	0.84	-	<0.01	0.06	0.07
-850°F Distillables ¹	95.6	86.8	45.4	70.5	71.9	67.4	6.1	4.4	6.1
Oils									4.6
Asphaltenes									56.2
Preasphaltenes								38.0	39.0
Used in Ref. Test No.	MM-5 E-15B 84A19 84A20	83A7 83A8 83A9 83A10 83A14 83A15	83A7 83A9	84A16	83A13	84A18	84A16	83A8 83A10	MM-5 E-15B 84A19 84A20

¹- 500°F X 0.1 mm distillation with G.C. correction of distillate.

Table D-2

ANALYSIS OF STARTING MATERIALS USED IN
BATCH REACTOR COAL LIQUEFACTION STUDIES

Material Type	Coal Wyoming Subbituminous	Coal Illinois No. 6 Bituminous	Hydro- Underflow	Hydro- Underflow	Hydro- Underflow	LDAR	DAR	LDAR
Sample Identification	Prog. C-23 Aug 83 Shipment	Prog. 83D5 Dec 83 Shipment	# 8836 Run 32	# 8835 Run 32	# 9129 Run 34	#9127 Run 34	#8837 Run 32	#1503 Prog GG5
Analysis, wt %								
Carbon	65.2	67.4	89.4	89.3	89.2	89.5	86.8	90.6
Hydrogen	5.14	5.26	10.48	10.10	9.72	6.54	5.49	7.68
Nitrogen	0.86	1.36	0.15	0.20	0.28	1.07	1.24	0.81
Sulfur	0.98	3.30	<0.01	0.01	0.02	0.55	0.41	0.02
Oxygen (by difference)	18.29	11.49	<0.1	<0.1	0.76	2.32	1.57	0.89
Ash	9.48	10.99	0.09	<0.01	-	-	4.49	-
-850°F Distillable ¹	-	-	83.3	73.0	77.1	10.6	2.5	62.8
% Moisture	26.5	4.93						
Used in Ref. Test #	83E3 84E12 83E5 84E15 83E6 84E16 83E7 84E17 83E8	84E21 84E22 84E23	83E3 83E5 83E6	84E15 84E16 84E17	83E7 83E8	83E8	83E3 83E5 83E6 84E15 84E16 84E17	84E21 84E22 84E23

¹- 500°F x 0.1 mm distillation with G.C. correction of distillate.

Table D-3
ANALYSES OF VARIOUS DISPOSABLE CATALYSTS

Material Type	Iron Oxide	Iron Ore	Mill Scale
Source	KM Mobile Plant	Wyoming	Buffalo, N.Y.
Ref. Prog.	83E1	83E2	83E4
Sample No.	IR-9301	8997	9146
<u>Analysis (-60 mesh fraction) ¹</u>			
Moisture	2.92	-0-	0.20
Loss on Ignition @ 850°F	5.04	0.15	0.11
Apparent Density, g/cc	4.65	4.88	5.21 ³
Surface Area ² , M ² /g	4.8	0.7	<0.1 ³
Bulk Density, g/cc	0.44	-	-
Dry Screen			
+ 60	0.8	1.9	40.6 ⁴
- 60/+100	3.2	24.4	48.2
-100/+200	10.4	20.1	11.2
-200/+325	67.1	14.2	-
-325	18.5	39.4	-
Chloride	0.74	0.04	<0.01
Iron Total	62.2	64.2	72.6
Fe ⁺⁺	2.41	0.9	44.7
Fe ⁺⁺⁺ (diff)	59.8	63.3	27.9
AA Metals			
Al	0.55	0.40	0.066
Ca	0.071	0.22	0.09
Cr	0.035	<0.01	0.10
Mg	0.33	0.16	0.014
Pb	0.03	0.038	<0.038
Si	0.20	3.2	0.31
Ti	1.1	0.072	<0.018
V	0.069	<0.019	0.02
Zn	0.074	0.0028	0.0015

¹ - All units in wt % sample basis unless specified otherwise.

² - By BET method.

³ - Sample was coated with an oily substance interfering with the density and surface area measurement. Reported density obtained after washing with acetone.

⁴ - Material tended to agglomerate badly and the dry screen analyses is somewhat questionable.