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**OPTIMIZATION OF REACTOR CONFIGURATION
IN COAL LIQUEFACTION****FIFTH QUARTERLY REPORT FOR THE PERIOD****1 OCTOBER - 31 DECEMBER 1992****DISCLAIMER**

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ABSTRACT

This quarterly report covers activities of Optimization of Reactor Configuration in Coal Liquefaction during the period October 1 - December 31, 1992, at Hydrocarbon Research, Inc. in Lawrenceville and Princeton, New Jersey. This DOE contract period is from October 1, 1991 to September 30, 1993.

The overall objective of the program is to achieve a new approach to liquefaction that generates an all distillates product slate at reduced cost of about \$25 per barrel of crude oil equivalent.

The quarterly report covers work on Laboratory Support, Laboratory Scale Operations, Technical Assessment, and Project Management.

SUMMARY

Work undertaken in this fifth quarter concerned with completing the analyses of recovered catalysts from the previous fixed bed tests and the planning of microautoclave tests to evaluate the three-stage and interstage stream reconcentration concepts.

INTRODUCTION

The studies conducted in this the fifth quarterly report of Contract No. DE-AC22-91PC-91052 is reported by task and in sequence.

Task 1 is the Project Work Plan and is complete. Task 2 is for Laboratory Support which includes feed and product characterization and general analytical support for Task 3. Task 3 is for Laboratory Scale Operations evaluating three reactor configurations: fixed bed reactor as a 'finishing' reactor, three-stage close-coupled Back-Mixed Reactor System, and interstage product stream concentration. Task 4 is the Technical Assessment, including modelling, design and economics. Task 5 is Project Management.

The contract period is October 1, 1991 to September 30, 1993.

TASK 2 - LABORATORY SUPPORT

Three intermediate coal conversion slurry samples were collected at the end of a bench scale run (Run 227-76) which was conducted under the Catalytic Two-Stage Liquefaction program using Illinois No. 6 coal. These samples were taken after the second stage, while the unit was cooling down from reaction conditions to relatively mild reaction severity. These samples are:

Sample E1	low conversion sample collected when the 1st and 2nd reactor temperature was at 750 and 600°F, respectively.
Sample E2	medium conversion sample collected when the 1st and 2nd reactor temperature was at 750 and 750-600°F, respectively.
Sample E3	higher conversion sample collected when both reactor temperature was at 750°F.

Each slurry samples was subsequently separated into a solid and a liquid fraction using a pressure filtration system. Analyses of the pressure filter cake and liquid from the slurry sample E3 are given in *Table 1* and *Table 2*.

TABLE 1
ANALYSIS OF PRESSURE FILTER CAKE
(Run 227-76 Period E3)

<u>Pressure Filtration:</u>	
Filter Cake, W%	29.42
Filter Liquids, W%	70.58
 <u>Elemental Analysis, W%</u>	
Carbon	16.0
Hydrogen	6.25
Nitrogen	1.70
Sulfur	0.85
SO ₃ -Free Ash	13.48
 ASTM Ash	 13.91
S in Ash	1.20
 <u>Quinoline Filtration, W%</u>	
Insolubles (QI)	36.77
Ash in QI, W% PFC	14.60
S in QI Ash	1.03
 <u>Coal Conversion, W%</u>	
	88.9

TABLE 2
ANALYSIS OF PRESSURE FILTER LIQUID
(Run 227-76 Period E3)

API Gravity	1.3
<u>Elemental Analysis, W%</u>	
Carbon	89.51
Hydrogen	8.84
Nitrogen	0.54
Sulfur	0.22
<u>Boiling Point Distribution, W%</u>	
IBP	583°F
IBP-650	5.35
650-850	42.68
850-975	12.95
975+	38.27
Loss	0.75

TASK 3 - LABORATORY SCALE OPERATIONS - REACTOR CONFIGURATION

A. MICROAUTOCLAVE SCREENING TESTS

Sixteen microautoclave tests were designed to evaluate the reactivity of the partially converted filter cake and liquid.

EVALUATION OF SOLID FROM RUN 227-76 PERIOD E3

To ensure the consistency of the charge (solid) to the microautoclave, the PFC sample (Run 227-76 Period E3) will be washed with THF to remove the residual oil on the cake.

The analysis of PFC is given in *Table 1*. In spite of the relatively low severity (1st stage: 750°F; 2nd stage: 750°F) at which this sample was taken, the coal conversion was fairly substantial around 88.9 W%. The observed high conversion was partly due to the high reactivity of Ill. #6 coal and, perhaps, the more dominant factor was the relatively wide age distribution of solids in the well mixed reactor.

A matrix of 7 tests are designed to determine the reactivity of the THF washed filter cake. The test conditions for the coal reactivity are listed below:

COAL REACTIVITY EVALUATION Test Conditions

THF Washed Cake	2 gm
Solvent	8 gm
(PFL Run 227-76 Period 13)	
Catalyst	2 gm
(Recovered 2nd stage AO-60 Catalyst)*	
Pressure, psig	2000

Test No.	Temperature (°F)	Time (min)
AC-01	750	15
AC-02	750	30
AC-03	800	15
AC-04	800	30
AC-05	825	15
AC-06	825	30
AC-07	800	60

* THF Washed, 2nd stage catalyst recovered from Run 227-76

The analytical procedure will be similar to that of the YY-series. The reactivity will be determined by coal conversion, residuum conversion and H/C ratio of the liquid product.

EVALUATION OF PFL AND RESIDUUM FROM RUN 227-76 PERIOD E3

Pressure Filtration of CAS bottom from Run 227-76 Period E3 yielded 70.6 W% of filter liquid. The 975°F+ fraction of PFL from Period E3 was about 8 W% higher than that of FPL from the last three periods (Period 11 to 13) of the run. Analysis of the PFL sample (Period E3) is shown in *Table 2*.

In order to alter the composition of the residuum fraction of the oil sample, diluent will be used in some tests. The effect of three different diluents will be evaluated. These diluent are:

Diluent A - 500°F+ material from CAS Bottom
Diluent B - IBP-850°F material from PFL (Period E3)
Diluent C - 850°F+ material from the PFL (Period E3)

The following tests will be conducted to determine the reactivity of residuum from Ill. #6 coal.

EVALUATION OF PFL TEST CONDITIONS

Test No.	PFL gm	Diluent gm	Cat./Oil	Temp. F	Time min
AC-08	2.0	0	1/1	800	30
AC-09	2.0	0	0.5/1	800	30
AC-10	2.0	0	1/1	825	30
AC-11	2.0	0	1/1	800	60
AC-12	1.33	A/0.67	1/1	800	30
AC-13	1.33	B/0.67	1/1	800	30
AC-14	1.33	C/0.67	1/1	800	30
AC-15	1.33	C/0.67	1/1	825	30
AC-16	2.0	0	0	800	30

Note: Recovered Catalyst (THF Washed) from the 2nd stage of Run 227-76.

Performance of the microautoclave tests will be measured by residuum conversion, HDN, HDS and hydrogen content of the Product.

B. SMALL FIXED BED REACTOR TESTS

Two recovered catalyst samples, ACL-1 Period 4, were analyzed to determine the degree of carbon and ash build-ups in the first small scale fixed bed test. Results of the analysis are given in *Table 3*.

A small fraction of the supported catalyst were broken during unpacking. As a result, the recovered catalyst contained 3.6 and 8.02 W% of fines (-20 mesh) as shown in *Table 4*. The remaining -20 mesh materials consisted of coal ash and carboneaous materials. (unconverted coal or coke). The carbon content of the recovered catalysts 12-13.0 W% were relatively less than typical recovered catalyst liquifying Black Thunder coal in an ebullated bed environment.

TABLE 3
INSPECTIONS OF RECOVERED CATALYSTS (ACL-1, PERIOD 4)
(Shell 317 1/20" TriCoke)

	<u>Fresh Catalyst (Presulfided)</u>	<u>Recovered Catalyst</u>	
		Reactor #1	Reactor #2
Solvent Extraction			
Toluene Soluble	34.9	31.6	28.9
Quinoline Soluble		36.9	35.4
On Toluene Washed Catalyst (W% Toluene Washed Catalyst)			
Screen Analysis			
+ 20 Mesh	--	89.50	85.95
20 - 100 Mesh	--	7.18	11.29
- 100 Mesh	--	2.75	2.58
ASTM Ash (+ 20 Mesh Fraction)		80.2	78.1
Molybdenum Content			
+ 20 Mesh	7.90	8.68	12.51*
20 - 100 Mesh	--	3.77	5.25
- 100 Mesh	--	0.63	1.60
Elemental Analysis (+ 20 Mesh Fraction)			
Carbon	3.03	12.28	12.59
Hydrogen	0.85	1.09	0.93
Sulfur	7.35	7.45	7.04
Nitrogen	0.07	0.16	0.15
H/C	--	1.06	0.89

* Value too high; to be re-analyzed.

TABLE 4
COMPOSITION OF FINES IN TOLUENE WASHED
RECOVERED CATALYSTS

Weight % of Toluene Washed Recovered Catalyst

	Reactor #1		Reactor #2	
	<u>Catalyst*</u>	<u>Coal Residual</u>	<u>Catalyst*</u>	<u>Coal Residual</u>
20 - 100 Mesh	3.42	3.76	7.50	3.79
- 100 Mesh	<u>0.22</u>	<u>2.53</u>	<u>0.52</u>	<u>2.06</u>
TOTAL	3.64	6.29	8.02	5.85

* Estimated W% of catalyst using molybdenum as "tie" element.

TASK 4 - TECHNICAL ASSESSMENT

No activity undertaken during this reporting report.

TASK 5 - PROJECT MANAGEMENT

An updated work schedule is attached, *Figure 1 and 2*.

FIGURE 1

FIGURE 2

END

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