**TWO-STAGE, CLOSE COUPLED
CATALYTIC LIQUEFACTION OF COAL
SEVENTH QUARTERLY REPORT FOR THE PERIOD
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ABSTRACT

This program report covers activities of the Two-Stage, Close-Coupled Catalytic Liquefaction of Coal program during the period April 1,-June 30, 1990, at Hydrocarbon Research, Inc.

Laboratory scale testing of an OTISCA micronized, cleaned coal, an unconverted coal, and cleaned coal reject were completed. The ultimate convertibility was examined and also the reactivity of the two convertible species.

Run CC-8, the evaluation of an OTISCA cleaned coal and CC-9, the evaluation of delayed coking were completed.

An updated schedule of remaining tasks is included.

SUMMARY

During the second quarter of 1990, experimental studies were carried out in the microautoclave and Bench Units. Updating of the economics of two-stage processing continued as did process modelling activities.

Bench-scale operations consisted of the completion of Run 227-66 (CC-8), an OTISCA cleaned coal evaluation, and Run CC-9, a solids separation study of delayed coking. The OTISCA Cleaned Coal Evaluation and Delayed Coking Trials were successfully concluded.

The economics of Eastern U.S. bituminous coal liquefaction were updated to a 1991 basis. For an 8400 ton/day plant, an investment of 1.6 billion is estimated.

INTRODUCTION

The studies conducted in this the seventh quarterly report of Contract No. DE-AC22-88PC88818 are reported by task and in sequence other than the experimental modelling tests which are reported under Task 2 - Laboratory Studies.

Task 1 is the Management Plan and is complete. Task 2 is for Laboratory Studies and includes microautoclave and microreactor work. Task 3 is for Bench-Scale studies and in this report covers results of Runs CC-8, and 9. Task 4 is for Mini PDU Scale-up. Task 5 is the Technical Assessment, including Modelling and Economics. Task 6 is Administration.

The contract period is from October 1, 1988, to September 30, 1991.

TASK 2 - MICROAUTOCLAVE STUDIES

During the second quarter of 1990 the following programs were carried out in the microautoclave:

COAL EVALUATIONS

Table 1 summarizes the results of coal evaluations in the microautoclave of OTISCA Micronized Cleaned Illinois No. 6 coal that was used as feed for CTSL Bench Unit 227-66 (CC-8), and micronized Run-of-Mine coal that was also used in that run. The table also includes results for microautoclave tests on: (1) the refuse from OTISCA cleaning operation, (2) the raw coal (not micronized and ground to -70 mesh at the HRI R&D center) and (3) earlier tests with Oil-agglomeration cleaned Illinois Coal supplied by Homer City.

Cleaned OTISCA coal (3.31 W% ash) and the micronized Run-of-Mine coal (10.57 W% ash) had very nearly the same coal conversions in the microautoclave catalytic test, around 93.4% of MAF coal. However, in the thermal test, with no catalyst, the conversion for the Run-of-Mine coal was only 3% lower than in the catalytic test, while for the cleaned coal there was a considerably greater difference in conversions, around 8%. This pattern was noted for the earlier microautoclave tests with Homer City Oil-agglomeration samples, indicating that some catalytically effective components are contained in the mineral matter removed in the cleaning operations.

The refuse from OTISCA's cleaning operation (64.54 W% ash) gave a comparatively low conversion in catalytic microautoclave evaluation, at 81% of MAF coal, and a relatively small difference between the catalytic and thermal test results, 4.8%. These results indicate that the cleaning operation does remove a high proportion of the less reactive coal components, although this is not necessarily a very appreciable factor. The ash contents of the various streams involved in the cleaning indicate that cleaned fraction amounts to about 88% of the coal feed, and contains about 95% of the organic content contained in the coal feed. The microautoclave results with the uncleaned coal and the cleaning refuse indicates that the low reactivity component (nominally unreactive) removed corresponds to about 14-17% of the MAF coal in the refuse, or to about 0.65-0.75% of the original MAF coal. Consequently, the cleaned portion should contain less inert coal in this proportion, so that coal conversions in comparable liquefaction operations would be about 0.7% higher with the cleaned coal than with the Run-of-Mine coal.

SOLVENT EVALUATION

The solvent quality of HRI 5757, 650°F+ Heavy Distillate Oil from Wilsonville, has been evaluated in the Microautoclave using Illinois No. 6 Coal, and Black Thunder Mine (Wyoming) Coal. This oil will be used as start-up and make-up oil in future CTSL Bench Unit operations. The solvent quality evaluation is a thermal 30 minute test at 750 F, under 2000 psig of nitrogen, using 2 grams of solvent per gram test coal.

The performance of the HRI 5757 solvent (61.9% conversion for Illinois Coal, and 58.7% conversion for the Black Thunder coal) was in the general range of those solvents used previously in the Bench Unit Tests as summarized in the following Table:

Solvent	Used in Runs	Coal Conversion, %	
		Illinois	Black Thunder
HRI 5757	---	61.9	58.7
HRI 5443	CC-1	57.8	56.1
HRI 5648	CC-2,3,4,6,7,8	70.1	61.5
L-742	CC-5	67.4	65.9

PROCESS MODELLING

Coal Conversion

A series of microautoclave tests were carried out using the solids produced during CTSL operations in Run 227-66 (CC-8) with the OTISCA cleaned and Run-of-Mine coal, and feed coals to those CTSL operations. The objective of these tests was to characterize the original coal in terms of various species of differing reactivity. Tables 2 and 3 summarize the results of the microautoclave tests. The microautoclave test conditions that were used are approximately of the same reaction severity as the second stage conditions in the CTSL Bench unit.

The additional processing of the product solids caused additional conversion of the coal, with the solids product from the CTSL operation on the cleaned coal going from a conversion level of 93.6% to as much as 96.3%, and the product from CTSL R-O-M operations increasing from 90.9% converted to 95.8% converted. Equivalent tests with the original coal feeds gave as much as 95.0% and 94.3% conversions for the cleaned and R-O-M coals, respectively.

The small change in the conversion levels with an increase in test time from 30 to 60 minutes indicates that even at very long test times not all of the coal can be converted. The generally lower conversions obtained with the Run-of-Mine coal derived material than with the cleaned coal derived material indicates that there is somewhat more of this unconvertible portion in the Run-of-Mine coal. Extrapolation of the microautoclave results with the CTSL product solids, based on batch operation first order kinetics, indicates that the unconvertible proportion amounts to about 4% of the Run-of-Mine coal and 3% of the cleaned coal.

The conversion levels in the microautoclave with the CTSL feed coals, only slightly below the combined CTSL-microautoclave results using the CTSL product solids, indicates that the portion of coal that had been converted in CTSL operations was of very much higher reactivity than demonstrated by the reactive portion remaining in the CTSL product solids. Preliminarily, the average of the microautoclave test results indicates that the Run-of-Mine coal consists of 88% of a higher reactivity component, 8% of the low reactivity component, and 4% inert coal, while for the cleaned coal these proportions are 89%, 8%, and 3%, respectively. The estimates

from the individual tests are $\pm 2.5\%$ of these averages. The relative reaction rate of the high reactivity component is poorly defined by these microautoclave test results.

Kinetics of Secondary Residual Oil Conversion

A test program in the microautoclave has been started to define the reactivity of various components of the residual oil produced and recycled during CTSL Bench Unit operations. Previous microautoclave tests have indicated that the best fit of the kinetics of the reaction of the residual oil is of a model of at least two residual oil components, of differing reaction rates, each following first order kinetics. The current program will solvent fractionate the residual oil into heptane soluble, heptane insoluble, cyclohexane soluble, and cyclohexane insoluble fractions and test each of these solvent fractions to establish which of them individually follow first order kinetics.

The test oils are to be derived from a fresh sample of the Recycle Solvent (PFL) from Run 227-55 (CC-1) which was used in the earlier microautoclave tests. To this time, eight microautoclave tests have been completed on the unfractionated PFL, and the analytical work on their products is in progress, to serve as a reference basis for the further work with the solvent-fractionated materials. Solvent fractionation of the residual oil components is also in progress.

TABLE 1

**MICROAUTOCLAVE TEST RESULTS WITH
CLEANED AND UNCLEANNED ILLINOIS NO. 6 COAL**

		Microautoclave Test Conditions - 30 minutes, 800°F, 2000 psig H ₂ 4 grams solvent/gram coal 1 gram catalyst/gram coal (catalytic tests)				Solvent - HRI 5198 Wilsonville Heavy Distillate Catalyst - DMS Presulfided Shell S-317 (HRI 5359P)				HOMER CITY (Oil Agglomeration)			
Source		OTISCA											
Processing		Cleaned	Cleaned	R-O-M	Refuse	R-O-M	R-O-M	Cleaned	R-O-M	Cleaned	R-O-M		
Ash, W%		1.26	3.31	10.57	64.54	9.95	4.53	15.21					
Size		< --- Micronized --- >				Ground -70 Mesh							
<u>Microautoclave Coal Conv., W% MAE</u>													
Thermal Test		80.0	85.7	90.0	76.4	74.2	76.3	88.2					
Catalytic Test		94.6	93.4	93.3	81.1	93.2	93.7	93.4					
<u>Microautoclave 975°F Conv., W% MAE</u>													
Thermal Test		62.2	60.5	52.7	51.6	46.6	56.0	52.2					
Catalytic Test		67.4	61.4	64.6	73.8	62.0	61.9	73.7					
<u>Microautoclave 975°F Yield, W% MAE</u>													
Thermal Test		17.8	25.2	37.3	24.8	27.6	20.3	36.0					
Catalytic Test		27.2	32.0	28.7	7.3	31.2	31.8	19.7					

TABLE 2

TESTING OF MICROAUTOCLAVE PROGRAM TO MODEL COAL CONVERSION

Testing Product Solids from CTSI Bench Unit Operations
(Run 227-66 (CC-1) Using OTISCA Illinois No. 6 Coal Samples)

Microautoclave Run	65	67	69	71
Microautoclave Feed	--Period 7C Product--		--Period 14 Product--	
Bench Unit Feed	---Cleaned Coal---		---R-O-M Coal---	
Microautoclave Conditions				
Time, Minutes	30	60	30	60
Temp., °F	825	825	825	825
Pressure, psig	2,000	2,000	2,000	2,000
Gas	H ₂	H ₂	H ₂	H ₂
Feed, grams	2.000	2.000	2.001	2.002
Catalyst HRI	5359P	5359P	5359P	5359P
Grams	2.00	2.00	2.00	2.00
Solvent	L-721 ¹	L-721	L-721	L-721
Grams	8.02	8.02	8.00	8.00
Loss from Autoclave, grams	0.19	0.90	0.32	2.42
Recovered Solids ² , grams	1.5366	1.4829	1.5287	1.2440
W% of Feed	76.82	7.139	76.40	62.18
Recovered Solvent, grams	8.25	7.43	8.05	6.51
THF Content, W% (TGA Anal)	3.4	3.33	2.46	2.24
Residual Oil, W% (TGA)	2.38	1.83	1.79	1.42
Solids Plus THF Free Oil, gms	9.79	10.03	9.58	7.75
Loss from Charge, grams	0.23	-0.01	0.42	2.25
Microautoclave Feed Analysis				
Ash, W%	-----36.09-----		-----56.42-----	
Product Solids ³ Analysis				
Ash, W%	44.40	49.55	71.29	73.85
Ash Balance, W%	94.50	98.05	96.45	81.38
Conversion, % of Microautoclave MAF Feed	29.28	42.53	48.14	54.16
Degree of Conversion, Percent of Original MAF Coal				
Microautoclave Feed	-----93.59-----		-----90.88-----	
Microautoclave Product	95.48	96.31	95.24	95.81
Residual Oil Recovered, grams	0.196	0.135	0.146	0.092
Percent of Coal Converted	5.23	24.8	35.0	18.5

¹ L-721 - Wilsonville Heavy Distillate

² THF Washed

³ Products taken to have 2.2 W% sulfur in ash.

TABLE 3
RESULTS OF MICROAUTOCLAVE PROGRAM
TO MODEL COAL CONVERSION

Testing OTISCA Illinois No. 6 Coal Samples

Microautoclave Run	66	68	70	72
Feed	-----HRI 5738----- -----Cleaned Coal---		-----HRI 5739----- -----R-O-M Coal-----	
Microautoclave Conditions				
Time, Minutes	30	60	30	60
Temp., °F	825	825	825	825
Pressure, psig	2,000	2,000	2,000	2,000
Gas	H ₂	H ₂	H ₂	H ₂
Feed, grams	2.001	2.002	2.002	2.002
Catalyst HRI	5359P	5359P	5359P	5359P
Grams	2.01	2.00	2.02	2.00
Solvent	L-721 ⁴	L-721	L-721	L-721
Grams	8.03	8.00	8.00	8.00
Loss from Autoclave, grams	0.45	1.04	0.90	0.82
Recovered Solids ⁵ , grams	0.2061	0.2209	0.3374	0.3275
W% of Feed	10.34	11.03	16.86	16.36
Recovered Solvent, grams	9.08	8.33	8.39	8.00
THF Content, W% (TGA Anal)	4.0	1.78	3.08	3.82
Residual Oil, W% (TGA)	5.96	7.47	4.31	4.33
Solids Plus THF Free Oil, gms	9.29	8.55	8.37	8.33
Loss from Charge, grams	0.74	1.45	1.27	1.67
Microautoclave Feed Analysis				
Ash, W%	-----3.55-----		-----10.57-----	
Product Solids ⁶ Analysis				
Ash, W%	39.29	51.80	65.29	68.15
Ash Balance, W% ⁷	116.41	163.81	104.13	105.50
Conversion, % of Microautoclave MAF Feed	93.62	94.98	93.86	94.32
Degree of Conversion, Percent of Original MAF Coal				
Microautoclave Feed	-----0.00-----		-----0.00-----	
Microautoclave Product	93.62	94.98	93.56	94.31
Residual Oil Recovered, grams	0.541	0.622	0.362	0.346
Percent of Coal Converted	30.0	33.9	21.6	20.4

4 L-721 - Wilsonville Heavy Distillate

5 THF Washed

6 Products taken to have 2.2 W% sulfur in ash.

7 Excess ash taken to be catalyst with 82.25% ash and 17.75% organics.

TASK 3 - BENCH SCALE STUDIES

RUN 227-66. CC-8

On March 20, 1990, testing of the OTISCA coal was resumed following a three day trial in run 227-65 that was terminated by a high pressure drop in the first stage. Run 227-66 continued for 15 days, operating with cleaned (3.5%), and uncleaned (10.6%) coal.

Water from the 35% coal/water slurry was removed in a batch slurry drying process by the addition of recycle solvent and driving off the water at 275°F with a nitrogen sparge. Moisture remaining in the feed slurry averaged 1 to 2%. A higher oil/dry coal solids ratio (1.7) was required to avoid circulation problems. A ratio of about 1.17 is used in most other operations.

PERFORMANCE AND RESULTS

Table 6 summarizes the operating results in Run CC-8, with a comparison with the results obtained in Run CC-6 with coal cleaned by oil agglomeration. For the first condition of Run CC-8 (227-66) coal conversion was about 92% of MAF coal, which was 2.5% lower than the conversion noted above in the first effort in Run 227-65 with the same operating conditions and proportion of oil-free recycle solvent. Possibly, the more strenuous drying effort in Run 227-66, contributed to this lower conversion level either by a detrimental impact upon the conversion characteristics of the coal, or by a favorable impact upon coal conversion of the higher water vapor pressure in the reactor environment during Run 227-65 consequent to the high water content of the slurry being fed to the reactor.

The second condition of Run CC-8 (227-66), with the major part of the recycle solvent being solids containing Topped Separator Bottoms product slurry, coal conversion was about 1.5% higher than obtained with the solids free recycle solvent. It is judged that this latter operation, which was maintained for only two days (from the end of Period 5 through Period 7), had not completely equilibrated in this regard and a somewhat higher conversion level would be obtained upon equilibration. Also note the higher distillate/lower resid yield with solids recycle. The final operation of Run CC-8 (227-66) with micronized Run-of-Mine coal had a coal conversion about 2.5% lower than obtained in the earlier comparable operation with the cleaned micronized coal.

The C₄-975°F distillate yield during Run CC-8 (227-66) operations was relatively low, at 69-74% of MAF coal. In part this was because of the low coal conversions obtained in this run, but also because of relatively high water yields that were obtained, about 3 W% of MAF coal higher than usually obtained with the Illinois coals. This difference was unexpected since the ultimate analysis oxygen-by-difference value for the micronized cleaned coal was not exceptionally high, at 11.3 W% compared to 10.1-11.4 W% for other cleaned Illinois coals. The exceptionally high amount of water involved in the overall operation of Run CC-8 (227-66), with the gross amount of water handled amounting to about 20 times the

apparent net water formation, was a considerable problem, although it was mitigated by basing the yield calculations on the analyzed water content of the feed slurry to the high pressure system. Further efforts are being made at defining the product distribution incorporating an oxygen balance technique.

TABLE 4
FEED COAL ANALYSES FOR RUN 227-66 (CC-8)

RUN 227-66 FEED: OTISCA PROCESSED ILLINOIS NO. 6 COAL

	5738	5739
Feed Designation, HRI No.		
Type		
Feed for Periods	Cleaned 1-7	Uncleaned 8-15
<u>Moisture Content</u>		
As Received Slurry, W%	67.22	67.01
Analyzed Sample, W%	1.25	1.61
<u>Proximate Analysis, W%</u>		
Moisture, W%	61.86	
Volatile Matter, (MAF Basis)	44.78	43.91
Fixed Carbon (MAF Basis)	55.22	56.09
<u>Ultimate Analysis (W%, Dry Basis)</u>		
Carbon	75.87	69.27
Hydrogen	5.17	4.53
Sulfur	2.65	3.53
Nitrogen	1.52	1.34
Chlorine	0.04	0.07
Ash	3.46	10.57
Oxygen (by difference)	11.33	10.69
Oxygen (Unterzaucher)	13.24	16.02
<u>Sulfur Forms (W%, Dry Basis)</u>		
Sulfate	0.07	0.11
Pyrite	0.60	1.28
Organic	2.07	1.97
Heating Value, Btu/lb (Dry)	13124	12303
Btu/Lb MAF	13594	13757
<u>Mineral Analysis of Ash (W%, Ignited)</u>		
Silica SiO ₂	33.50	44.57
Alumina, Al ₂ O ₃	16.50	19.79
Titania, TiO ₂	1.90	0.78
Ferric Oxide, Fe ₂ O ₃	33.16	22.40
Lime, CaO	5.98	5.00
Magnesia, MgO	1.20	0.88
Potassium Oxide, K ₂ O	1.30	1.80
Sodium Oxide, NaO	0.44	0.42
Sulfur Trioxide, SO ₃	5.20	3.43
Phosphorous Pentoxide, P ₂ O ₅	0.10	0.70
Strontium Oxide, SrO		0.03
Barium Oxide, BaO		0.05
Manganese Oxide, Mn3O4		0.01
Undetermined	0.76	1.14

Continued.....

TABLE 4 (continued)

FEED COAL ANALYSES FOR RUN 227-66 (CC-8)

RUN 227-66 FEED: OTISCA PROCESSED ILLINOIS NO. 6 COAL

Feed Designation, HRI No.	5738	5739
Type Feed for Periods	Cleaned 1-7	Uncleaned 8-15
<u>Particle Size Distribution, W%</u>		
> 11.5 microns	4.9	9.7
11.5	3.7	1.8
10.5	1.6	5.2
9.5	2.4	3.3
8.5	5.2	4.7
7.5	4.8	6.6
6.5	7.8	8.85
5.5	10.3	8.35
4.5	11.3	9.9
3.5	12.7	11.5
2.5	14.0	12.85
1.5	17.2	13.35
0.5	4.8	3.85
<u>Microautoclave Results*</u>		
Thermal Coal Conversion	85.7	90.0
Catalytic Coal Conversion	93.4	93.3

- * 1989 tests - 800°F, 30 minutes, 2000 psig H₂,
4/1(1) Solvent/Coal/Shell(Shell S-317 Catalyst)
Solvent - Wilsonville coal liquefaction distillate product.

TABLE 5
RUN 227-66 OPERATING SUMMARY
BENCH-SCALE TWO STAGE COAL LIQUEFACTION

COAL: Illinois No. 6 - Periods 1-7C: OTISCA Cleaned (HRI-5738)
 - Periods 8A-9C: OTISCA Uncleaned (HRI-5742)
 - Periods 10A-15C: OTISCA Uncleaned (HRI-5739)

CATALYST: Both Stages: Shell S-317 NiMo 1/32" (HRI-5394)

Period Number	5	7	14
Hours of Run (End of Period)	120.	168.	335.
Stage 1 Catalyst Age, Lb Dry Coal/Lb Cat	143.	198.	418.
Coal Treatment	-----Cleaned-----		R-O-M
Moisture in Coal/Water Slurry, W%	63.19	65.51	67.20
Moisture to Reactor, W% of Dry Coal	1.00	1.38	0.83
<u>Temperatures, °F</u>			
First Stage Reaction	750.	775.	750.
Second Stage Reaction	798.	806.	800.
Slurry Mix Tank	187.	208.	265.
Slurry Drying Max Temp.	246	247	277
<u>Pressures, PSIG</u>			
Unit Back Pressure	2799.	2799.	2795.
<u>Space Velocities</u>			
Dry Coal Sp. Vel. - Stg. 1, Lb/Hr/Ft ³ (catalyst)	45.1	41.1	52.6
<u>Recycles, W% of Dry Coal</u>			
Pressure Filter Liquid	190.1	54.1	168.8
PFL to Buffer - Stage 1	4.4	4.7	2.8
PFL to Buffer - Stage 2	4.0	4.2	2.9
Separator Bottoms	0.0	205.7	0.0
Make-Up Oil (HRI 5648)	0.0	0.0	7.9
Oil/Solids in Feed Slurry	1.98	2.15	1.77
Total Material Recovery (Gross)	97.22	98.83	98.13
<u>Estimated Normalized Yields, W% Dry Fresh Feed</u>			
C ₁ -C ₃ in Gases	6.48	8.19	4.94
C ₄ -390°F in Liquids	17.21	21.06	16.15
390-650°F in Liquids	30.35	38.72	26.00
650-975°F in Liquids	21.03	11.59	17.15
975°F+ Residual Oil	3.89	1.40	6.66
Unconverted Coal	7.84	6.43	9.56
Ash	3.31	3.31	10.57
Water	13.39	13.37	11.67
CO	0.00	0.00	0.00
CO ₂	0.01	0.01	0.06
NH ₃	1.61	1.66	1.24
H ₂ S	2.20	2.24	2.71
Total (100 + H₂ Reacted)	107.23	107.99	106.71
<u>Process Performance</u>			
C ₄ -975°F Distillates, W% of MAF Coal	69.4	73.8	65.8
975°F+ Conversion, W% MAF	88.1	91.9	82.0
Coal Conversion, W% MAF	91.9	93.3	89.3
HDS, W%	78.3	79.5	72.2
HDN, W%	87.5	89.9	76.3

TABLE 6

**CTSL PERFORMANCE OF OTISCA MICRONIZED
AND OIL AGGLOMERATION CLEANED ILLINOIS NO. 6 COAL**

BENCH-SCALE TWO STAGE COAL LIQUEFACTION

Run Coal Treatment Condition Period Number	CC-8 Micronized				CC-6 Oil Agglomeration			I-19 Heavy Media Cleaned	
	Cleaned 5	ROM 14	ROM 7	ROM 12	Cleaned 4	ROM 9	ROM 12	Cleaned 10	Cleaned 8
Run CC-8: OTISCA Micronized Cleaned Coal, Periods 1-7									
OTISCA Micronized Run-of-Mine Coal, Periods 8-14									
Run CC-6: Bechtel Oil Agglomerated Cleaned Coal, Periods 1-9									
Run-of-Mine Coal, Periods 10-12									
Run I-19: Bituminous Coal Research Heavy Media Cleaned Coal									
Run I-27: Bituminous Coal Research Heavy Media Cleaned Coal									
Demonstration Run with Solvent Fractionation for Bottoms Extraction									
Hours of Run (End of Period)	120.	335.	168.	285.	96.	213.	285.	240.	192.
Sig. 1 Catalyst Age, Lb Dry Coal/Lb Catalyst	143.	418.	198.	319.	106.	233.	319.	290.	209.
Moisture in Coal/Water Slurry, W%	63.19	67.20	65.51						
Moisture to Reactor, W% of Dry Coal Feed	1.00	0.83	1.38	3.23	6.81	6.81			
Temperature, °F									
First Stage Reaction	750.	750.	775.	749.	751.	753.	749.	751.	753.
Second Stage Reaction	798.	800.	806.	812.	800.	811.	812.	800.	807.
Slurry Mix Tank	187.	265.	208.	283.	191.	299.			
Slurry Drying Mix Temp	247	287	247	2463.	2521.	2524.	2463.	2488.	2480.
Unit Back Pressure, PSIG	2799.	2795.	2799.						
Space Velocity Stage									
Lb Dry Coal/hr/FT ³ Catalyst	45	53	41	46	44	41	46	44	43

Continued.....

TABLE 6 (continued)
CTSL PERFORMANCE OF OTISCA MICRONIZED
AND OIL AGGLOMERATION CLEANED ILLINOIS NO. 6 COAL
BENCH-SCALE TWO STAGE COAL LIQUEFACTION

Run Coal Treatment Condition Period Number	CC-8		CC-6		ROM		Oil Agglomeration		ROM		I-19		I-27	
	5	7	14	4	9	12	10	8	10	8	10	8	10	8
Recycles, W% of Dry Coal	198.5	63.0	174.5	177.7	131.0	114.3	194.0	149.0						
Pressure Filter Liquid	0.0	205.7	0.0	0.0	0.0	0.0	0.0	0.0						
Topped Separator Bottoms	0.0	0.0	7.9	2.0	4.2	4.6	0.0	0.0						
Make-Up Oil	1.98	2.15	1.77	1.69	1.23	1.08	0.0	1.17						
Oil/Solids in Feed Slurry	97.22	98.83	98.13	99.35	96.97	100.51	100.08	97.37						
Material Recovery, W%														
Normalized Yields, W% of Dry Coal														
C ₁ -C ₃ in Gases	6.48	8.19	4.94	7.57	7.28	7.57	5.78	6.76						
C ₄ -390°F in Liquids	17.21	21.06	16.15	19.82	22.30	17.63	17.13	17.75						
500-650°F in Liquids	30.35	33.85	26.00	33.49	33.07	29.15	33.07	41.21						
650-850°F in Liquids	21.03	11.58	17.15	18.19	17.32	11.57	18.44	15.91						
975°F+ Oil	3.89	1.39	6.66	1.94	4.88	5.99	8.16	2.94						
Unconverted Coal	7.84	6.43	9.56	5.28	4.43	5.34	3.75	3.63						
Ash	3.31	3.31	10.57	4.62	4.62	15.21	5.33	5.77						
Water	13.39	13.36	11.67	12.04	8.96	9.59	11.27	9.37						
CO	0.00	0.00	0.00	0.04	0.05	0.07	0.24	0.04						
CO ₂	0.01	0.01	0.06	0.04	0.04	0.04	0.08	0.00						
NH ₃	1.61	1.66	1.24	1.67	1.56	1.35	1.49	1.62						
H ₂ S	2.20	2.24	2.71	2.52	2.51	2.74	2.34	2.51						
Total (100 + H ₂ Reacted)	107.32	107.99	106.71	107.23	106.99	106.25	107.07	107.19						
Process Performance, W% of MAF Coal														
C ₁ -975°F Distillates	69.4	73.8	65.8	75.0	76.2	68.8	72.5	79.6						
975°F+ Conversion	88.1	91.9	82.0	92.4	90.2	86.6	87.4	93.5						
Coal Conversion	91.9	93.3	89.3	94.5	95.4	93.7	96.0	96.2						
Hydrodesulfurization, W%	78.3	79.5	72.2	81.6	81.3	67.9	82.9	88.3						
Hydrodenitrogenation, W%	87.5	89.9	76.3	90.1	83.8	83.8								

RUN CC-9 (227-66E)

Objectives

The objectives for this run were to produce atmospheric-still-bottoms as feedstock for delayed coking and to study delayed coking as a means for solids separation.

Scope Of Work

The run was designed to study delayed coking as a means of solids separation, obtaining liquid product from coking primarily to slurry feed coal. Two batches of atmospheric-still-bottoms were produced. Run CC-9 was an extension of Run CC-8 using Illinois No. 6 (OTISCA micronized) coal as feed.

The bottoms were then processed in a minicoker by Foster Wheeler Development Corporation.

Coal

Since Run CC-9 was an extension of Run CC-8, coals used in CC-8 were also used in CC-9. Cleaned and uncleaned batches of Illinois No. 6 coal were used. The feed coal supplied by OTISCA Industries was available as slurry containing around 30 W% coal in micronized form. Typical particle size analysis is shown in Table 4. The analyses of the feed coals are given in Table 7.

Catalyst

Run CC-9 was a continuation of Run CC-8, the latter was started with a batch of fresh Shell 317 (NiMo) catalyst.

Makeup Oil

The run plan called for a makeup oil to be used as a part of the recycle oil requirements to compensate for the atmospheric-still-bottoms removed from the system for coking tests. L-756, a composite pressure-filter-liquid obtained from the earlier CTSL runs, was primarily used. As described later, the most of the second condition had to be executed with larger portions of makeup oil. On the last day of the run, HRI-5648, a Wilsonville derived oil was used when L-756 was exhausted. The analyses of the makeup oils are shown in Table 8.

Run Plan

Run CC-9 was a continuation of Run CC-8 using Illinois No. 6 coal (cleaned and uncleaned) supplied by OTISCA Industries as a stable slurry of micronized coal. The slurry was available with moisture contents typically in the range 65-70 W%. The feed coal was fed to the unit after on-line drying using the procedure described for Run CC-8. Typically, the moisture content of the dried coal (as fed to the reactors) was under 2 W%.

The products from the reactors were separated as Separator Overhead (SOH), atmospheric-still-overhead (ASOH), and continuous-atmospheric-still (CAS) bottoms. A portion of the CAS bottoms was saved for delayed coking and the rest was pressure-filtered in a batch mode to obtain pressure-filter-liquid (PFL) and pressure-filter-solids (PFS). A makeup oil was used to balance the recycle oil requirements.

Two batches of CAS bottoms, one from each condition, were prepared and sent to Foster Wheeler Development Corporation for delayed coking tests.

Performance and Results

The liquefaction performance is shown in Table 9. The normalized yields for the second condition could not be calculated accurately as the process was not at steady state due to changes in the feed composition.

Table 10 shows the analyses of the atmospheric-still-bottoms used for delayed coking. The tests were conducted by Foster Wheeler in a 4-inch diameter minicoker with the objective of maximizing liquid yields. One test per feed was performed.

Two key parameters, temperature and steam/feed ratio, were selected as the variables in order to meet the objective of producing high liquid yields. The effects of other potential variables are either known (pressure and throughput ratio) or have a lesser effect on yield (coking cycle time, feed rate, and steamout period); therefore, these variables were held constant.

Initially, a scoping test was done at high coking temperature and a steam rate significantly above normal commercial ratios. The drum temperature was at or near the top of standard commercial operation. The feedstock (Condition 1 product) was not thermally stable at these conditions and the coil was quickly plugged. A test (Run 1) was completed successfully at a lower temperature. For the second run, lower coking temperature and higher steam/feed ratio was chosen. The test conditions and the yields are shown in Table 11. The tests showed that it is possible to operate a delayed coker to process liquefaction bottoms. The nature of the feedstock requires that the operating temperatures be different than practised commercially for the petroleum based feeds.

The coking products are now being analyzed for coker performance in terms of residuum conversion, coke yield, etc. and also for assessing the quality of the liquid product in terms of its reactivity, solvent quality, etc. The results will be reported later.

TABLE 7**FEED COAL ANALYSES FOR RUN 227-66E (CC-9)****RUN 227-66E FEED: OTISCA PROCESSED ILLINOIS NO. 6 COAL**

Feed Designation, HRI No. Type Feed for Periods	5739	5743
	UnCleaned 16-19	Cleaned 20-23
Moisture Content of Slurry, W%	68.89	67.01
<u>Ultimate Analysis (W%, Dry Basis)</u>		
Carbon	68.78	76.05
Hydrogen	4.69	5.21
Nitrogen	1.35	1.44
Sulfur	3.37	2.68
Ash	10.77	3.58

Note: The detailed analyses (ash analysis, proximate analysis, sulfur, etc.) on these samples are quite similar to those used in CC-8, the analyses on the latter samples are shown in Table 4.

TABLE 8
ANALYSES OF MAKE-UP OILS

Identification	<u>L-756</u>	<u>HRI-5648</u>
<u>Elemental Analysis, W%</u>		
Carbon	87.99	90.14
Hydrogen	11.04	9.85
Nitrogen	0.33	0.37
Sulfur	0.01	0.13
<u>ASTM D-1166 Distillation</u>		
		<u>Temp., °F</u>
IBP	--	735
5 V%	--	776
10 V%	519	798
20 V%	564	810
30 V%	599	830
40 V%	627	842
50 V%	656	858
60 V%	690	876
70 V%	723	888
80 V%	762	902
90 V%	850	930
95 V%	854*	952
IBP	--	1,008

* At 93 V%, 5-10 W% above 975°F.

TABLE 9
RUN CC-9 OPERATING SUMMARY

Period Number	18	19	22	23
Stg. 1 Catalyst Age, Lb Dry Coal/Lb Cat.	578.	624.	761.	800.
Moisture in Coal/Water Slurry, W%	70.78	67.01	66.16	70.00
Moisture in Feed Slurry, W%	0.23	0.19	0.94	0.41
<u>Temperatures, °F</u>				
First Stage Reaction	752.	751.	773.	773.
Second Stage Reaction	801.	800.	811.	809.
Unit Back Pressure, PSIG	2799	2797	2800.	2812.
Space Velocities (Per Stage) Lb/Hr/Ft ³	61.6	72.9	70.7	62.0
<u>Recycles, W% of Dry Coal</u>				
Pressure Filter Liquid	163.4	151.5	17.2	0.0
PFL to Buffer - Stage 1	3.2	2.5	2.5	3.0
PFL to Buffer - Stage 2	3.8	2.0	2.4	2.7
CAS Bottoms	0.0	0.0	42.3	0.0
Make-Up Oil	44.7	34.3	151.7	211.0
Oil/Solids Ratio	2.08	1.86	2.01	2.11
Total Material Recovery (Gross)	99.05	99.60	89.04	101.21
<u>Estimated Normalized Yields, W% Dry Fresh Feed</u>				
C ₁ -C ₃ in Gases	4.79	3.29		
C ₄ -c ₇ in Gases	6.66	5.32		
IBP-390°F in Liquids	8.94	8.92		
390-500°F in Liquids	9.84	9.84		
500-650°F in Liquids	11.20	14.88		
650-850°F in Liquids	15.63	12.27		
850-975°F in Liquids	7.62	5.83		
Toluene Soluble 975°F* Oil	9.42	9.81		
Toluene Insoluble 975°F* Oil	0.60	0.79		
Unconverted Coal	10.57	12.27		
Ash (SO ₃ -free)	10.36	10.36		
Water	6.72	8.65		
CO	0.00	0.00		
CO ₂	0.07	0.06		
NH ₃	1.17	1.17		
H ₂ S	2.59	2.55		
Total (100 + H₂ Reacted)	106.18	105.99		
<u>Process Performance</u>				
C ₄ -975°F Distillates, W% of MAF Coal	66.8	63.6		
975°F+ Conversion, W% MAF	77.0	74.5		
Coal Conversion, W% MAF	88.2	86.3	93.6	92.1
HDS, W%	72.4	71.1		
HDN, W%	71.3	71.1		

TABLE 10
ANALYSES OF COKING FEEDSTOCK

Condition	1	2
Sample Identification	LO-5274/75	LO-5276
*API	-1.5	-0.5
IBP, °F	549	500
IBP-650°F, W%	10.00	15.00
650-850°F, W%	41.51	40.09
850-975°F, W%	12.40	10.74
975°F+, W%	36.09	34.17
Carbon, W%	84.18	85.69
Hydrogen, W%	9.22	9.27
Nitrogen, W%	0.49	0.55
Sulfur, W%	0.56	0.52
Ash, W%	4.87	3.11
Quinoline Insolubles, W%	11.35	8.41
Toluene Insolubles, W%	13.10	12.05
Pentane Insolubles, W%	23.77	21.83
CCR (Liquid Portion), W%	8.54	7.64

TABLE 11
DELAYED COKING TESTS

Run No.	1	2
Feed	LO-5274/75	LO-5276
Coke Drum Pressure, psig	Base	Base
Steam/Feed Ratio	Base	2 x Base
Average Drum Temp., °F	Base	Base - 25
<u>Yields, W% Feed</u>		
Gas	3.5	3.2
Distillate Oil	72.0	81.1
Steamout Oil	1.1	0.4
Coke	20.2	19.1
Material Balance Recovery, (%)	96.8	103.8

TASK 4 - MINI-PDU

A cost estimate was prepared to restart the coal handling and drying facility at HRI's Research and Development Center. The cost of refurbishing and restarting and/or installing a new state of the art facility was greater than \$400,000. Thus, plans are to purchase coal prepared by an outside coal processing facility as is practiced at Wilsonville.

TASK 5 - TECHNOLOGY ASSESSMENT

Using the economic basis shown in Table 12 and projecting construction costs from 1984 to 1991, a 14.4 rate of return was obtained from an Eastern U.S. bituminous coal plant processing 8,400 tons/day. See Table 13.

TABLE 12**ECONOMICS OF CTSL PROCESSING OF AN
EASTERN U.S. BITUMINOUS COAL****ECONOMIC BASIS**

Type of Financing, % Equity	100
Construction Period, Years	4
<u>Draw-Down, %</u>	
First Year	5
Second Year	25
Third Year	45
Fourth Year	25
Escalation Rate During Construction	5
Depreciation Period, Years	25
Depreciation Type	Straight Line
<u>Capacity Factor, %</u>	
First Year	65
Second Year	80
Thereafter	100
Inflation Rate, %	4
Income Tax Rate, %	33
Service Factor, %	90
Product Pricing vs Crude Oil	1.3
<u>Crude Oil Price \$1/Bbl DOE Forecast</u>	
1990	20
1995	26
2000	33

TABLE 13**ECONOMICS FOR EASTERN U.S. BITUMINOUS COAL****ILLINOIS #6
BURNING STAR**

Feed Coal, T/D	8,400
Cost, \$/T	25.
Power, KW/HR	66,037
Natural Gas, MM BTU/HR	4,344
Ash Disposal, T/D	832
Raw Water, gpm	3,361
Liquid Products, B/D	43,095
Sulfur, LT/D	179
Ammonia, T/D	143
Investment, \$MM, 1991	1,661
<u>Rate of Return, %</u>	
DOE Basis	14.4

TASK 6 - ADMINISTRATION

Samples from run-of-mine and cleaned coals from oil agglomeration and OTISCA have been forwarded to Penn State for maceral analysis.

Samples from runs CC-2, CC-5, CC-6 and CC-8 have been forwarded to Consolidation Coal Co. for analysis and characterization of the first and second-stage oils.

An updated schedule of the Bench-Scale and the Mini-PDU run is shown as Figure 1.

FIGURE 1

END

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