THE DIRECT LIQUEFACTION PROOF OF CONCEPT PROGRAM

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INTRODUCTION: - Demonstration Operations at the Direct Liquefaction Proof-Of-Concept Facility at the HRI Research and Development Center in Lawrenceville, N.J. are funded by the U.S. Department of Energy, Hydrocarbon Research Inc. and the Kerr-McGee Corporation. The facility is operated and managed by Hydrocarbon Research Inc.

The Proof of Concept Facility consists of several distinct process units, a Coal Handling System, a Slurry Preparation-Pumping-Preheating System, Two Ebullated-Bed Reactors in Series, an On-Line Hydrotreater, Separation and Pressure Let-Down System, Scrubbing and Oil-Water Separation, Flash Vessels and Atmospheric and Vacuum Distillation Equipment, a Rose-SR℠ and U.S. Filter Solid Liquid Separation Systems and Product Storage. The POC Process Development Unit (PDU) processes about 3 tons a day of coal producing about 15 barrels per day of clean distillate liquid product and can operate with or without solids containing recycle solvent. The Rose-SR unit and On-Line Hydrotreater were added along with other improvements to the equipment and control systems prior to the CTSL™ Process Scale-Up with Illinois #6 Bituminous Coal. *Figure 1* shows a schematic of the process.

The Catalytic Two Stage Liquefaction Process (CTSL) is an advanced direct coal liquefaction process that utilizes a low temperature first stage to foster hydrogenation in the presence of a nickel molybdenum ebullated bed catalyst and a higher temperature second stage to increase conversion and heteroatom removal. In POC Run 01, the main objective was to scale-up the CTSL Process with Illinois #6 coal in the extinction recycle mode to produce an all distillate slate of products.

The Proof of Concept Program started in October 1992 with the first year devoted to a facility upgrade, followed by operations on POC Run 01 through February 1994.

PROGRAM AND POC-01 OBJECTIVES - The overall program objective is to develop direct coal liquefaction and associated transitional technologies which are capable of producing premium liquid fuels, which are competitive with petroleum and which can be produced in an environmentally acceptable manner.

POC-1 objectives were to confirm equipment operability, to collect data in the CTSL mode of operation while feeding Illinois #6 Crown II Mine coal, to collect 2500 gallons of distillate for upgrading studies, to evaluate the ROSE-SR℠ and U.S. Filter Systems, and to provide a comparison with the existing data base.

FACILITY UPGRADE - During the first year the POC facility was modified to improve reliability and to provide the flexibility to operate in several alternate modes. Kerr-McGee's Rose-SR℠ unit from Wilsonville, Alabama was redesigned and installed at HRI to allow a comparison with the U.S. Filter solids separation system. Also included was modifying a reactor from Wilsonville, enlarging the reactor structure, installing an on-line hydrotreater, providing interstage sampling, upgrading the computer control and data acquisition system, revising the preheaters and connecting to an alternate power supply grid.

OPERATIONS - Run POC-1 started on October 29, 1993 feeding Illinois #6 coal using 1/16th inch Ni-Mo on alumina catalyst in each of two ebullated reactors and with hydrogenated Cat Cycle oil for start-up. The operating conditions plan as shown in the following Operating Summary consisted of five main conditions with some variations in conditions 3 & 4, (*See Table 1*). As indicated the space velocity and reaction temperatures were increased and the solvent/coal ratio was decreased as the run progressed. In addition

the catalyst addition rate was increased as planned and inadvertently decreased by mal-operation of the catalyst addition system in periods 47-58. Fifty-eight days on coal feed were completed concluding in a planned shutdown in February 1994.

RESULTS & ACCOMPLISHMENTS - This run was a successful scale-up of the CTSL processing of Illinois #6 coal with a Rose-SR solid separation unit. In the first two operating conditions at moderate temperatures and at low space velocity (2 tons/day), high distillate yields and conversion were observed; C4-975F yields of 73-82% of MAF coal and 975F+ Conversions of 86-95%. These results confirmed bench scale data and as seen in *Table 2* were sustained in ensuing periods despite variations in Deasher performance as optimum operating conditions and procedures were being developed. In the latter stages of the operation, resid conversion and distillate yield decreased due to problems with the catalyst addition system and low resid recovery from the Rose-SR Deasher due to a 10% increase in asphaltene yield. The downward trends of key performance indicators as the run progressed are depicted in Figure 2.

Some of the major accomplishments of this initial run are:

- Successfully operated a new 3 ton/day two stage ebullated-bed reactor system incorporating a Rose-SR solid separation unit.
- Demonstrated distillate production at C4-975F levels of 70-75wt% MAF and coal conversions of 94-96% with Illinois #6 coal.
- Achieved a Bottoms Energy Rejection of 12% for a sustained period of Rose-SR operation. Retrograde reactions were not observed.
- Achieved operation with a more concentrated feed slurry; coal concentrations up to 53%.
- Collected 3500 Gallons of 150-650F distillate (0.06% N, 0.03% S) for upgrading and engine testing.
- Evaluated various materials of construction in a high temperature hydrogen environment.

Plans - Run POC-2 is starting in May of this year. It is scheduled to be a 40 day operation on sub-bituminous Black Thunder Mine Wyoming coal investigating scale-up of the CTSL Process with on-line hydrotreating, Rose-SR and Filtration Separation and most importantly whether or not solid deposition occurs in the process equipment and lines. POC-3 is scheduled for October 1994 and will study the co-processing of sub-bituminous coal and a high metals, California Petroleum Resid over a 40 day period. POC-4 is planned for early 1995 with the objective to study the scale-up of the processing of sub-bituminous coal using dispersed and supported catalysts.

Acknowledgement

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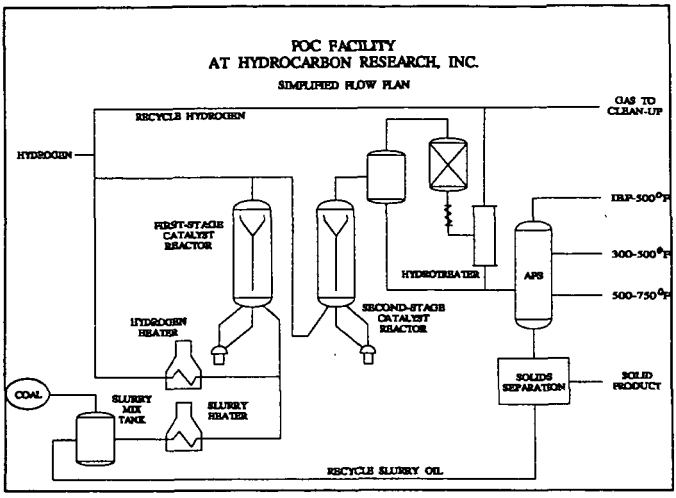

Figure 1

POC-01 Operating Summary Illinois No. 6 Crown II Mine Coal Akzo AO-60 1/16" Extrudate Catalysts							
Condition **Period No**	**1** **13-19**	**2** **20-26**	**3** **27-32**	**3B** **41-44A**	**4 A/B** **47-50**	**4C** **54-57**	**5** **58**
Recycle Mode	Ashy	< Solid-Free >					
Space Velocity [lb mf coal/h/ft³ reactor]	19	20	29	29	27	29	28
Solvent/Coal Ratio	1.2	1.25	1.26	1.39	1.4	1.0	1.1
First Stage							
Temp F	768	765	775	770	775	775	775
Cat. Repl. Rate [lb/ton mf coal]	0.5	1.5	1.5	1.5	0	0	0
Second Stage							
Temp [F]	799	810	815	810	812	820	824
Cat. Repl. Rate [lb/ton mf coal]	1.0	3.0	3.0	3.0	3.0	0	0

TABLE 1

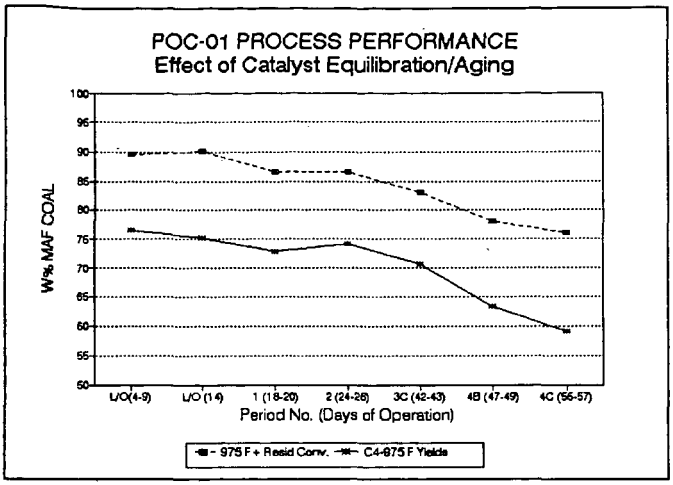

Figure 2

POC-01 PROCESS PERFORMANCE Coal: Illinois No. 6 Crown II Mine (10.4 w% Dry Ash) Catalyst: Akzo AO-60 1/16" NiMo Extrudates in both Reactors						
Process Conditions Period/s	L/O Rose-SR 14	1 18-20	2 24-26	3B 42-43	4B 47-49	4C 55-57
Recycle Type	Ashy	Ashy	< Ash-free >			
Reactor K-1: Temperature, Deg. C	409	408	407	411	414	413
Reactor K-2: Temperature, Deg. C	426	432	432	433	433	436
Flow Rates Coal Feed, Kg/hr	68.6	69.5	70.2	87.9	89.8	102.8
Process Performance						
Chemical H_2 Consumption, W% MAF	7.6	7.1	7.1	6.1	5.9	5.3
Coal Conversion, W% MAF	95.2	95.6	95.0	94.7	95.1	95.4
524 C+ Conversion, W% MAF	90.1	86.6	86.6	83.0	78.0	76.0
Desulfurization (Organic), W%	98.3	98.0	97.7	96.0	94.4	94.0
Denitrogenation, W%	88.2	86.0	82.5	78.2	75.9	78.0
C4-524 C Distillates, W% MAF	75.1	72.8	74.2	70.64	63.2	58.8
Deasher Performance Deasher	Vacuum Still	ROSE-SR				
Energy Rejection, % Deasher Coal Conversion, W% MAF	23.1 94.8	25.2 95.7	16.5 95.1	12.8 95.2	22.5 95.2	33.0 94.9

TABLE 2