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UPGRADING OF COAL LIQUIDS

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Progress Report
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ABSTRACT

First-stage hydrocracking process variable studies on H-Coal gas oil were concluded. At base pressure, data were obtained at one temperature and three space velocities. At 500 psig below base pressure, data were obtained at one temperature and one space velocity.

The liquid products from the preparative first-stage H-Coal gas oil hydrocracking run, completed in June, were blended to provide one drum of charge stock for second-stage hydrocracking to gasoline. All required analyses for this stock have been completed. One second-stage hydrocracking run has been completed.

A second preparative run has been started to produce feedstock for second-stage hydrocracking to fuel oil.

The second shipment of H-Coal atmospheric still bottoms intended for hydrotreating-fluid catalytic cracking is being rerun.

Routine inspections of EDS process naphtha have been completed.

Task 1

Work Accomplished

Process variable studies for first-stage hydrocracking of H-Coal gas oil were concluded with experiments at lower pressures. Inspections of the feedstock (No. 96-3330A) can be found in any of the four earlier ERDA reports (FE-2566-01 to FE-2566-04).

At base pressure hydrocracking studies were made at a temperature about 8°C below base and at three space velocities. The run (Run 857) was concluded

with a study at 500 psig below base pressure, ca. 7°C below base temperature, and a reduced space velocity of 1.56. Data obtained are summarized in Table 1. The table also shows data (Periods 21-26) obtained from rechecking an earlier data point.

Figure 1 is a semi-logarithmic plot of percentage residual nitrogen (100 - % conversion) versus reciprocal reduced space velocity. These data points (Periods 27-42) were obtained at base pressure. As mentioned in previous reports, these raw data are subject to adjustment for deviation in temperature.

The products (stripper bottoms) from the preparative first-stage H-Coal gas oil hydrocracking run (Plant 638H, Run 7) were blended to provide one drum of feedstock for second-stage hydrocracking studies with fractionator at maximum gasoline-yield mode. Inspections of this hydrocrackate are given in Table 2. A total of six separate runs, each with fresh catalyst, is scheduled to be carried out in Plant 601.

The first run (Run 749) was conducted at 500 psig below base pressure, 0.725 X base fresh feed (FF) space velocity, and 1.25 X base combined feed ratio (CFR). Startup temperature was 33°C below base (Figure 2). The temperature was then increased stepwise until the conversion to 375°F⁻ distillate reached 100%. This level of conversion was attained at 108 hours on stream when the temperature reached 16°C above base. The run was then continued for an additional period of 138 hours during which a 3°C increase in temperature was required to maintain the activity for distillate conversion. Analyses of these products are in progress.

Another second-stage hydrocracking run has now been started to obtain data at 1.45 x base FF space velocity and otherwise identical pressure and CFR.

Another first-stage H-Coal gas oil hydrocracking run (Plant 638H, Run 9) has been started at conditions similar to those of Run 7. The object of this run is to produce a 600°F+ stock with a nitrogen content of <10 wt-ppm and a 400°F+ stock with <500 wt-ppm N. The first stock will be used as feed for second-stage hydrocracking to maximum fuel-oil. The second stock will be a feed for a series-flow second-stage hydrocracking to maximum gasoline.

Task 1

Work Forecast

The second first-stage hydrocracking preparative run will be continued into September until a sufficient amount of 600°F+ stock is produced.

Second-stage H-Coal gas oil hydrocracking studies with fractionator at maximum gasoline-yield mode should be completed by the middle of September.

Task 2

Work Accomplished

The second shipment of 90 gallons of H-Coal process liquid arrived at Des Plaines on March 31, 1977. This consisted of two drums of atmospheric still bottoms which were assigned for work under Task 2. Inspections of these two samples are shown in Table 3.

This charge stock is being rerun in a batch vacuum flash unit to reduce the heptane insolubles to <0.1%.

Task 2

Work Forecast

Hydrotreating process variable studies for rerun H-Coal gas oil is scheduled to begin in August, 1977 (Table 5).

Task 3

Work Accomplished

The shipment of EDS process naphtha arrived at Des Plaines on July 18, 1977. The drum was inspected immediately and found to contain about 15 gallons. Distillation data obtained for the sample indicate that it is not necessary to remove any heavy ends. Analyses show the sample contains 9978 wt-ppm sulfur, 2096 wt-ppm nitrogen, and 13,700 wt-ppm oxygen. These are relatively high compared to petroleum naphthas. Table 4 shows inspections for this liquid.

In accordance with current UOP procedures for evaluating Platformer charge stocks, it will be necessary to hydrotreat this naphtha to bring the hetero-cyclics content within acceptable limits.

Task 3

Work Forecast

Hydrotreating of EDS naphtha will be started the beginning of August. Platforming® of the upgraded EDS naphtha will begin in the latter part of August.

Platforming® of the naphtha (H-Coal process) from Task 1 is scheduled to start in the beginning of November, 1977 (Table 5) when sufficient amount of feedstock is available.

TABLES AND FIGURES

Table 1

First-Stage Hydrocracking of H-Coal Atmospheric Still Bottoms

Plant 505, Run 857

Period No.	Hours on Stream	p-p (base), psig	LHSV / LHSV (base)	T-T (base), °C	Prod. N Content, ppm	N Conv., Wt-%
Feed					3900	
21	250-260	500	1.56	-16	597.0	84.69
22	260-270	500	1.56	-19	699.9	82.05
23	270-280	530	1.56	-23	882.5	77.37
24	280-290	530	1.56	-17	1037.1	73.41
25	290-300	500	1.56	-22	1279.9	67.18
26	300-310	500	1.56	-23	816.0	79.08
27	320-330	0	0.63	-10	15.2	99.61
28	330-340	0	0.67	-8	9.7	99.75
29	340-350	0	0.61	-8	10.1	99.74
30	350-360	0	0.66	-8	11.1	99.72
31	370-380	0	1.56	-8	880.0	77.44
32	380-390	0	1.56	-8	927.9	76.21
33	390-400	0	1.56	-8	599.4	84.63
34	400-410	0	1.53	-7	425.6	89.09
35	420-430	0	3.86	-8	1640.4	57.94
36	430-440	0	3.92	-9	2807.2	28.02
37	440-450	0	3.92	-4	2389.6	38.73
38	450-460	0	3.92	-8	2563.8	34.26
39	474-484	0	0.64	-8	18.8	99.52
40	484-494	0	0.64	-8	12.1	99.69
41	494-504	0	0.64	-8	7.6	99.81
42	504-514	0	0.63	-8	26.6	99.32
43	524-534	-465	1.56	-6	883.4	77.35
44	534-544	-475	1.56	-6	836.7	78.55
45	544-554	-460	1.56	-6	1015.3	73.97
46	554-564	-470	1.56	-7	927.6	76.22
47	564-574	-555	1.56	-6	977.6	74.93
48	574-587	-480	1.56	-7		

Table 2

Inspections of First-Stage H-Coal Hydrocrackate

Sample No.	3531-5
API @ 60°F	19.2
Sp. Gr. @ 60°F	0.9390
Distillation (ASTM D-1160)	
IBP, °F	315
5%	420
10%	458
20%	479
30%	498
40%	514
50%	530
60%	550
70%	573
80%	605
90%	660
95%	711
EP	748
% Over	99.0
% Bottoms	1.0
Hydrogen, Wt-%	10.69
Carbon, Wt-%	88.41
Sulfur, Wt-ppm	1.4
Nitrogen, Wt-ppm	4.8
Oxygen, Wt-ppm	530.4
Molecular Weight	216
Pour Point, °F	-25
Viscosity, cst, 100°F	3.434
FIA, Vol-%	
A	69.7
O	0.0
P&N	30.3

Table 3

Inspections of H-Coal Atmospheric Still Bottoms

	As Received		After Flash Distillation
HRI Sample No.	LO-585	LO-586	
UOP No.	37-1118	37-1117	
°API @ 60°F	7.4	12.1	
Sp. Gr. @ 60°F	1.0187	0.9854	
Distillation, ASTM D-1160			
IBP, °F	412	410	
5%	463	467	
10%	485	474	
20%	509	495	
30%	530	519	
40%	558	545	
50%	580	566	
60%	603	591	
70%	631	621	
80%	661	666	
90%	715	710	
95%	768	772	
EP	857	855	
% Over	99.0	99.0	
% Bottoms	1.0	1.0	
Hydrogen, Wt-%			
Carbon, Wt-%			
Sulfur, Wt-%	951	690	
Nitrogen, Wt-%	3534	2966	
Oxygen, Wt-%	5006	5480	
Con. Carbon, Wt-%	0.41	0.49	
Heptane Insoluble, Wt-%	0.37	0.53	
FIA, Vol-%			
A	90.9	83.7	
O	0	0	
P & N	9.1	16.3	
Stm. Jet Gum, mg/100 ml	620	933	

Table 4

Inspections of EDS Process Naphtha

Sample No.	3531-7
°API @ 60°F	38.4
Sp. Gr. @ 60°F	0.8328
Distillation, ASTM D-86	
IBP, °F	142
5%	178
10%	208
15%	228
20%	244
30%	268
40%	286
50%	302
60%	316
70%	319
80%	339
90%	348
95%	356
EP	380
% Over	98.5
% Bottoms	1.5
Sulfur, Wt-ppm	9978
Nitrogen, Wt-ppm	2096
Oxygen, Wt-ppm	13700
Chloride, Wt-ppm	18
FIA, Vol-%	
A	
O	
P & N	
RON, Clear	
Bromine No.	39.0
N_2 Jet Gum, mg/100 ml	44

r. rerunning
p.v. process variable study
prep. preparative run
U Unifining
P Platforming

Table 5

Work Plan and Progress

Legend

— --- H-Coal Liquids
═ --- EDS Liquids

Scheduled
Completed

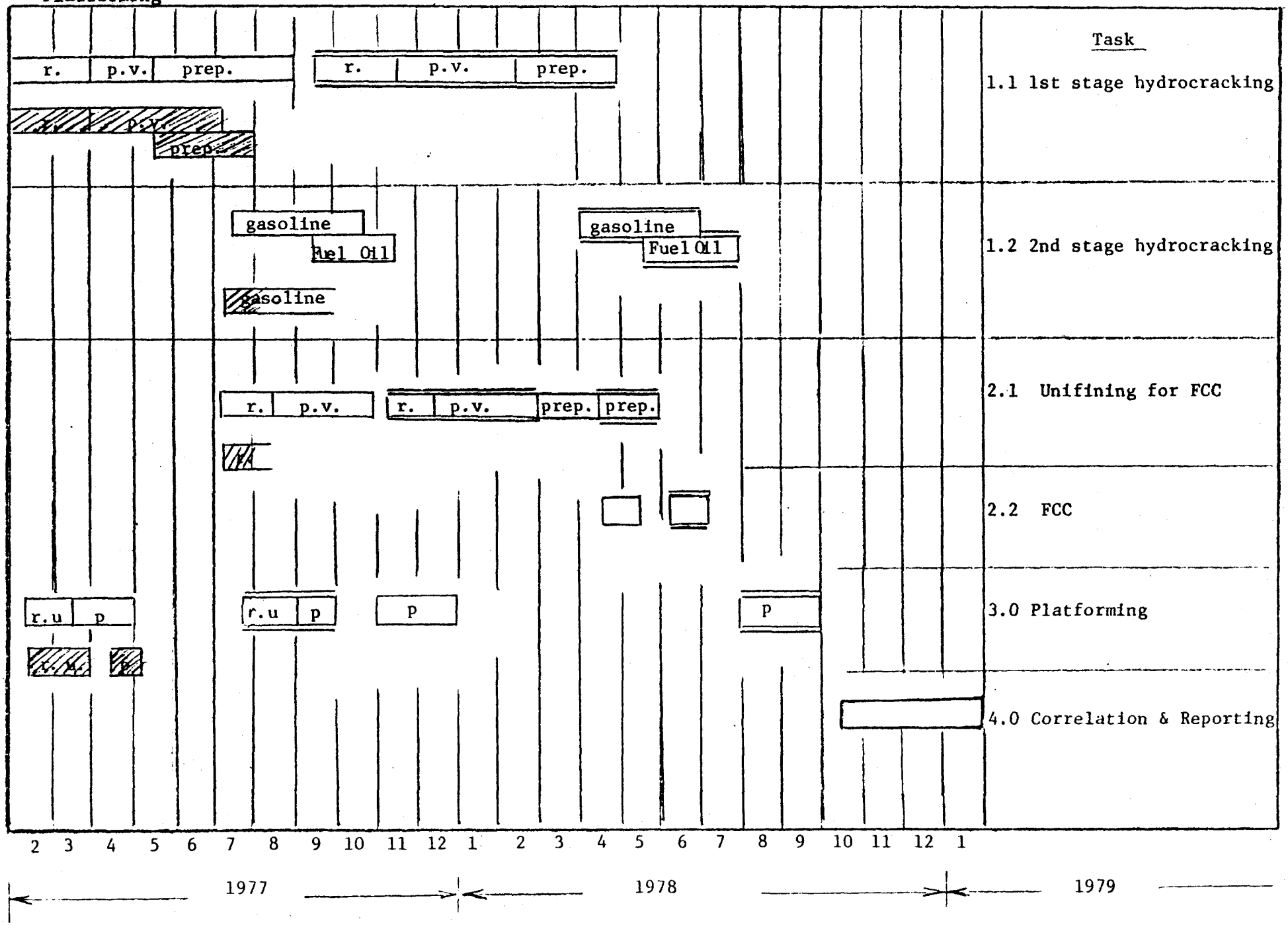

Figure 1

Percentage Residual Nitrogen vs. Reciprocal Reduced Space Velocity

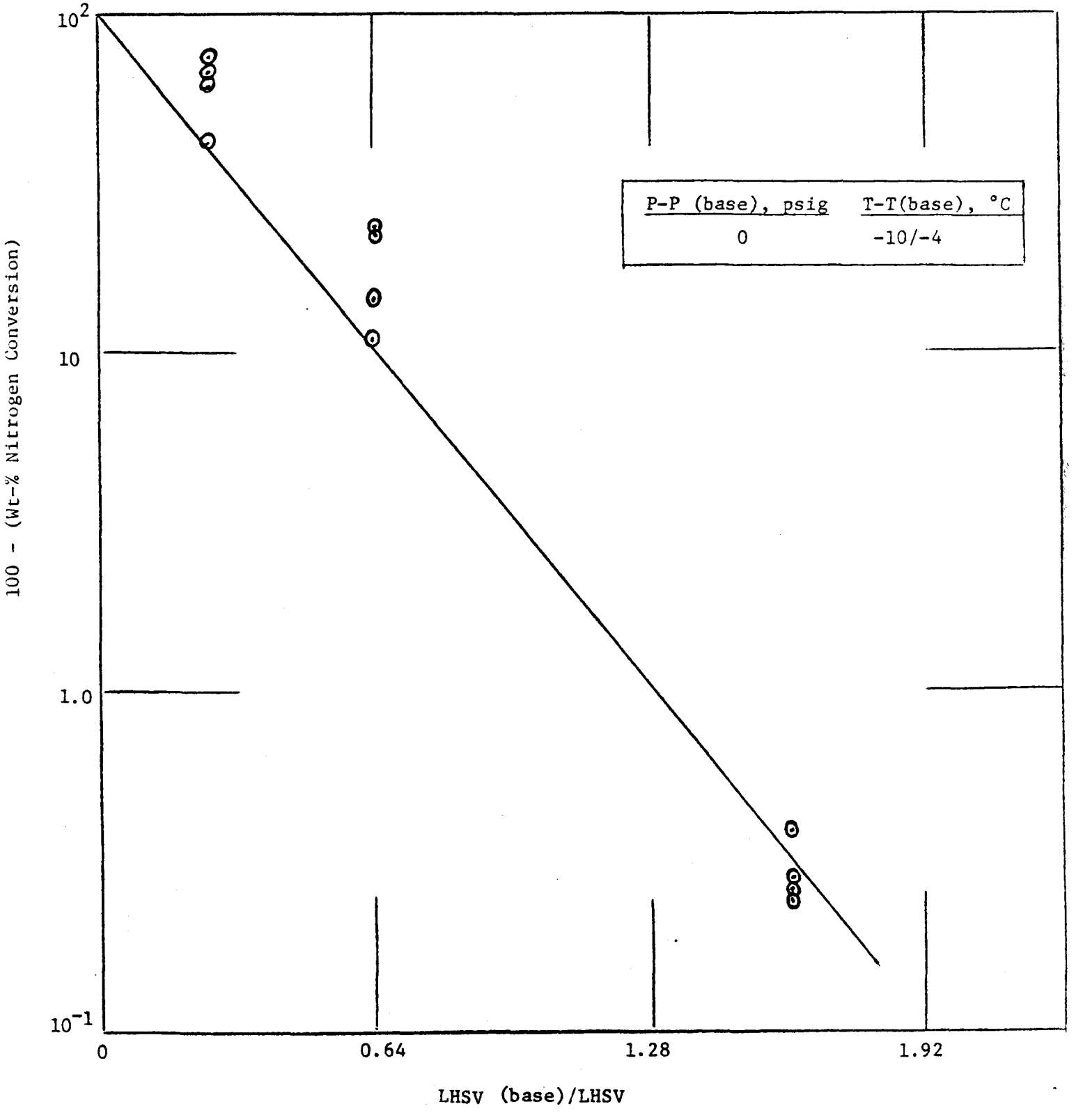

Figure 2

Temperature vs. Time

Second-Stage Hydrocracking of H-Coal Gas Oil with Fractionator at Maximum Gasoline Yield Mode

(Plant 601, Run 749)