FE 2315-14

REFINING AND UPGRADING OF SYNFUELS FROM COAL AND OIL SHALES BY ADVANCED CATALYTIC PROCESSES

Monthly Report for the
Period May 1977

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ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. EF-76-C-01-2315

I. Objective

The objective of this program is to determine the feasibility and to estimate the economics of hydroprocessing four synthetic crude feedstocks to distillate fuels using presented available technology. The first feedstock is shale oil. The shale oil used in this evaluation is Paraho crude shale oil, produced in the indirectly heated mode. Pilot plant studies evaluating hydroprocessing of the whole shale oil have been in progress for about seven months. The second feedstock is solvent refined coal (SRC), obtained from the Pittsburg and Midway Coal Company pilot plant near Tacoma, Washington. Studies with the SRC started in April of this year.

Encl. - Tables I-X (RE 772863, RE 772864,
RD 772865, RE 772952-RE 772958)
Figures 1-5 (RD 772348-1, RE 772854,
RE 772959-RE 772961)

II. Shale Oil Processing

Task 2--Whole Shale Oil Hydrofining

Run 86-51 is a large-scale, whole shale oil hydrofining pilot plant run made with 650 ml of whole ICR 106 catalyst. This unit contains six reactors in series. The first reactor is charged with a low cost material to serve as a guard bed to provide a surface for deposition of iron, arsenic, and other materials that might otherwise act as catalyst poisons. The remaining five reactors each contain 130 ml of catalyst.

Conditions are as follows: (a) liquid hourly space velocity (LHSV), based on ICR 106 catalyst, is 0.6; (b) target product nitrogen is 500 ppm in the whole product, with manual temperature adjustment to control product nitrogen; (c) recycle gas rate is 8000 SCF/B; (d) during the first 210 hours on-stream, total pressure was 2200 psig; at 210 hours on-stream, the total pressure was increased to 2350 psig to adjust the hydrogen pressure to the target of 1750-1800 psig.

Figure 1 shows the first 1500 hours of the run. Hydrogen pressure decreased as the temperature was increased during the course of the run. Then, as was noted last month, between 965 and 1200 hours on-stream, a leak in the recycle gas served as a bleed, causing the hydrogen purity to increase. As a result of the higher hydrogen pressure, catalyst activity increased. At 2100 psia hydrogen, the catalyst is 25°F more active than at 1625 psia.

After correction of the recycle gas leak, the hydrogen partial pressure decreased again to 1625 psia, and the activity also decreased by about 25°F.

Without the recycle gas bleed, the catalyst began to foul rapidly. The catalyst temperature, normalized to 500 ppm product nitrogen, increased by about 20°F in the period between 1300 and 1440 hours on-stream. The hydrogen partial pressure further decreased to 1550 psia.

Based on the temperature profile, the first catalyst bed appears to be aged more severely than the other catalyst beds. Figure 2 shows a temperature profile taken at 1365 hours on-stream. This can be compared to the temperature profile at 500 hours (shown in the March 1977 Progress Report). At 500 hours, the first catalyst bed operated at a maximum temperature 35°F higher than the fifth bed. At 1365 hours, the difference is 22°F.

A recycle gas bleed (first, 100 SCF/B; then, 500 SCF/B) was again introduced. After 40 hours, the hydrogen pressure had increased to 1800 psia and about 15°F of activity had been regained.

At 1500-hours on-stream, our supply of raw shale oil feed was exhausted. The additional shale oil expected from the Laramie ERDA laboratory had not arrived. Therefore, in order to continue the run, the feed was changed to a blend of "off-test" partially hydrofined shale oil product, WOW 3495, containing 2000 ppm of nitrogen. This nitrogen level is higher than we believe to be optimum for downstream processing. Therefore, our plan is to hydrofine this material to a whole product nitrogen level of 500-1000 ppm. It can then be used as a supplemental feed if needed for downstream tests. Space velocity was increased to 1.0 LHSV to maintain the catalyst temperature in the range used for the whole shale oil processing. (A catalyst temperature of about 770°F is required for 500 ppm product nitrogen with this partially hydrofined feed and the aged catalyst.)

Shortly before the feed change was made, it was noted that some pressure drop had developed across the guard bed. It was, however, possible to continue running with this pressure drop until 1836 hours on-stream. At that time, the water pump for recycle gas scrubbing failed. As a result, a plug (presumably ammonium sulfide) developed in the recycle gas system. This, in turn, caused a bursting disk (safety release) to rupture and the unit suddenly lost pressure. Subsequently, the run was restarted. However, by this time a 700-psia pressure drop had developed across the guard bed. Presumably, this pressure drop is a result of iron arsenide that had been deposited throughout the run in the guard bed plus "coke" formed when the unit was suddenly depressured. At 1925 hours, the feed was stopped, the unit cooled down, and the guard bed removed. The contents of the guard bed are being examined to determine the nature of the plug. The run is being restarted with a fresh guard bed and the aged catalyst.

Task 3--Downstream <u>Processing Studies</u>

A. <u>Feed Preparation</u>

Product from Run 86-51 was blended and distilled in a batch still to obtain feeds for downstream processing tests. Table I gives a summary of the properties of the two blends distilled. Table II summarizes the yields obtained in the distillations. The 650°F+ fraction from SGQ 6127 was used in catalytic cracking pilot plant studies; the 650°F+ fraction from WOW 3497 is to be used in single-stage hydrocracking tests.

Inspections of the 650°F+ fractions from these distillations are shown in Table III. Also shown in Table III are properties of the two feeds used in the small-scale catalytic cracking tests described last month. Inspections of the lower boiling fractions will be presented next month.

B. Catalytic Cracking of 650°F+ Hydrofined Shale Oil

Last month the catalytic cracking of two hydrofined Paraho shale oils in a small cyclic fixed-fluidized bed cracking unit was discussed. Single tests were made on two hydrofined shale oils whose nitrogen contents were 385 and 870 ppm, respectively.

This month a third hydrofined shale oil with a 1300 ppm nitrogen content has been catalytically cracked in a continuous catalyst circulation pilot plant. Inspections of the feed (SGQ 6139; CCL-5420) are shown in Table III. Three runs have been completed and inspection of the liquid products is progressing. The tabulation below shows the cracking test conditions and gives preliminary estimates of conversions and coke yields.

Run PP158-	511	512	513
Reactor Temperature, °F	930	930	975
WHSV, g/Feed/Hour/g Catalyst	9.07	9.00	7.61
C/O, g Catalyst/g Feed	6.52	4.31	7.05
Catalyst	Equilibrium CBZ-1 (CCL-4904)		
Conversion LV % (430°F-)	69.1	60.5	77.3
Coke, Wt %	3.86	3.12	5.27

The catalyst used in this study was essentially the same equilibrium catalyst (CBZ-1) employed in the fixed-fluidized bed tests of the previous month. The first two runs (511 and 512) were made at a reactor temperature of 930°F. Catalyst/oil ratio, i.e., catalyst circulation rate was varied to give different conversions. The third run (513) was made at 975°F reactor temperature; the temperature of the tests in the fixed-fluidized bed pilot unit made the previous month.

Last month comparisons were made between the yields obtained in the cracking of the hydrofined Paraho shale oils with the yields obtained in the cracking of two hydrofined Arabian Light vacuum gas oils of similar nitrogen content. In making the comparison, it was noted that the two sets of data were obtained as different pilot units but that yield differences were expected to be minor because of this. The present three runs were made in the same pilot unit as was used in the cracking study of the hydrofined Arabian Light gas oils. Consequently, yield and product quality comparisons with the Arabian Light gas oils should be more satisfactory.

Program

The shipment of additional shale oil arrived from Laramie during the week of May 20. This shale oil has been blended and is being dewatered. It will be hydrofined and will provide additional feed for our downstream processing studies. Our first test for recycle hydrocracking of the 650°F+ fraction of the hydrofined shale oil is expected to start about June 10.

III. SRC Processing

Task 1-A--Preliminary Feedstock Analysis

Table IV shows an updated comparison of analyses of the SCR samples obtained from the Pittsburg and Midway Coal Mining Company. Table V shows that the metal content of SRC by emission spectrochemical analysis varies from batch to batch.

Task 1-C--HDF Feed Preparation

We prepared a homogeneous feed blend of 50 wt % SRC (WOW 3406) and 50 wt % 400-700°F creosote oil (WOW 3366) by heating under a N_2 blanket to 370°F with stirring. Table VI shows the inspections of the blend (WOW 3476) and its components.

It was reported last month that the blend appeared to be homogeneous at 200°F without stirring. Table VII shows inspections of samples taken near the top and bottom of the feed pot after standing at 200°F with no stirring. The analyses of the two samples agree within experimental error and support the conclusion that the sample is homogeneous.

Task 2-A--Hydrofining Tests

A. Preliminary HDF Test

The preliminary HDF pilot plant test with the 50/50 wt % SCR/creosote oil (WOW 3476) was started the week of May 2 using ICR 106 catalyst at 0.2 LHSV, 2,500 psig total pressure and 10,000 SCF/B recycle H_2. The pilot plant has six reactors in a series to obtain good feed dispersion. Reactor temperatures are controlled by a furnace containing a fluidized sand bath.

The first reactor has 390 cc of a low cost material to serve as a guard bed and operates upflow. Each of the other reactors has 130 cc of ICR 106 catalyst and operate downflow. The space rate is based on 650 cc of ICR 106 catalyst.

Figure 3 shows the temperature profiles of all catalyst beds after 180 hours on-stream at an average catalyst temperature of 746°F. The temperature of the first catalyst bed is highest, that of the second bed is somewhat lower, and that of the last three beds lower still. The last three beds are essentially equal in temperature.

Tables VIII and IX show the inspections of the feed and the products from the start of run to 330 hours. Figure 4 shows average catalyst temperature and whole liquid product nitrogen plotted versus time. The feed was brought in at an average catalyst temperature of 720°F. Then the catalyst temperature was raised to 747°F. At 234 hours the catalyst temperature was lowered to 725°F to compare with the initial activity. Then the catalyst temperature was raised again to 748°F. There is almost no fouling in denitrification. Figure 5 shows the ASTM D 1160 (2 mm) distillations of the feed and the product of Run 87-67. Table X shows the results from a spinning band distillation of the product at 306 hours.

At 330 hours, a pressure drop of 150 lb developed in the 6 in series reactors. We flushed the reactors with an aromatic solvent at 500°F and hexane at 200°F to remove the product on the catalyst. The sand furnace was cooled down to room temperature.

The outlet pressure of each reactor was measured at a constant inlet pressure to the first reactor. The restriction was in the third catalyst bed (No. 4 reactor).

We removed the catalyst in No. 4 reactor layer by layer and found that the restriction was in a 40 cc section in the middle of the catalyst bed. From preliminary analyses, the catalyst fines from this section contain about 13% carbon. If we compare this result to typical results obtained in petroleum processing, we conclude that it is unlikely that this amount of carbon represents enough "coke" to cause the plug.

Probably the restriction is caused by the deposition of involatile inorganic materials. However, this needs to be verified by additional analyses.

Program

We will attempt to restart pilot plant Run 87-67 after replacing the plugged portion of the catalyst bed with fresh catalyst. If this is unsuccessful, we will start a new run. Several minor pilot plant modifications made as a result of this first test should improve pilot plant operation in future runs. We plan to use the 50/50 SRC/creosote oil blend as feed until we have prepared sufficient product in an appropriate boiling range to use as a solvent for dissolving SRC for further processing studies.

:jlf

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TABLE I

INSPECTIONS OF BLENDS OF HYDROFINED SHALE OIL

Blend Identification	SGQ 6127	WOW 3497
Inspections of Blend		
Gravity, °API	35.8	36.5
Aniline Point, °F	173.7	170.3
Sulfur, ppm	25	11
Nitrogen, ppm	850	616/575
Oxygen, ppm		50
Molecular Weight (Avg.)		270
Pour Point, °F		+75
Ramsbottom Carbon, %	0.09	0.11
Hot Heptane Asphaltenes, ppm		37
TBP Distillation, °F (Simulated by Chromatography)		
St/5	86/273	80/252
10/30	340/485	316/460
50	596	573
70/90	712/857	681/833
95/99	916/	890/
ASTM D 86/D 1160 Distillation, °F (at 2 mm)		
St/5		213/313
10/30		354/522
50		615
70/90		710/822
95/EP		949/970
% Overhead		97
Carbon, Wt %		84.73
Hydrogen, Wt %		13.85
Viscosity, cSt		
at 122°F		3.229
at 210°F		1.795
Ash, ASTM D 486, Wt %		<0.003
Bromine Number		2.2
Neutralization Number, mg KOH/g		
Acid Number		<0.1
Base Number		1.0
pH		7.9

TABLE II

LARGE-SCALE DISTILLATIONS OF HYDROFINED SHALE OIL TO PRODUCE FEEDS FOR DOWNSTREAM PROCESSING

Blend Identification	SGQ 6127			WOW 3497		
Nitrogen in Blend, ppm	850			617/575		
Distribution, % of Whole Liquid Product	Fraction Identification	Wt %	LV %	Fraction Identification	Wt %	LV %
St-180°F	[SGQ 6137]	[13.15]	[14.56]	SGQ 6147	0.40	0.51
180-300°F				SGQ 6148	6.89	7.84
300-400°F				SGQ 6149	10.44	11.05
400-550°F	[SGQ 6138]	[46.74]	[47.05]	SGQ 6150	25.16	25.04
550-650°F				SGQ 6151	19.37	19.07
650°F+	SGQ 6139	40.11	38.40	SGQ 6152	37.74	36.12

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TABLE III

650°F+ HYDROFINED SHALE OIL FEEDS FOR
CATALYTIC CRACKING AND HYDROCRACKING TESTS

Downstream Processing Test Type	Catalytic Cracking			Hydrocracking
Identification No.	ASL 1864 Btms (CCL-5406	SGQ 6115 (CCL-5407)	SGQ 6139 (CCL-5420)	SGQ 6152
Inspections				
Gravity, °API	30.9	30.8	28.4	29.6
Aniline Point, °F	212.3	210.4	209.1	208.4
Sulfur, ppm	<10	<10	10	<10
Nitrogen, ppm	385	870*	1300	885
Oxygen, ppm	150	200	60	150
Molecular Weight (Avg.)	365	383	384	
Pour Point, °F	+105	+100	+105	+105
Ramsbottom Carbon, %	0.12	0.19	0.17	0.15
Hot Heptane Asphaltenes, ppm	477	756	128	(647)
TBP Distillation (Simulated by Chromatography), °F				
St/5	631/661	535/666		
10/30	674/721	682/734		
50	774	787		
70/90	830/917	841/933		
ASTM D 1160 Distillation, °F (2 mm)				
St/5			691/717	550/716
10/30			727/760	729/756
50			811	802
70/90			860/954	842/916
95/EP			1015/1059	944/1073
% Overhead			97	99
Carbon, %		86.40	86.14	
Hydrogen, %	13.22	13.67	12.80	
Viscosity, cSt				
at 122°F	16.37		22.03	
at 210°F	4.787		5.703	
at 300°F	2.253		2.594	

*870 ppm nitrogen is weighted average of components of blend.

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TABLE IV

ANALYSES ON SRC FROM THE PITTSBURG AND MIDWAY COAL MINING COMPANY

Sample	SRC Flakes[1]	SRC Flakes[1]	SRC Flakes[2]	SRC Flakes[2]	SRC Fines[3]	SRC Fines[3]
Data Source	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway
Sample No.	WOW 3406		WOW 3453		WOW 3450	
Density, g/cc, 75°F	1.214		1.235		1.246	
Ash, Wt %	0.22	0.15	0.17	0.14	0.20	0.20
H, Wt %	6.12	5.86	5.92	5.88	6.01	5.85
C, Wt %	87.78	86.34	87.59	86.77	87.03	86.83
O, Wt %	4.52	4.94	5.15	-	4.62	4.22
S, Wt %	0.89	0.71	0.69	0.74	0.67	0.66
Cl, Wt %	0.005		0.005		0.005	
Total N, Wt %	2.04	2.00	2.21	2.11	2.13	2.24
Basic N, Wt %	0.86		0.78		0.91	
Hot Heptane Insolubles, %	96.0		97.2		94.0	
Distillation	TGA[4]		TGA[4]		TGA[4]	
Start	159		163		155	
5%	943		957		935	
10%	1017		1019		1010	
30%	1161		1160		1156	
50%	1232		1241		1231	
70%						
90%						
95%						
End Point	1281		1283		1281	
Rec., %	55.2		55.4		54.8	

[1]Shipped to Chevron Research on December 14, 1976.
[2]Shipped to Chevron Research on March 21, 1977.
[3]Shipped to Chevron Research on March 15, 1977
[4]Distillation simulated by thermogravimetric analysis.

TABLE V

METALS IN SOLVENT REFINED COAL

Sample Sample No.	SRC Flakes[1] WOW 3406	SRC Flakes[2] WOW 3453	SRC Fines[3] WOW 3450
Al	41	72	121
B	17	32	-
Ca	60	74	80
Cr	1	2	8
Fe	98	182	-
Mg	2	6	20
Mn	5	-	-
Si	4	24	-
Ti	147	237	323
V	3	-	-
Zn	6	-	-
Na		5	18
K		1	4

[1]Shipped to Chevron Research on December 14, 1976.

[2]Shipped to Chevron Research on March 21, 1977.

[3]Shipped to Chevron Research on March 15, 1977.

TABLE VI

THE INSPECTIONS OF HDF FEED
AND ITS COMPONENTS

Sample	SRC Flakes	Creosote Oil[1]	SRC/Creosote Oil 50/50	
Sample No.	WOW 3406	WOW 3366	WOW 3476	
Specific Gravity	1.21	1.11	1.14	
Ash, Wt %	0.22	<0.003	0.11	
H, Wt %	6.12	6.14	5.73, 5.67	
C, Wt %	87.78	90.62	90.90, 88.97	
O, Wt %	4.52	1.11	2.70	
S, Wt %	0.89	0.64	0.95, 0.84	
Total N, Wt %	2.04	0.78	1.46	
Basic N, Wt %	0.86	0.45	0.58, 0.33	
Pour Point, °F			115	
Hot Heptane Insolubles, %	96.0	0.0023	52.2	
Benzene Insolubles, %		<0.03	30.2	
Ramsbottom Carbon, %		0.60	29.0	
Viscosity at 210°F, cSt			304.1	
Distillation	TGA[2]	TBP[3]	TGA[2]	ASTM D 1160
Start	159	343	153	483
5	943	406	573	516
10	1017	446	648	572
30	1161	534	802	659
50	1232	599	977	725
70		631	1231	
90		680		
95		687		
End Point	1281	726	1284	1002
Rec., %	55.2	99.0	72.2	61

[1]70% overhead from Allied Chemicals creosote oil (Identification 24-CA), water washed and filtered through a 15-μ filter.

[2]Distillation simulated by thermogravimetric analysis.

[3]TBP distillation simulated by chromatography.

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TABLE VII

HOMOGENEITY OF 50 WT % SRC AND
50 WT % CREOSOTE OIL BLEND AT 200°F
WITHOUT STIRRING

Sample	Top of Pot	Bottom of Pot
S, Wt %	0.89	0.88
Total N, Wt %	1.43	1.45
Oxygen, Wt %	2.23	2.30
Viscosity at 210°F, cSt	189.1	188.8
Ash, Wt %	0.05	0.06

TABLE VIII

HYDROPROCESSING SRC AT 0.2 LHSV,
2500 PSIG AND 10,000 SCF/B RECYCLE H_2

	Run 87-67						
	Feed SRC/Creosote 50/50	← Product →					
Sample No.	WOW 3476						
Run Hour		79	103	127	151	175	234
Average Catalyst Temp., °F		719	721	749	746	746	725
Average LHSV		0.18	0.18	0.22	0.20	0.21	0.22
Specific Gravity	1.14	1.04	1.06	1.05	1.04	1.05	1.04
Gravity, °API		4.3	2.2	3.3	4.0	3.5	
Wt %							
H	5.67, 5.73	9.80	9.08	9.54	8.85	9.04	9.49
C	88.97, 90.90	88.78	88.40	90.91	86.75	89.31	89.13
O	2.70	0.64	0.89	0.70	-	0.85	
S	0.84, 0.95	0.04	0.06	0.04	0.08	0.05	0.05
Total N	1.46	0.48	0.59	0.46	0.46	0.49	0.59
Basic N	0.33, 0.58	0.24					
Viscosity, cSt							
at 100°F	No Movement	86.4	134.2	65.5	56.4	60.8	105.7
at 210°F	304.1	7.35	8.41	6.50	-	6.14	7.80
Hot C_7 Insolubles, %	52.2	11.5	13.3	10.8	9.39	11.1	12.6
Ramsbottom Carbon, %	29.0	11.6	12.4	9.66	11.0	11.3	12.7
Benzene Insolubles, %	30.2	3.12	2.58	2.50	2.87	2.53	3.45

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TABLE IX

HYDROPROCESSING SRC AT 0.2 LHSV, 2500 PSIG AND 10,000 SCF/B RECYCLE H_2

	Run 87-67			
	Feed SRC/Creosote 50/50	← Product →		
Sample No.	WOW 3476			
Run Hour		282	306	330
Average Catalyst Temp., °F		748	747	747
Average LHSV		0.19	0.20	0.22
Specific Gravity	1.14			
<u>Composition, Wt %</u>				
H	5.67, 5.73	7.70	8.29	7.49
C	88.97, 90.90	83.53	87.80	82.43
O	2.70	0.68	0.57	0.72
S	0.84, 0.95	0.04	0.04	0.03
Total N	1.46	0.45	0.43	0.56
Basic N	0.33, 0.58			
<u>Viscosity, cSt</u>				
at 100°F	No Movement	46.9	41.6	
at 210°F	304.1	5.10	5.16	
Hot C_7 Insolubles, %	52.2	9.41	8.01	11.1
Ramsbottom Carbon, %	29.0	10.4	10.5	12.5
Benzene Insolubles, %	30.2			

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TABLE X

SPINNING BAND DISTILLATION OF PRODUCT FROM HYDROPROCESSING 50/50 SRC/CREOSOTE OIL BLEND WITH ICR 106 CATALYST

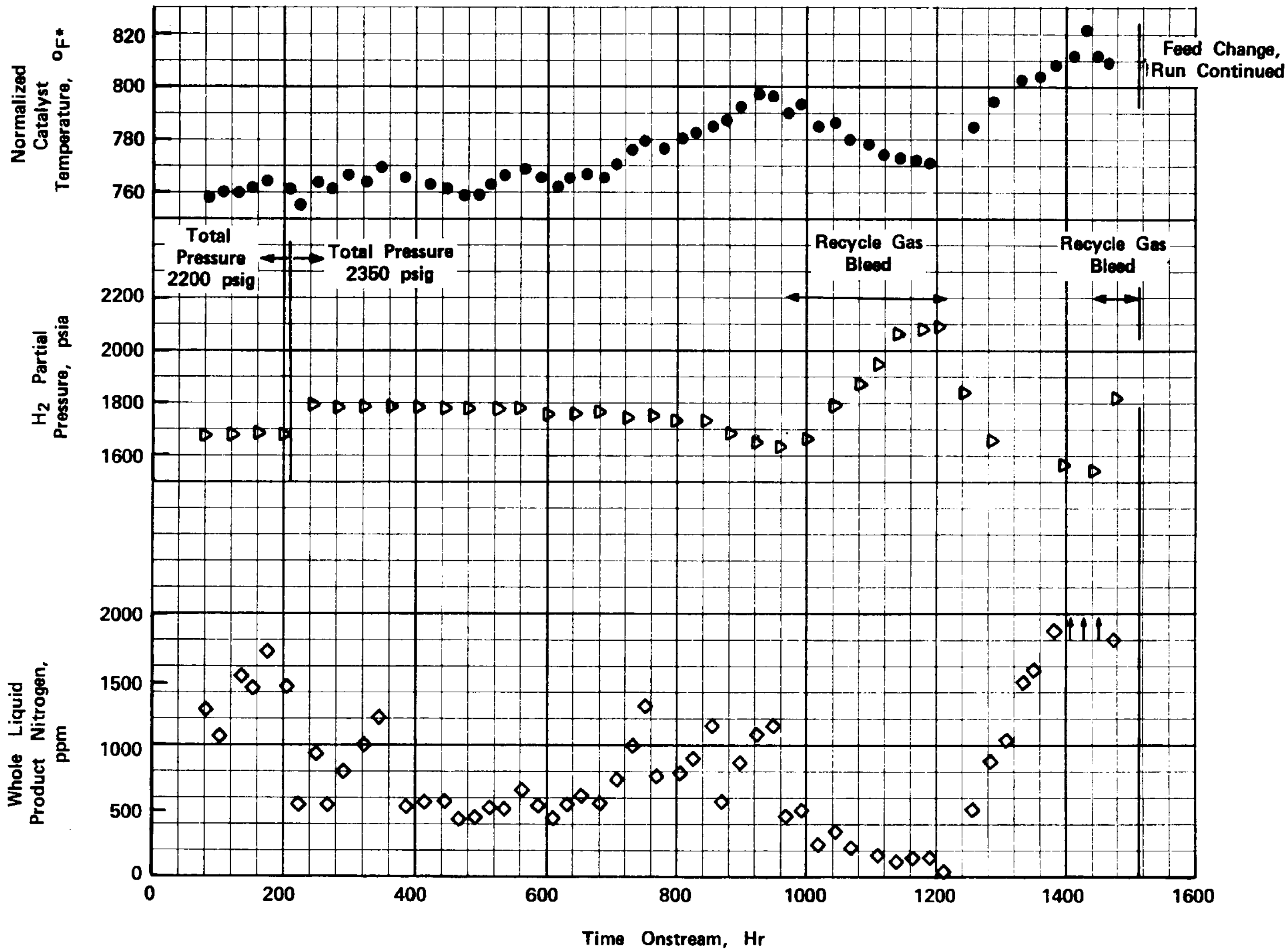
	Run 87-67 at 306 Hours			
	Wt %[1]	Vol %[1]	°API	Density, g/cc
Boiling Range, °F				
St-300	1.8	2.3	47.6	0.790
300-550	25.7	28.0	16.4	0.957
550-650	27.8	28.2	6.4	1.026
650-850	16.9	16.5	1.2	1.066
850+	27.8	24.9	-9.8	1.163

[1]Based on whole liquid product.

FIGURE 1

ERDA CONTRACT EF-76-C-01-2315 – PILOT PLANT RUN 86-51
WHOLE SHALE OIL HYDROFINING WITH ICR 106L CATALYST

0.6 LHSV – 8000 SCF/BBL RECYCLE GAS RATE



*Catalyst Temperatures Normalized to 500 ppm Product Nitrogen

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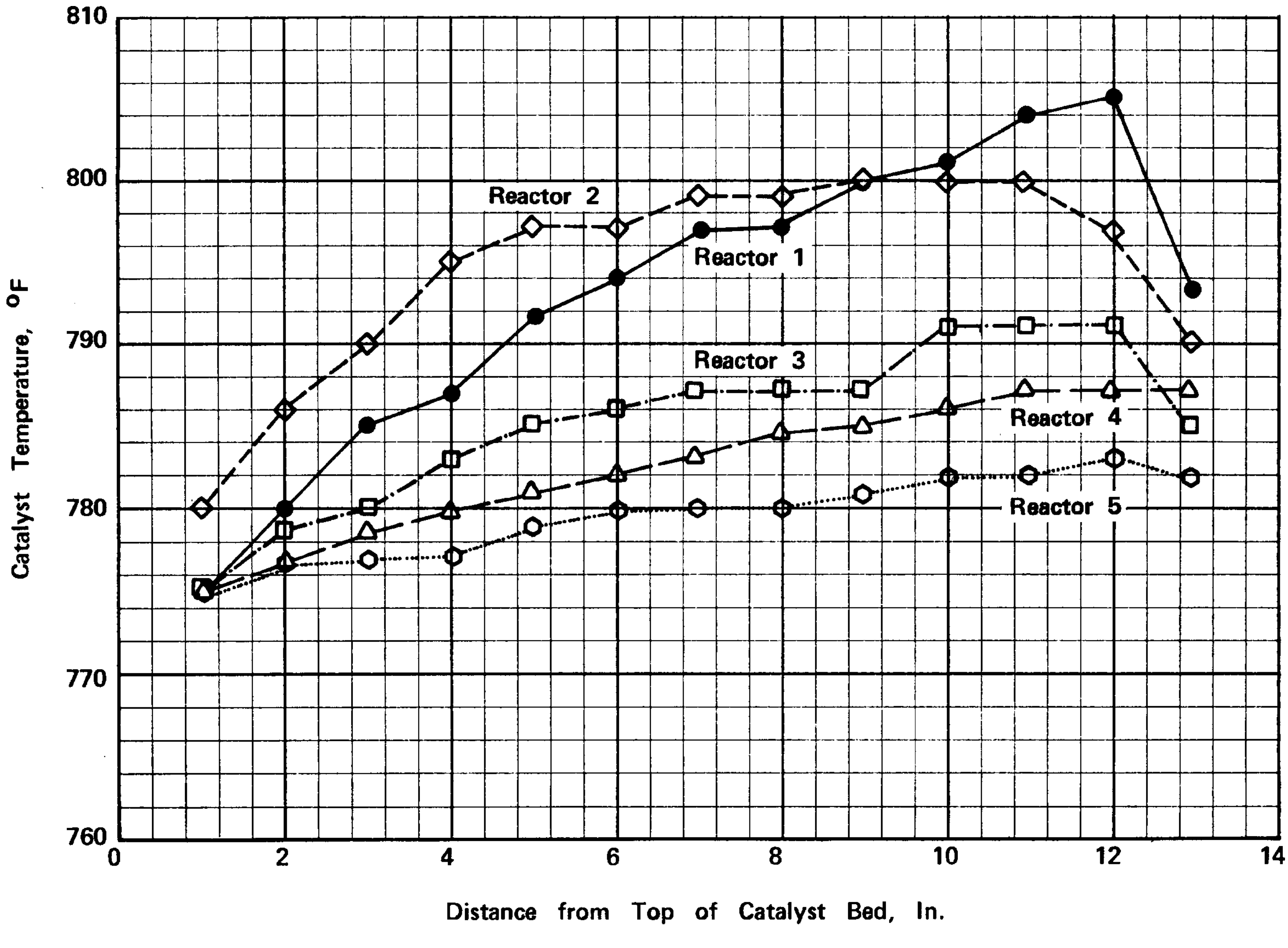
FIGURE 2

PILOT PLANT RUN 86–51
ERDA CONTRACT EF–76–C–01–2315
WHOLE SHALE OIL HYDROFINING WITH ICR 106

CATALYST TEMPERATURE PROFILE AFTER 1365 HR
AVERAGE CATALYST TEMPERATURE ~787°F



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RFS RE 772854

FIGURE 3

PILOT PLANT RUN 87–67

ERDA CONTRACT EF–76–C–01–2315

50/50 SRC/CREOSOTE OIL HYDROPROCESSING WITH ICR 106

CATALYST TEMPERATURE PROFILE AFTER 180 HR
AVERAGE CATALYST TEMPERATURE 746°F

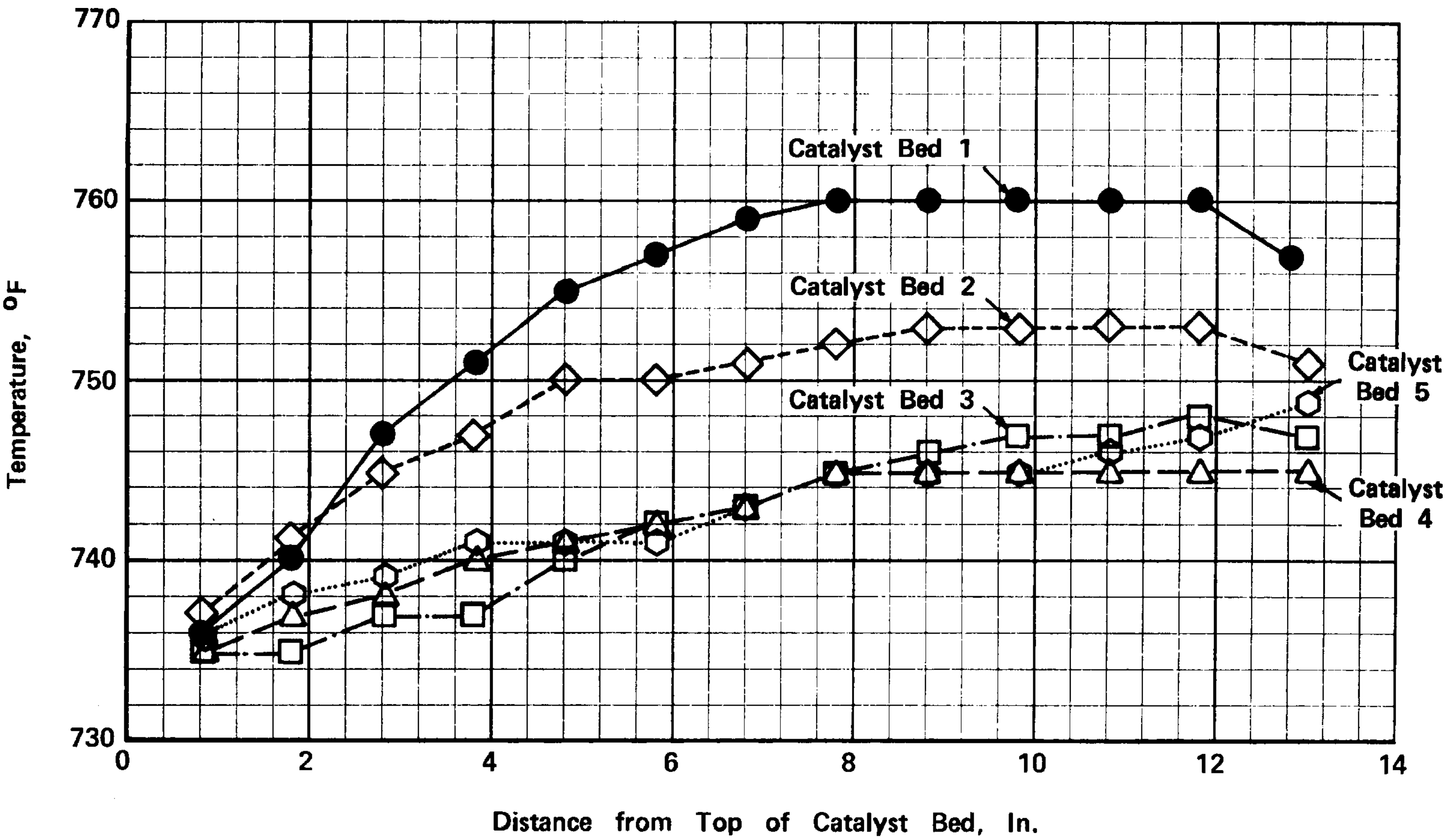

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FIGURE 4

PILOT PLANT RUN 87-67

ERDA CONTRACT EF-76-C-01-2315

50/50 SRC/CREOSOTE OIL HYDROPROCESSING WITH ICR 106

WHOLE LIQUID PRODUCT NITROGEN AND
AVERAGE CATALYST TEMPERATURE VERSUS RUN HOUR

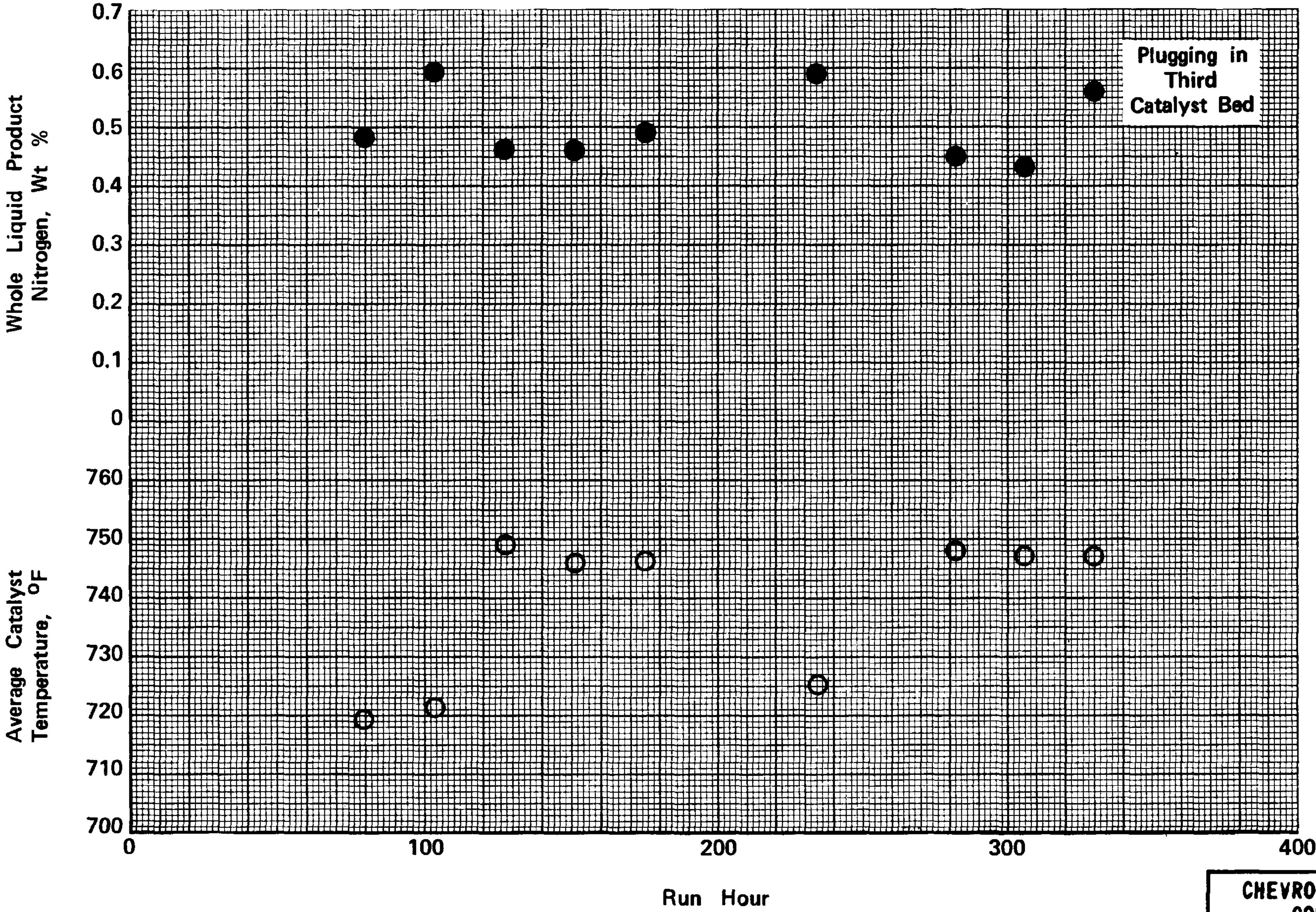

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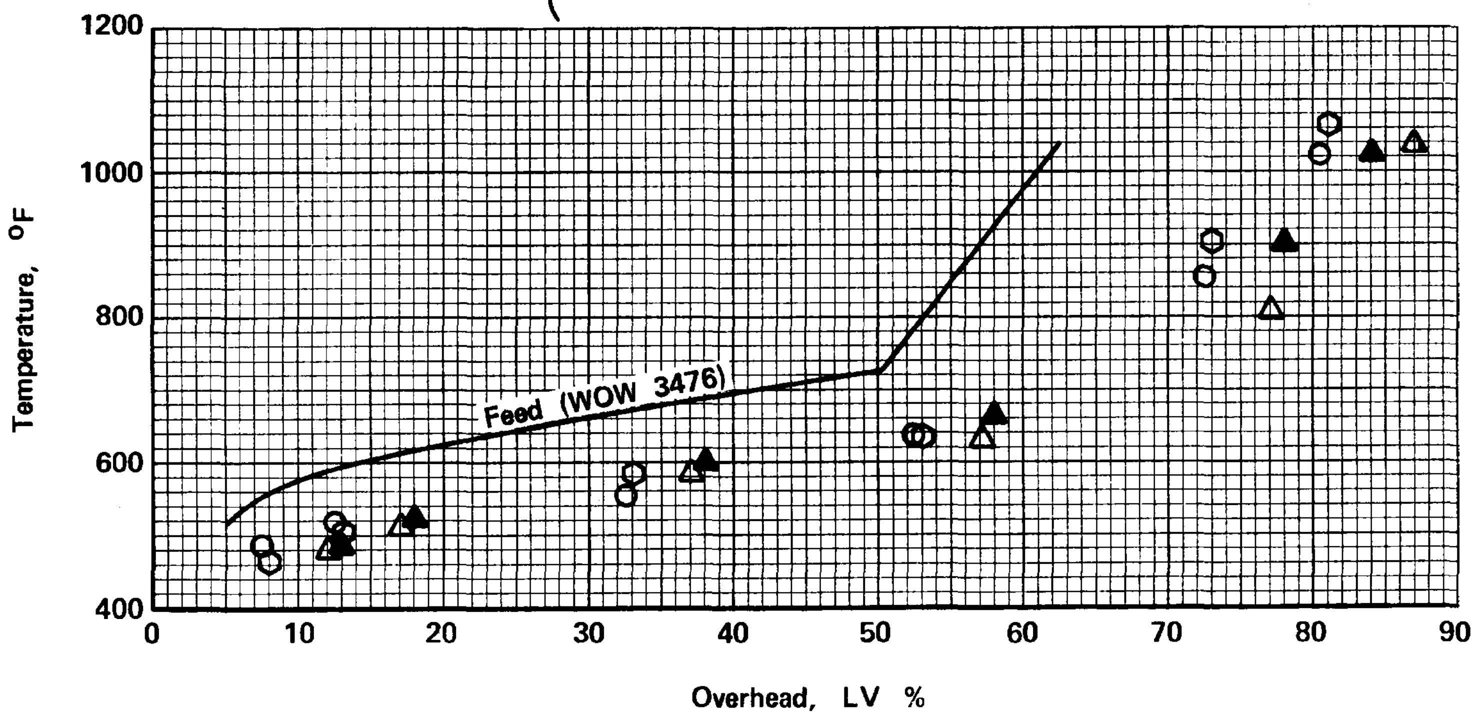
FIGURE 5

ASTM D 1160 (2 mm) DISTILLATIONS OF
FEED AND PRODUCTS OF RUN 87–67

	Run Hr	Avg. LHSV	Avg. Catalyst Temp., °F
Products	○ 103	0.18	721
	▲ 151	0.20	746
	⬡ 234	0.22	725
	△ 306	0.20	747



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