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

Quarterly Report for the
Period January-March 1977

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PREPARED FOR
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Abstract

The objective of this program is to determine the feasibility and estimate the economics of hydroprocessing four synthetic fuels to distillate fuels, including high octane gasoline, using presently available technology. The feedstocks include three coal-derived synthetic crudes and shale oil. The first feedstock is Paraho crude shale oil, produced in the indirectly heated mode.

Whole shale oil was hydrofined in a 2000-hour pilot plant test using ICR 106 catalyst. The results show that shale oil containing 2.2% nitrogen can be hydrofined to residuum-free product containing 1-2 ppm nitrogen in a single stage. Process design studies indicate that it is preferable to hydrofine the whole shale oil to about 500 ppm nitrogen and then to fractionate the product before conventional downstream processing to produce transportation fuels. The product resembles the fraction of a waxy petroleum crude boiling below 1000°F. This report includes yields and product properties determined from the small-scale pilot plant test. A larger-scale pilot plant demonstration run is now in progress.

I. Contract Objectives and Scope of Work

The objective of the program is to determine the feasibility and estimate the economics of hydroprocessing four synthetic crude feedstocks to distillate fuels, including high octane gasoline, using presently available technology. The four feedstocks are: a Chevron pyrolysis liquid from coal; a catalytic hydroliquefaction liquid which will be produced by the Synthoil or H-Coal processes; solvent refined coal of the type produced at the ERDA pilot plant in Tacoma, Washington; and shale oil.

The feasibility of hydroprocessing each of the synthetic liquids mentioned above will be compared through catalyst tests and evaluations whereby commercial plant yields, hydrogen consumption, product distribution, and product inspection will be estimated. The necessary tests and

Encl. - Table I (RD 771980)
Table II (RE 771341-1)
Table III (RE 771342-1)
Tables IV-A Through IV-E
(RE 771958-1-RE 711962-1)
Tables V-A Through V-E
(RE 771974-1-RE 771975-1,
RD 771976-1-RD 771978-1)
Figures 1-8 (RD 753220-1, RD 771945,
RD 770712-1, RE 771234-1, RE 770736-1,
RE 771235-1, RE 771936, and RE 771937)

evaluations for each feedstock will be done to support "process comparison" type estimates for each of the major refining steps. The results of the contract, insofar as hydroprocessing is concerned, will be obtained with the commercial catalysts identified in the contract.

Catalyst activity and stability information for each feedstock will be obtained as needed to define commercial operating conditions. These data will provide the basis for the overall refining plan, plant cost estimates, utility and hydrogen requirements, etc. If tests show that refining a particular feedstock using presently existing information is not feasible, it is not intended under this program to conduct any research or development work to solve the problems encountered.

Tests will be conducted only to the extent needed to enable making reasonable estimates of commercial plant performance and only to the extent a commercial plant is feasible using presently existing technology, subject to the mutual agreement of ERDA and Chevron Research. Tests will be made for each whole synthetic oil and, where appropriate, for the fractions derived therefrom. Tests will not be carried out for processes which can be reliably estimated. No tests will be included for hydrogen manufacture or use of chars from pyrolysis or coking operations.

II. Summary of Progress to Date

It was agreed that the first feedstock to be evaluated is to be a shale oil. ERDA supplied shale oil produced during operating in the indirect heating mode at the Paraho retort.

Figure 1 shows the original timing estimate for an individual feedstock. Figure 2 is the revised schedule as it applies to shale oil processing studies, which started with the arrival of the feedstock in mid-September 1976.

Based on results to date, the key process in the overall processing scheme is the hydrofining of the shale oil. With the permission of the ERDA project manager, pilot plant studies of the hydrofining of shale oil have been extended beyond the original schedule. During the course of this task, feed for downstream processing studies is being prepared. It is planned to hydrocrack the 650°F+ portion of the hydrofined shale oil in a single stage rather than a two-stage process as was originally proposed. Therefore, the downstream processing pilot plant studies will take less time than originally proposed, and we expect to complete our studies of shale oil processing on schedule.

III. Description of Technical Progress

Task I - Feedstock Analysis

Table I summarizes inspections to date for the pilot plant feed, whole Paraho shale oil.

From the filter residue ash tests using X-ray diffraction, the ash was shown to consist of $Ca_2Al_2SiO_7$, $CaMgSi_2O_7$, and Fe_3O_4. The filtrate from this test was analyzed for metals by emission spectrochemical analysis. Results are given in Table II.

In particular, it should be noted that the filtrate contains 70 ppm of iron that is not removed by the 0.45-μ filter. In addition to the metals listed in Table I, the shale oil contains 28 ppm of arsenic that is also not removed by filtration.

Task 2 - Whole Shale Oil Hydrofining

Run 81-4

It was reported previously that in catalyst screening tests, ICR 106 was selected as the catalyst of choice for whole shale oil hydrofining. Pilot plant Run 81-4, started as the screening test for ICR 106 catalyst, was continued to obtain preliminary yield information, to determine catalyst deactivation rate (fouling rate), and to study the effects of process variables on catalyst performance.

Figure 3 summarizes the conditions and whole liquid product nitrogen for the first 1200 hours on-stream. Catalyst behavior is typical of that observed for many hydroprocessing catalysts for processing high end point feedstocks. The catalyst undergoes a transient activity loss early in the run which is attributed to "equilibration" with the feed. Following this transient period, the catalyst reaches a stable or "lined out" activity; and the fouling rate is very low. In the case of ICR 106 for whole shale oil hydrofining, the transient period is about 750 hours. Figure 3 shows that between 750 and 1200 hours on-stream, no catalyst deactivation or fouling can be detected within the limits of the experimental data.

Figure 4 shows additional tests with ICR 106 in pilot plant Run 81-4. After about 1200 hours on-stream, the temperature was increased from 745°F to 767°F. Product nitrogen decreased to approximately 700 ppm.

Figure 4 also shows that about 100 hours after the temperature increase, the liquid hourly space velocity (LHSV) was lowered to 0.3, half of its previous value. As a result, the product nitrogen was lowered to 30 ppm.

At 1440 hours on-stream, the LHSV was decreased to 0.2. After 150 hours at this low LHSV, the product nitrogen decreased to 1.6 ppm. It should be pointed out, however, that the pilot plant is not ideally suited for operation at 0.2 LHSV. The total catalyst volume is 130 ml, and the feed rate is only 26 ml/hr. Because of the relatively large holdup in the product high pressure separator and low pressure product handling system, a large volume of higher nitrogen product from the previous period of operation remains to contaminate the low nitrogen product for a relatively long period. Also, with the high level of denitrification required to convert 2.18% nitrogen shale oil feed to 1-2 ppm nitrogen product, any effects of channeling or catalyst bypassing is emphasized in this single-reactor system. Maintaining the desired recycle gas rate is also difficult at the low gas throughput required at 0.2 LHSV. Despite these operation problems, the results of this experiment are encouraging and demonstrate that essentially all of the nitrogen can be removed from shale oil in a single hydrofining step.

Figure 5 compares the liquid product boiling range at three product nitrogen levels with whole shale oil. As expected, more hydrocracking occurs at the higher levels of denitrification; the less nitrogen in the product, the lower its boiling range. Essentially all of the 1000°F+ residuum has been hydrocracked to lower boiling product at product nitrogen levels of 1000 ppm and lower.

Preliminary process design studies indicate that rather than denitrifying to 1 ppm, it is more economically attractive to reduce the product nitrogen to 500-700 ppm in the whole shale oil hydrofiner. The lighter products can then be separated from the higher boiling fraction before further hydrocracking. Therefore, after the brief 0.2 LHSV study described above, the LHSV was changed back to 0.6 in Run 81-4. Figure 4 shows that within experimental error, no activity was lost during the periods at the lower space velocities.

Figures 3 and 4 also show no catalyst deactivation can be detected for about 1000 hours between 750 and 1750 hours on-stream. However, after about 1800 hours on-stream, a rapid catalyst deactivation occurred. This was accompanied by problems in maintaining gas flow across the catalyst bed and plugging within the reactor. Because of pressure drop problems and the apparent loss of catalyst activity, the run was shut down after about 2000 hours on-stream.

The catalyst was removed from the reactor in layers for analysis. The catalyst itself was free flowing, and there appeared to be no plugging in the catalyst bed itself. Rather, the plugging was due to a large deposit of black material in the preheat section of the reactor. This deposit appears to be the result of thermal decomposition of constituents of the shale oil in the presence of hydrogen prior to any contact of the feed with the catalyst. This deposit was analyzed by a combination of energy-dispersive and wavelength-dispersive X-ray fluorescence as follows: 30% iron, 11.4% arsenic, 2.8% zinc, 1% calcium, and 0.23% selenium. Analyses for carbon and sulfur are incomplete.

Table III shows the arsenic and iron distribution throughout the catalyst bed as determined by X-ray fluorescence.

Run 86-50

A large-scale, whole shale oil hydrofining pilot plant run was made with 650 ml of whole ICR 106 catalyst (rather than the crushed catalyst that was used in Run 81-4). This unit contains six reactors in series. The first reactor was charged with 130 ml of a low cost material to serve as a guard bed for removal of a large fraction of the feed arsenic and iron. The remaining five reactors each contained 130 cc of ICR 106 catalyst. This run was made at 0.6 LHSV, based on the ICR 106 catalyst volume. Other conditions were 2200 psig total pressure, 5000 SCF/B recycle gas rate, and 500 ppm target product nitrogen.

The purpose of Run 86-50 was to serve both as a demonstration run for whole shale oil hydrofining and as a feed preparation run for the scheduled downstream processing studies.

Figure 6 is a summary of average catalyst temperature and observed product nitrogen. After 250 hours on-stream, the run was interrupted in order to reinsulate the furnace. Upon restarting, a plug developed in a 1/4-in. external diameter Aminco preheat line (0.081-in. internal diameter) carrying the feed to the guard bed. The material that caused the plug was similar to the powdery black material that caused the preheat plugging problem in the 2000-hour pilot plant test, Run 81-4. The temperature of the line that plugged in Run 86-50 was approximately 750°F.

Run 86-51

In order to avoid the plugging problem that occurred in Run 86-50, the preheat and guard bed section of the pilot plant was altered so that the feed and hydrogen mixture is first heated in the guard bed itself rather than the very narrow preheat line. The guard bed is a reactor with an

0.951-in. internal diameter filled with the low cost material to provide a surface for deposition of iron, arsenic, and other materials that might otherwise act as catalyst poisons. The shale oil-hydrogen mixture moves upward through the 352-ml guard bed, then through a short segment of 0.359-in. internal diameter tubing, then downflow through an additional 83 ml of guard bed before reaching the first catalyst zone.

With this new arrangement, it is hoped that small amounts of solid deposits will not cause plugging. Therefore, sudden decreases in recycle gas rate and the resulting catalyst deactivation are much less likely to occur.

In Run 86-51, the recycle gas rate is 8000 SCF/B rather than 5000 SCF/B as used in the previous run. This higher target gas rate allows the operator of the pilot plant additional time to make appropriate corrections to prevent catalyst damage should any plugging or pressure drop problems occur.

In contrast to the smaller-scale pilot plant tests which were essentially isothermal, Runs 86-50 and 86-51 show an exothermic reaction occurs in the first catalyst bed. The catalyst in the first bed reaches a temperature about 30°F higher than the overall average catalyst temperature. Also, the second catalyst bed operates at a temperature about 10°F higher than the average. Figure 7 shows the temperature profile across each of the five catalyst beds after about 500 hours on-stream in Run 86-51. Changes in temperature gradients as the run progresses should provide us with a means of following catalyst deactivation.

More light gases (methane and ethane) are produced in the runs with the temperature profile than the small-scale isothermal run. As a result, the hydrogen content of the recycle gas is lower. Therefore, in order to maintain the target hydrogen pressure of about 1750-1800 psia in Run 86-51, the total pressure was increased to 2350 psig from 2200 psig after 225 hours on-stream.

Figure 8 is a condensed run summary of Run 86-51 to 600 hours on-stream. Catalyst temperature at 600 hours is 765°F, and the product nitrogen is 500 ppm. The catalyst activity is in good agreement with results of the small-scale test, Run 81-4. After the initial "transient" activity change, the catalyst appears stable, and the fouling rate appears to be low. Additional time on-stream will be required to define the catalyst fouling rate.

Yields and Product Properties

We are accumulating a considerable amount of yield information and product inspections in each of the runs. Tables IV and

V summarize some of the results from Runs 81-4 and 86-50. Additional yield periods and analyses are being obtained and will be reported later.

IV. Planned Work

We have enough feed to continue the whole shale oil hydrofining demonstration run for approximately another 1000 hours. We are expecting to obtain additional feed from ERDA to continue this run beyond that time. We believe that the initial hydrofining step is the key process for handling the shale oil commercially in a conventional petroleum refinery: therefore, our efforts to date have been concentrated on this step. The product from the whole shale oil hydrofining in most ways resembles the portion of a waxy petroleum crude boiling below 1000°F. Therefore, we do not anticipate unusual problems in downstream processing this product to produce transportation fuels. We are producing hydrofined product at the rate of about 2.5 gal./day and will begin our downstream processing studies of this product within a few weeks, as shown in the schedule in Figure 2.

Samples of solvent refined coal (SRC) have been provided by the Pittsburg and Midway Coal Mining Company from their facility at Du Pont, Washington. This will be the second feedstock under ERDA Contract EF-76-C-01-2315. Processing studies with SRC as feed will begin this month (April 1977).

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ERDA Technical Information
Center-1 (Reproduction Master)

TABLE I

PROPERTIES OF DEWATERED PARAHO SHALE OIL, WOW 3394
(PRODUCED BY INDIRECTLY HEATED MODE)

	Pilot Plant Feed
Gravity, °API	20.2
Sulfur, Wt %	0.66
Total Nitrogen, Wt %	2.18
Oxygen, Wt %	1.16
Arsenic, ppm	28
Pour Point, ASTM, °F	90
Carbon, Wt %	83.13
Hydrogen, Wt %	11.43
Hydrogen/Carbon Atom Ratio	1.64
Chloride, ppm	<0.2
Sodium, ppm	5
Ash, Wt % (ASTM D 486)	0.03
Filter Residue Ash (0.8 μ-Filter)	
Total Solids, ppm	234
Ash, ppm	168
Filter Residue Ash (0.45 μ-Filter)	
Total Solids, ppm	252
Ash, ppm	194
Sediment (Plus Trace Water), Vol %	0.1
Bromine No.	51
Average Molecular Weight	326
Viscosity, cSt	
122°F	25.45
210°F	5.541
Acid Neutralization No., mg KOH/g	2.3
Base Neutralization No., mg KOH/g (Equivalent)	38
pH	9.2
Maleic Anhydride No., mg/g	40.6
Navy Heater Test	No. 1 Rating (1, 1, 1)
Hot Heptane Asphaltenes (Including Any Fines), Wt %	0.17
ASTM D 1160 Distillation, °F (At 10 mm)	
St/5	386/456
10/30	508/659
50	776
70/90	871/995
EP	/1022 (94%)
% Overhead (Excl. Trap)	94
% in Trap	1
% in Flask	5

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TABLE II

ANALYSIS OF PARAHO SHALE OIL
FOR METALS BY EMISSION SPECTROCHEMICAL METHOD

Identification	Pilot Plant Feed, WOW 3394	Filtrate from WOW 3394	
Size Filter, μ	15	0.8	0.45
Metals, ppm			
Aluminum	5	-	1.0
Boron	0.3	0.2	0.2
Calcium	20	0.1	0.1
Chromium	0.04	0.1	0.1
Cobalt	0.6	0.9	0.9
Copper	0.1	0.1	0.1
Iron	>11	>9	70
Magnesium	>3	1.9	1.9
Nickel	1.8	2.4	2.3
Silicon	>15	0.5	0.2
Tin	-	0.7	0.6
Titanium	0.8	0.1	0.2
Vanadium	0.2	0.2	0.2
Zinc	2.8	4.3	5.3

TABLE III

ARSENIC AND IRON IN
AGED CATALYST FROM RUN 81-4
AFTER PROCESSING 1060 VOLUMES WHOLE SHALE OIL
PER VOLUME OF CATALYST

Layer No.	Wt of Layer, g*	Cumulative (From Top), g	Iron, Wt %	Arsenic, Wt %
1	4	4	3.4	3.3
2	5	9	2.5	2.0
3	11	20	1.7	1.3
4	11	31	1.3	0.89
5	10	41		
6	11	52	0.90	0.50
7	10	62		
8	11	73	0.75	0.28
9	10	83		
10	13	96	0.20	0.062
11	12	108		
12	9	117		
13	12	129		
14	5	134	0.043	0.0083

*In addition, a total of 5.6 g of fines (smaller than 24 mesh) was distributed throughout the bed. Analyses are incomplete.

TABLE IV-A

HYDROFINING OF WHOLE SHALE OIL WITH ICR 106

Whole Liq. Prod. N, ppm	1325		2800		645		505	
RUN	81- 4		81- 4		81- 4		81- 4	
FEED	WOW 3394		WOW 3394		WOW 3394		WOW 3394	
RUN HOURS	556.0- 580.0		844.0- 868.0		1252.0-1276.0		1636.0-1660.0	
AVG.CAT.TEMP., F.	744.		742.		766.		767.	
LHSV	0.59		0.61		0.58		0.59	
TOTAL PRESSURE,PSIG	2202.		2203.		2206.		2199.	
H2 MEAN PRESS.,PSIA	1874.		1824.		1858.		1846.	
TOTAL GAS IN,SCF/B	12304.		6522.		7370.		7226.	
RECYCLE GAS, SCF/B	10308.		4906.		5195.		5132.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.
C1	0.42		0.39		0.32		0.27	
C2	0.78		0.66		0.80		0.69	
C3	0.70		0.59		0.85		0.76	
I-C4	0.15	0.25	0.13	0.21	0.19	0.31	0.17	0.28
N-C4	0.48	0.77	0.38	0.61	0.57	0.91	0.53	0.85
C5-400 F.	10.56	13.01	11.19	14.29	12.36	15.23	13.24	16.32
400-650 F.	36.61	39.84	35.08	38.99	39.32	43.78	38.72	43.08
650-800 F.	22.95	24.53	23.18	24.64	22.61	24.27	22.56	24.24
800- EP F.	25.82	26.94	26.29	27.19	21.76	22.93	21.72	22.91
TOTAL C5+	95.96	104.33	95.75	105.03	96.06	106.22	96.26	106.57
ACT./NO LOSS RECOV.	102.16/103.13		100.94/102.54		102.05/103.44		99.84/103.32	
H2 CONS(GROSS),SCF/B	1996.		1616.		2175.		2094.	
H2 CONS(HCSON),SCF/B	1923.		1558.		2110.		2038.	

....LIQUID PRODUCT INSPECTIONS....

C5-400 F. PRODUCT[1]				
GRAVITY, API	50.6	60.5	51.4	52.2
ANILINE PT., F.	128.2	127.3	128.2	129.0
SULFUR, PPM.	30.0		14.0	25.0
NITROGEN, PPM.	513.00	506.00	125.00	67.00
LOW MASS,LV.PC.				
PARAFFINS	48.7	49.4	47.3	49.4
NAPHTHENES	38.7	36.8	40.7	38.9
AROMATICS	12.6	13.8	12.0	11.6
O.N.,F-1 CLEAR			38.0	38.1
BROMINE NO.				0.60
VISCOSITY, CS,100F.			0.834	
VISCOSITY, CS,122F.			0.740	
TBP DIST., F.				
ST/ 5	94./ 206.	85./ 190.	99./ 198.	60./ 185.
10/30	240./ 295.	215./ 285.	222./ 285.	213./ 278.
50	336.	330.	324.	322.
70/90	365./ 395.	365./ 401.	356./ 392.	356./ 395.
95/99	406./ 428.	412./ 463.	408./ 467.	409./ 444.

[1] Inspections uncorrected for any C_5-C_6 measured as gas.

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TABLE IV-B
HYDROFINING OF WHOLE SHALE OIL WITH ICR 106

Whole Liq. Prod. N, ppm	1325	2800	645	505
RUN	81- 4	81- 4	81- 4	81- 4
FEED	WOW 3394	WOW 3394	WOW 3394	WOW 3394
RUN HOURS	556.0- 580.0	844.0- 868.0	1252.0-1276.0	1636.0-1660.0
AVG.CAT.TEMP., F.	744.	742.	766.	767.
LHSV	0.58	0.61	0.58	0.59
....LIQUID PRODUCT INSPECTIONS....				
400-650 F. PRODUCT				
GRAVITY, API	33.7	36.8	37.5	37.4
ANILINE PT., F.	154.3	151.7	153.7	155.5
SULFUR, PPM.	3.7	35.0	14.0	6.0
NITROGEN, PPM.		388.00	453.00	356.00
MOLECULAR WEIGHT	198.	207.	202.	196.
POUR PT.,ASTM, F.	5.	5.		-5.
FREEZE PT.,ASTM, F.				9.
HIGH MASS,LV.PC.				
PARAFFINS	34.5	34.3	33.9	33.6
NAPHTHENES	45.5	45.2	46.5	46.5
AROMATICS	20.0	20.5	19.5	19.8
CLOUD PT.,ASTM, F.			4.	4.
BROMINE NO.			3.00	2.40
VISCOSITY, CS,100F.		3.300	3.030	
VISCOSITY, CS,122F.		2.480	2.390	
VISCOSITY, CS,210F.		1.250	1.180	
TBP DIST., F.				
ST/ 5	328./ 410.	341./ 412.	336./ 396.	322./ 402.
10/30	427./ 485.	429./ 487.	420./ 477.	425./ 482.
50	532.	536.	527.	530.
70/90	583./ 624.	585./ 632.	580./ 625.	581./ 626.
95/99	638./ 669.	649./ 751.	640./ 683.	643./ 763.
ASTM D-86 DIST.,F				
ST/ 5	441./ 455.	446./ 464.	431./ 451.	
10/30	465./ 502.	467./ 494.	458./ 486.	
50	532.	521.	514.	
70/90	554./ 583.	553./ 588.	547./ 582.	
95/EP	610./ 616.	597./ 621.	597./ 620.	
LV.PC.OVHD	99.5	99.5	99.5	
COLOR, ASTM				2.0

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TABLE IV-C

HYDROFINING OF WHOLE SHALE OIL WITH ICR 106

Whole Liq. Prod. N, ppm	1325	2800	645	505
RUN	81- 4	81- 4	81- 4	81- 4
FEED	WOW 3394	WOW 3394	WOW 3394	WOW 3394
RUN HOURS	556.0- 580.0	844.0- 868.0	1252.0-1276.0	1636.0-1660.0
AVG.CAT.TEMP., F.	744.	742.	766.	767.
LHSV	0.58	0.61	0.58	0.59
....LIQUID PRODUCT INSPECTIONS....				
650-800 F. PRODUCT				
GRAVITY, API	30.7	29.9	31.5	31.6
ANILINE PT., F.	184.9	179.3	188.0	187.3
SULFUR, PPM.	20.0	55.0	13.0	11.0
NITROGEN, PPM.	1600.00	3000.00	960.00	702.00
MOLECULAR WEIGHT	314.	318.	310.	310.
POUR PT.,ASTM, F.		70.	75.	70.
22 COMP., LV. PC.[1]				
PARAFFINS			(24.6)	(32.3)
NAPHTHENES			(59.7)	(40.6)
AROMATICS			(15.4)	(26.9)
SULFUR COMPOUNDS			(0.3)	(0.2)
BROMINE NO.			5.50	5.70
VISCOSITY, CS,100F.		16.790	14.479	
VISCOSITY, CS,122F.		11.170	9.970	
VISCOSITY, CS,210F.		3.440	3.160	
TBP DIST., F.				
ST/ 5	534./ 637.	407./ 633.	463./ 635.	479./ 618.
10/30	653./ 687.	652./ 694.	651./ 689.	642./ 688.
50	723.	731.	724.	727.
70/90	757./ 792.	766./ 801.	758./ 793.	767.
95/99	807./ 842.	814./ 844.	806./ 836.	

[1]These 22-component group-type analyses do not appear consistent and may be in error.

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TABLE IV-D

HYDROFINING OF WHOLE SHALE OIL WITH ICR 106

Whole Liq. Prod. N, ppm	1325	2800	645	505
RUN	81- 4	81- 4	81- 4	81- 4
FEED	WOW 3394	WOW 3394	WOW 3394	WOW 3394
RUN HOURS	556.0- 580.0	844.0- 868.0	1252.0-1276.0	1636.0-1660.0
AVG.CAT.TEMP., F.	744.	742.	766.	767.
LHSV	0.58	0.61	0.58	0.59
....LIQUID PRODUCT INSPECTIONS....				
600- EP F. PRODUCT				
GRAVITY, API	26.9	25.5	28.4	28.6
ANILINE PT., F.	218.0	208.7	225.8	228.6
SULFUR, PPM.	20.0	65.0	<10.0	20.0
NITROGEN, PPM.	1600.00	4500.00	995.00	710.00
MOLECULAR WEIGHT	453.	472.	437.	452.
POUR PT.,ASTM, F.		110.	115.	115.
22 COMP., LV. PC. [1]				
PARAFFINS			(32.7)	(20.6)
NAPHTHENES			(40.1)	(57.6)
AROMATICS			(27.0)	(21.3)
SULFUR COMPOUNDS			(0.2)	(0.5)
BROMINE NO.			6.90	5.80
VISCOSITY, CS,122F.		74.000	41.480	55.169
VISCOSITY, CS,210F.		11.640	8.820	8.770
TGA DISTILLATION				
ST/ 5	159./ 749.	135./ 780.		
10/30	797./ 877.	816./ 884.		
50	918.	919.		
70/90	949./ 993.	950./ 997.		
95/EP	1017.	1043.		
LV.PC.OVHD	98.0	96.3		
TBP DIST., F.				
ST/ 5			518./ 786.	
10/30			801./ 841.	
50			876.	
70/90			923.	
95/99				
ASTM D-1160 DIST.,F				
ST/ 5				809./ 821.
10/30				825./ 833.
50				858.
70/90				900./ 976.
95/EP				1010./1073.
LV.PC.OVHD				99.0

[1]These 22-component group-type analyses do not appear consistent and may be in error.

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TABLE IV-E
HYDROFINING OF WHOLE SHALE OIL WITH ICR 106

Whole Liq. Prod. N, ppm	1325	2800	645	505
RUN	81- 4	81- 4	81- 4	81- 4
FEED	WOW 3394	WOW 3394	WOW 3394	WOW 3394
RUN HOURS	556.0- 580.0	844.0- 868.0	1252.0-1276.0	1636.0-1660.0
AVG.CAT.TEMP., F.	744.	742.	766.	767.
LHSV	0.58	0.61	0.58	0.59
....LIQUID PRODUCT INSPECTIONS....				
WHOLE LIQUID PRODUCT				
GRAVITY, API	34.1	34.4	35.7	36.2
ANILINE PT., F.	179.0	173.5	177.8	178.1
SULFUR, PPM.	33.0	145.0	42.0	45.0
NITROGEN, PPM.	1325.00	2800.00	645.00	505.00
MOLECULAR WEIGHT	291.	289.	279.	236.
POUR PT.,ASTM, F.	55.	80.	80.	85.
BROMINE NO.	3.90	5.90	2.80	3.00
22 COMP., LV. PC.				
PARAFFINS	38.8	33.8	37.8	
NAPHTHENES	38.2	36.7	37.1	
AROMATICS	22.9	29.3	24.2	
SULFUR COMPOUNDS	0.1	0.2	0.9	
VISCOSITY, CS,100F.	7.390	7.429	5.730	5.570
VISCOSITY, CS,210F.	2.150	2.170	1.860	1.880
TBP DIST., F.				
ST/ 5		134./ 299.	136./ 286.	100./ 285.
10/30		373./ 520.	347./ 491.	349./ 492.
50		638.	604.	604.
70/90		762./ 898.	729./ 890.	724./ 874.
95/99				
ASTM D-1160 DIST.,F				
ST/ 5	329./ 440.	328./ 422.	346./ 428.	302./ 397.
10/30	456./ 573.	451./ 569.	450./ 561.	435./ 550.
50	672.	683.	667.	666.
70/90	788./ 908.	791./ 932.	769./ 913.	765./ 903.
95/EP	1016.	968./1021.	1012.	1003.
LV.PC.OVHD (Excl. Trap)	94.0	95.0	93.0	93.0
Trap, %	2.5	4	6	6
In Flask, %	3.5	1	1	1
Hot Heptane Asphaltenes, ppm	56	108	22	15

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TABLE V-A

HYDROFINING OF WHOLE SHALE OIL WOW 3394 WITH ICR 106 AT 640 PPM WHOLE PRODUCT NITROGEN

Run	81-4		86-50	
Run Hours	1660-1684		175-223	
Avg. Cat. Temp., °F	775		760	
LHSV	0.60		0.61	
Total Pressure, psig	2199		2199	
H_2 Mean Pressure, psia	1836		1558	
Total Gas In, SCF/B	6970		6583	
Recycle Gas, SCF/B	4923		4769	
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %
C_1	0.35		0.45	
C_2	0.78		0.59	
C_3	0.81		0.54	
$I-C_4$	0.18	0.30	0.12	0.19
$N-C_4$	0.54	0.85	0.32	0.50
C_5-180°F	5.56	7.29	1.62	2.30
180-300°F			5.25	6.54
300-550°F	30.22	34.32	32.64	36.96
550-650°F	17.52	19.20	17.18	18.81
650°F-End Point	42.56	45.08	39.60	41.95
Total C_5+	95.97	105.89	96.28	106.55
Act./No Loss Recov.	100.61/103.24		99.14/102.91	
H_2 Cons. (Gross), SCF/B	2047		1814	
H_2 Cons. (Chemical), SCF/B	1986		1782	
Whole Liquid Product Insp.				
Gravity, °API	35.6		35.8	
Sulfur, ppm	18		43	
Nitrogen, ppm	640		630	
TBP Distillation, °F (Simulated by Chromatography)				
St/5	90/278		81/267	
10/30	343/489		331/476	
50	605		586	
70/90	724/867		702/852	
95	966/		911/	
Hot Heptane Asphaltenes, ppm			76	
Ramsbottom Carbon, %			0.10	

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TABLE V-B

HYDROFINING OF WHOLE SHALE OIL WOW 3394
WITH ICR 106 AT 640 PPM WHOLE PRODUCT NITROGEN

Run	81-4	86-50
Run Hours	1660-1684	175-223
Avg. Cat. Temp., °F	775	760
H_2 Mean Press., psia	1836	1558
Liquid Product Inspections		
C_5-180°F		
Gravity, °API		84.6
Nitrogen, ppm		10
180-300°F	[1]	
Gravity, °API	58.8	57.6
Aniline Point, °F	129.4	124.4
Sulfur, ppm	5.9	60
Nitrogen, ppm	5.1	11.4
Octane, F-1 Clear		51.3
Low Mass, LV %		
Paraffins	52.1	50.4
Naphthenes	38.0	39.3
Aromatics	9.9	10.3
TBP Distillation, °F (Simulated by Chromatography		
St/5	54/148	(48)/179
10/30	174/223	193/233
50	256	256
70/90	280/303	278/296
95/99	311/329	305/(468)

[1]A small amount of 180°F- is present.

TABLE V-C

HYDROFINING OF WHOLE SHALE OIL WOW 3394
WITH ICR 106 AT 604 PPM WHOLE PRODUCT NITROGEN

Run	81-4	86-50
Run Hours	1660-1684	175-223
Avg. Cat. Temp., °F	775	760
H_2 Mean Pressure, psia	1836	1558
Liquid Product Inspections		
300-550°F		
Gravity, °API	40.9	40.4
Aniline Point, °F	140.7	136.8
Sulfur, ppm	1.5	6.2
Nitrogen, ppm	276	310
Molecular Weight	178	171
Cloud Point, °F		-46
Pour Point, °F	-45	-50
Freeze Point, °F	-32	-31
Smoke Point, mm	21	18
High Mass, LV %		
Paraffins	31.5	32.6
Naphthenes	46.2	42.4
Aromatics	22.2	25.0
FIAM, LV %		
Paraffins Plus Naphthenes	78	75
Aromatics	22	25
Viscosity, cSt, 122°F	1.455	1.451
ASTM D 86 Distillation, °F		
St/5	356/371	345/365
10/30	378/410	379/412
50	439	441
70/90	467/495	470/500
95/EP	505/528	510/521
% Overhead	99	99
TBP Distillation of (Simulated by Chromatography)		
St/5	263/321	270/321
10/30	343/409	342/410
50	455	454
70/90	496/534	495/535
95/99	547/564	548/-
Bromine Number		1.6
Color, ASTM D 156		-16
Copper Strip, ASTM D 130	1A	1B
Doctor Test	Negative	Negative
Flash Point, °F	146	
Oxygen, Wt %	0.007	

TABLE V-D

HYDROFINING OF WHOLE SHALE OIL WOW 3394 WITH
ICR 106 AT 640 PPM WHOLE LIQUID PRODUCT NITROGEN

Run	81-4	86-50
Run Hours	1660-1684	175-223
Avg. Cat. Temp., °F	775	760
H_2 Mean Press., psia	1836	1558
Liquid Product Inspections		
550-650°F		
Gravity, °API	34.9	34.7
Aniline Point, °F	169.8	166.7
Sulfur, ppm	1.3	4.9
Nitrogen, ppm	430	805
Molecular Weight	247	246
Cloud Point, °F		+28
Pour Point, °F	+25	+25
Freeze Point, °F	+27	+36
Group Type, LV %		
High Mass		
Paraffins	36.4	
Naphthenes	47.4	
Aromatics	16.2	
22 Component		
Paraffins	43.0	41.8
Naphthenes	36.0	31.6
Aromatics	21.1	26.6
Viscosity, cSt, 122°F	4.10	4.21
Viscosity, cSt, 210°F	1.77	1.735
ASTM D 86 Distillation, °F		
St/5	565/573	566/571
10/30	576/580	573/576
5	584	581
70/90	590/603	587/599
95/EP	609/614	604/616
% Overhead	99.5	99.5
TBP Distillation, °F		
St/5	373/549	517/533
10/30	561/587	564/587
50	608	609
70/90	628/650	629/649
95/99	657/667	656/664
Bromine Number		2.2
Copper Strip, ASTM D 130	1A	
Doctor Test	Negative	
Oxygen, Wt %	0.032	

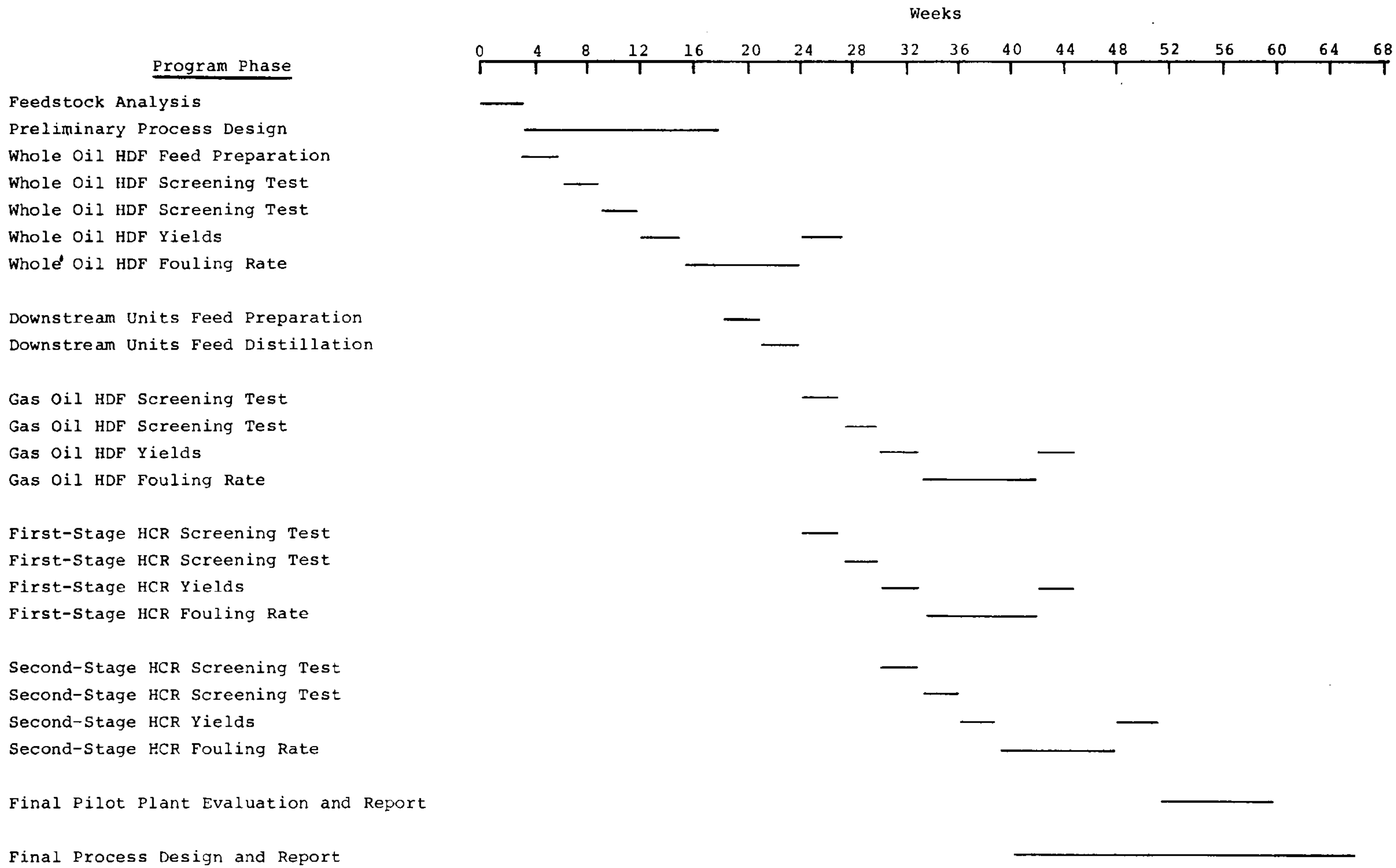
TABLE V-E

HYDROFINING OF WHOLE SHALE OIL WOW 3394 WITH
ICR 106 AT 640 PPM WHOLE LIQUID PRODUCT NITROGEN

Run No.	81-4	86-50
Run Hours	1660-1684	175-223
Avg. Cat. Temp., °F	775	760
H_2 Mean Press., psia	1836	1558
Liquid Product Inspections		
650°F+ Product		
Gravity, °API	29.3	29.3
Aniline Point, °F	212.7	208.3
Sulfur, ppm	20	20
Nitrogen, ppm	890	1405
Molecular Weight	391	379
Pour Point, °F	+105	+110
Group Type, LV %		
22 Component		
Paraffins	31.5	27.8
Naphthenes	43.2	35.2
Aromatics	25.3	37.1
Viscosity, cSt		
122°F	22.14	19.98
210°F	5.709	5.33
ASTM D 1160 Distillation, °F		
10 mm		
St/5	706/714	
10/30	721/754	
50	799	
70/90	859/931	
95/EP	976/1011	
% Overhead	95.5	
% In Trap	0	
% In Flask	4.5	
2 mm		
St/5	684/716	693/706
10/30	729/772	718/754
50	807	795
70/90	868/950	849/940
95/EP	993/1077	975/1054
% Overhead	99.5	97.5
% In Flask	0.5	2.5
Hot Heptane Asphaltenes, ppm	345	600
Ramsbottom Cabon, %	0.15	0.26

FIGURE 1

PROGRAM TIMING ESTIMATE FOR
AN INDIVIDUAL FEEDSTOCK



Note: HDF is Hydrofiner; HCR is Hydrocracker

FIGURE 2

REVISED PROGRAM TIMING ESTIMATE
SHALE OIL PROCESSING
ERDA CONTRACT EF-76-C-01-2315

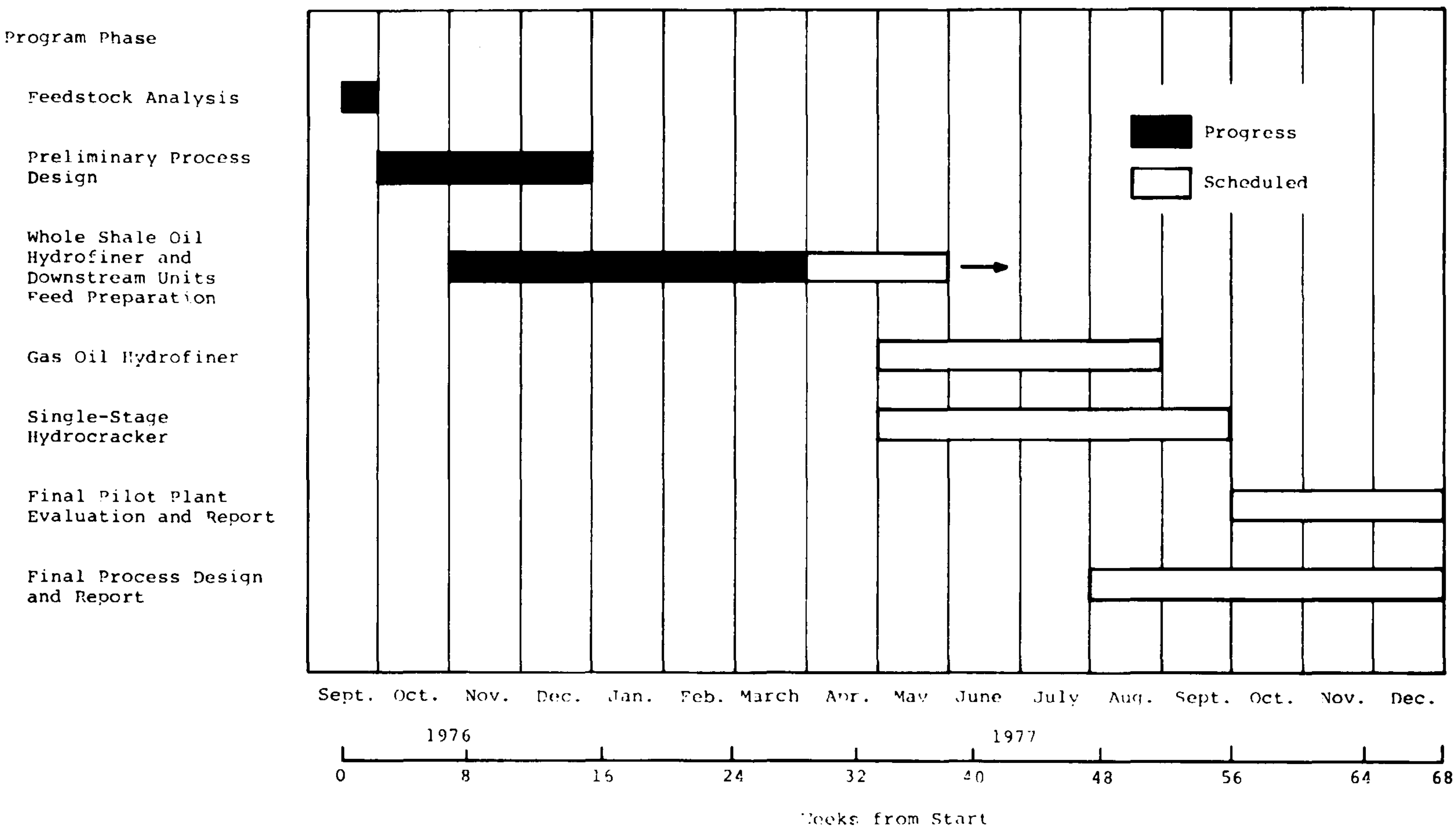


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FIGURE 3

PILOT PLANT RUN 81–4 – PART I
ERDA CONTRACT EF–76–C–01–2315
WHOLE SHALE OIL HYDROFINING WITH ICR 106 CATALYST

2200 PSIG (~1850 PSIA H_2) – 0.6 LHSV
745°F AVERAGE CATALYST TEMPERATURE

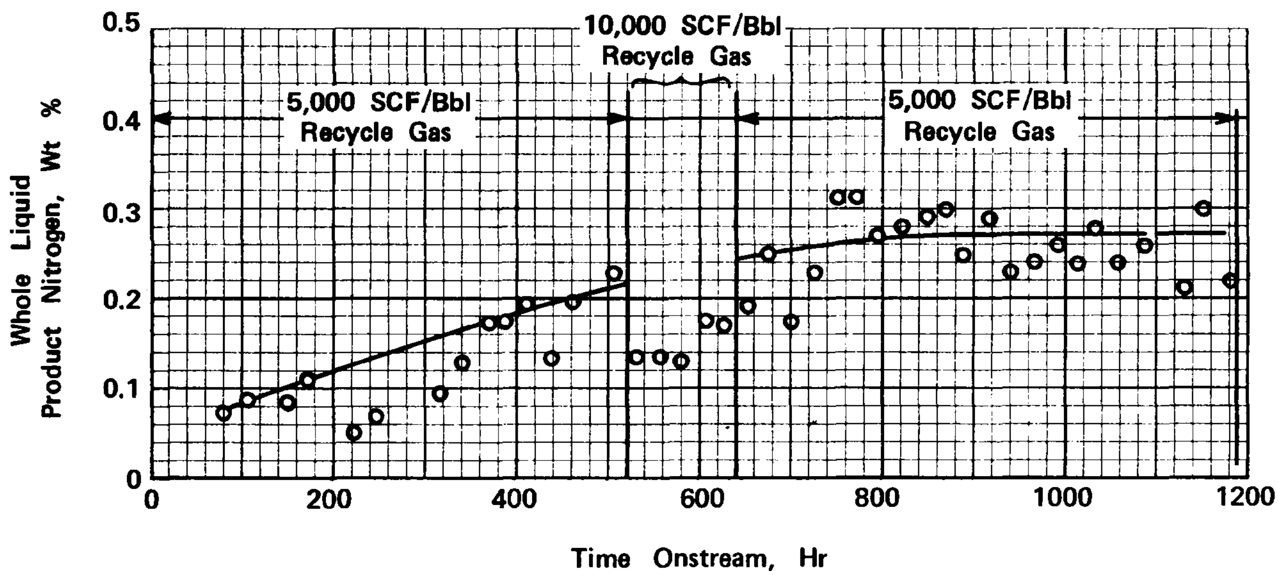

FIGURE 4

PILOT PLANT RUN 81–4 – PART II
ERDA CONTRACT EF–76–C–01–2315
WHOLE SHALE OIL HYDROFINING WITH ICR 106 CATALYST
767°F AVERAGE CATALYST TEMPERATURE
2200 PSIG (~1850 PSIA H_2 → ~1600 PSIA H_2)
5000 SCF/BBL RECYCLE GAS RATE
130 ML CRUSHED ICR 106 CATALYST (10–16 MESH)

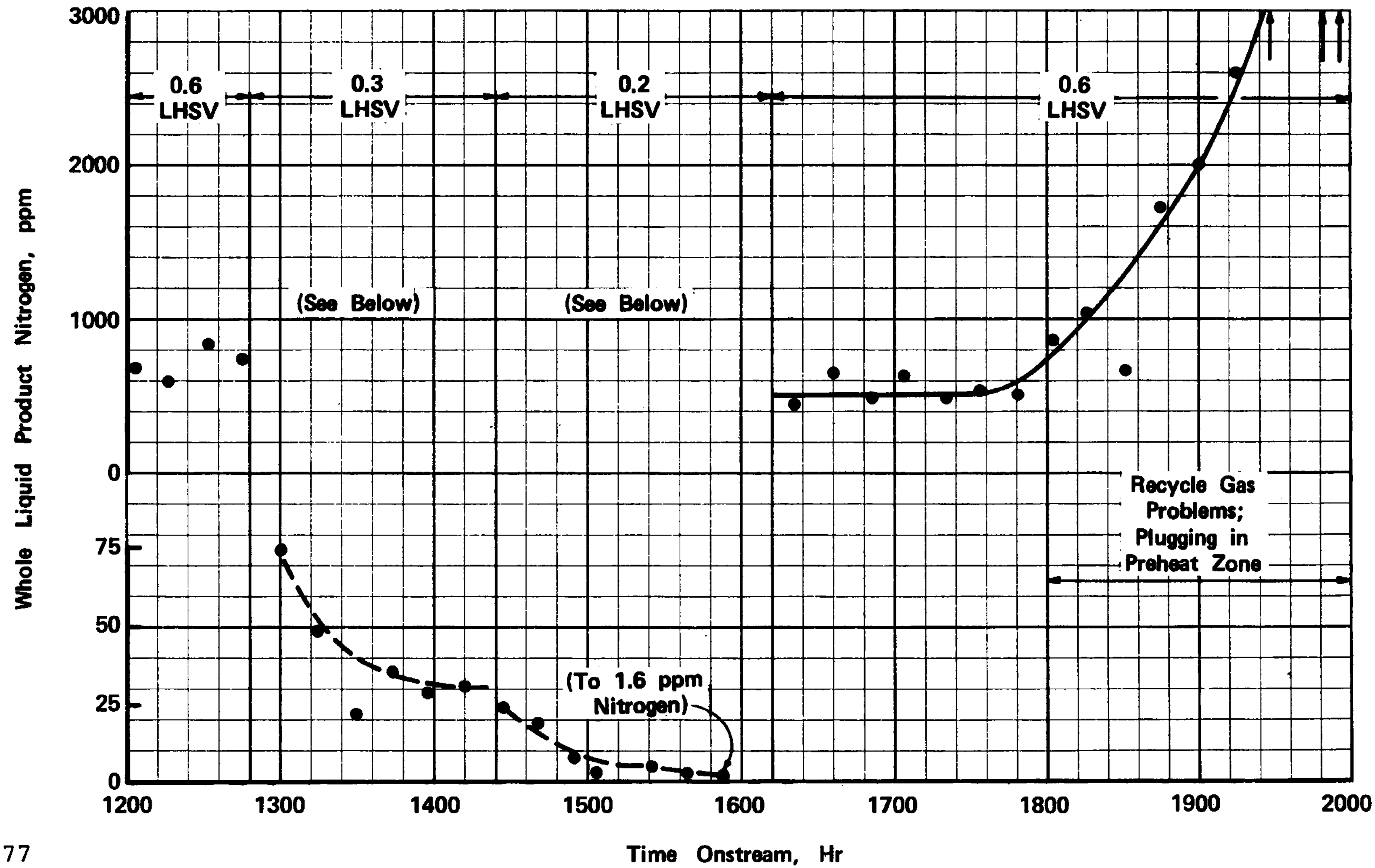

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FIGURE 5

PILOT PLANT RUN 81–4 – ERDA CONTRACT EF–76–C–01–2315
TBP DISTILLATIONS (SIMULATED BY CHROMATOGRAPHY) OF
WHOLE SHALE OIL AND WHOLE LIQUID PRODUCTS WITH ICR 106 CATALYST

767°F AVERAGE CATALYST TEMPERATURE

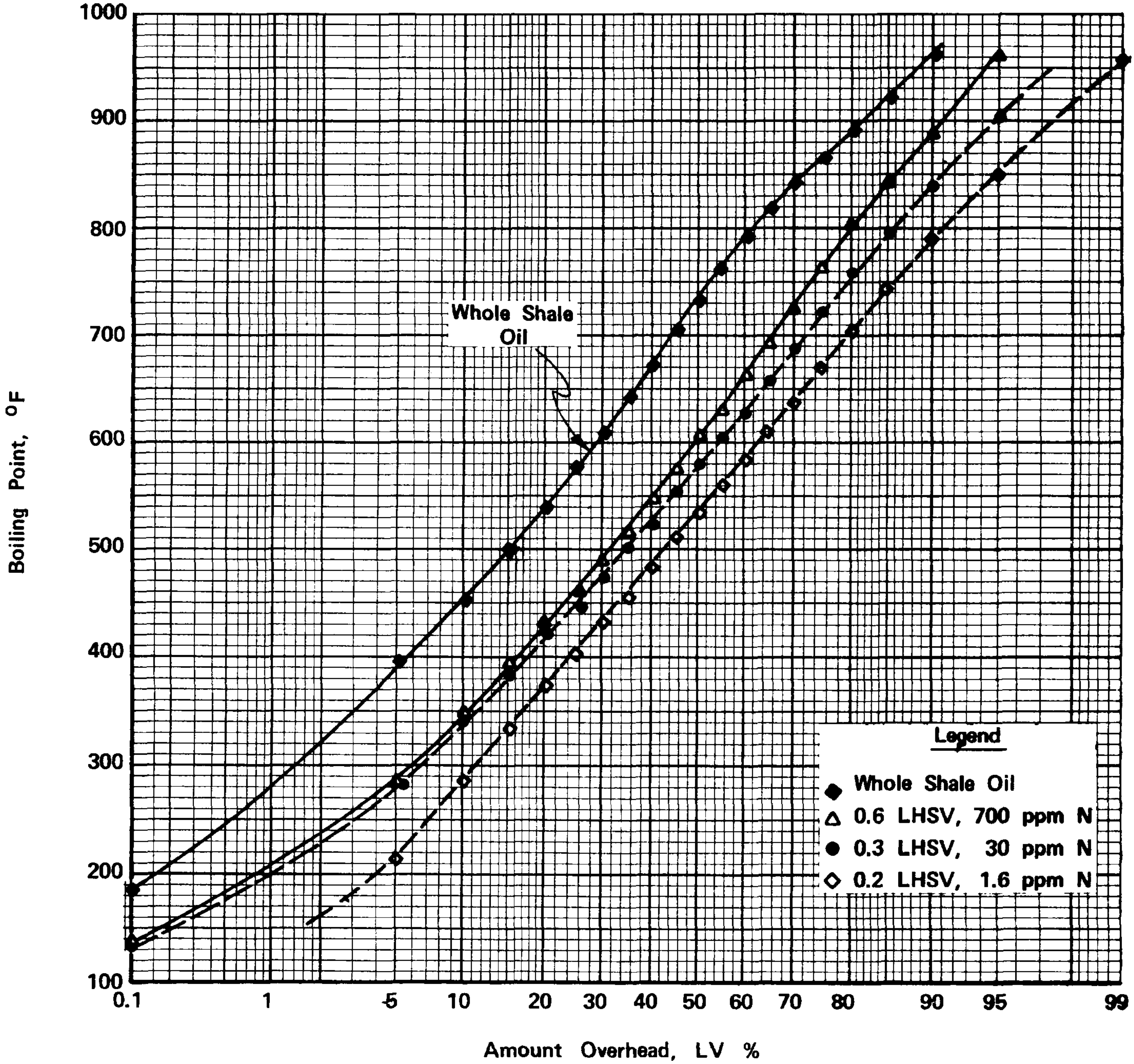

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FIGURE 6

PILOT PLANT RUN 86–50
ERDA CONTRACT EF–76–C–01–2315
WHOLE SHALE OIL HYDROFINING WITH ICR 106L CATALYST

2200 PSIG (~1850 PSIA H_2 → ~1600 PSIA H_2) – 0.6 LHSV
5000 SCF/BBL RECYCLE GAS RATE

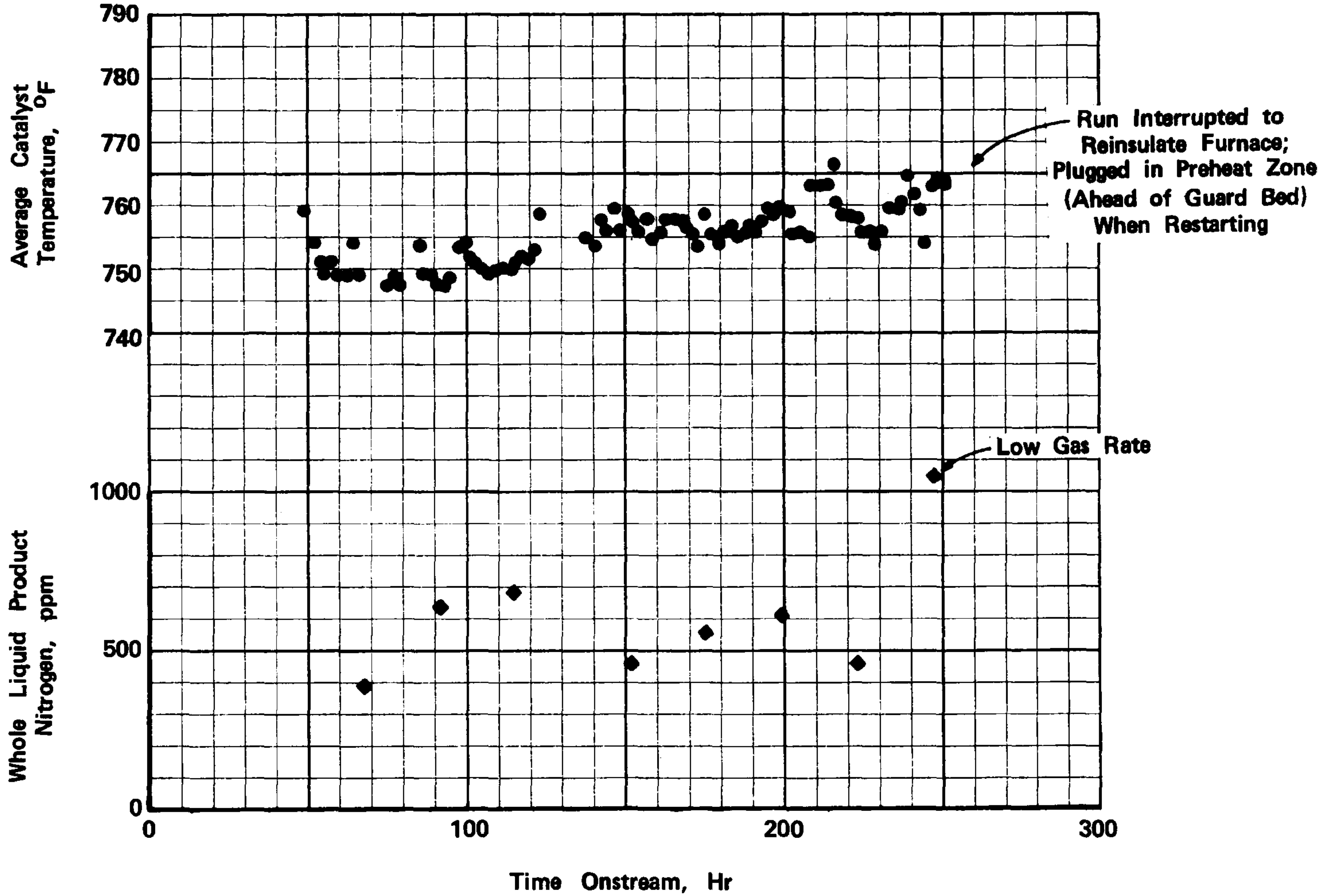

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FIGURE 7

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

CATALYST TEMPERATURE PROFILE AFTER 500 HR
AVERAGE CATALYST TEMPERATURE ~762°F

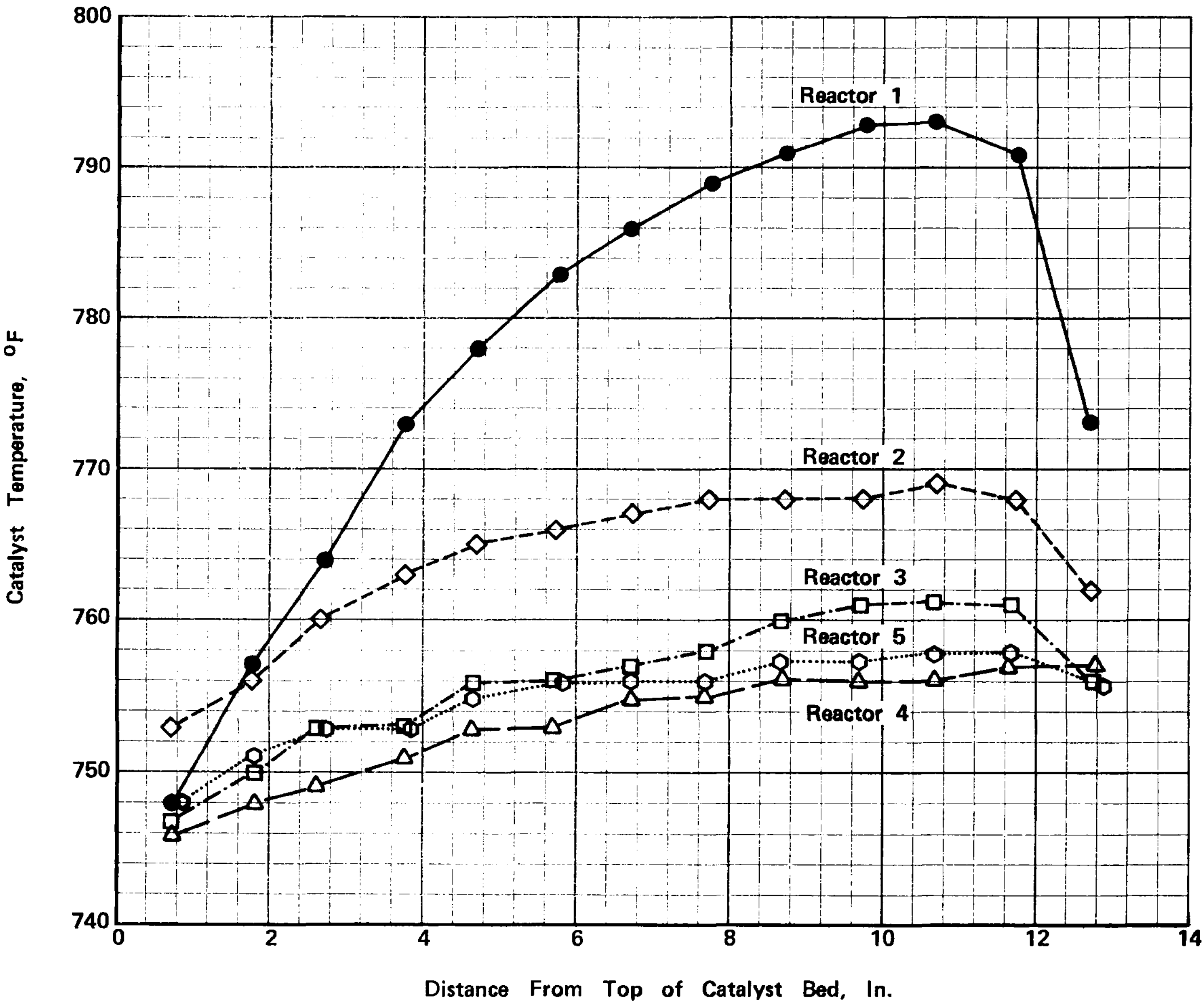


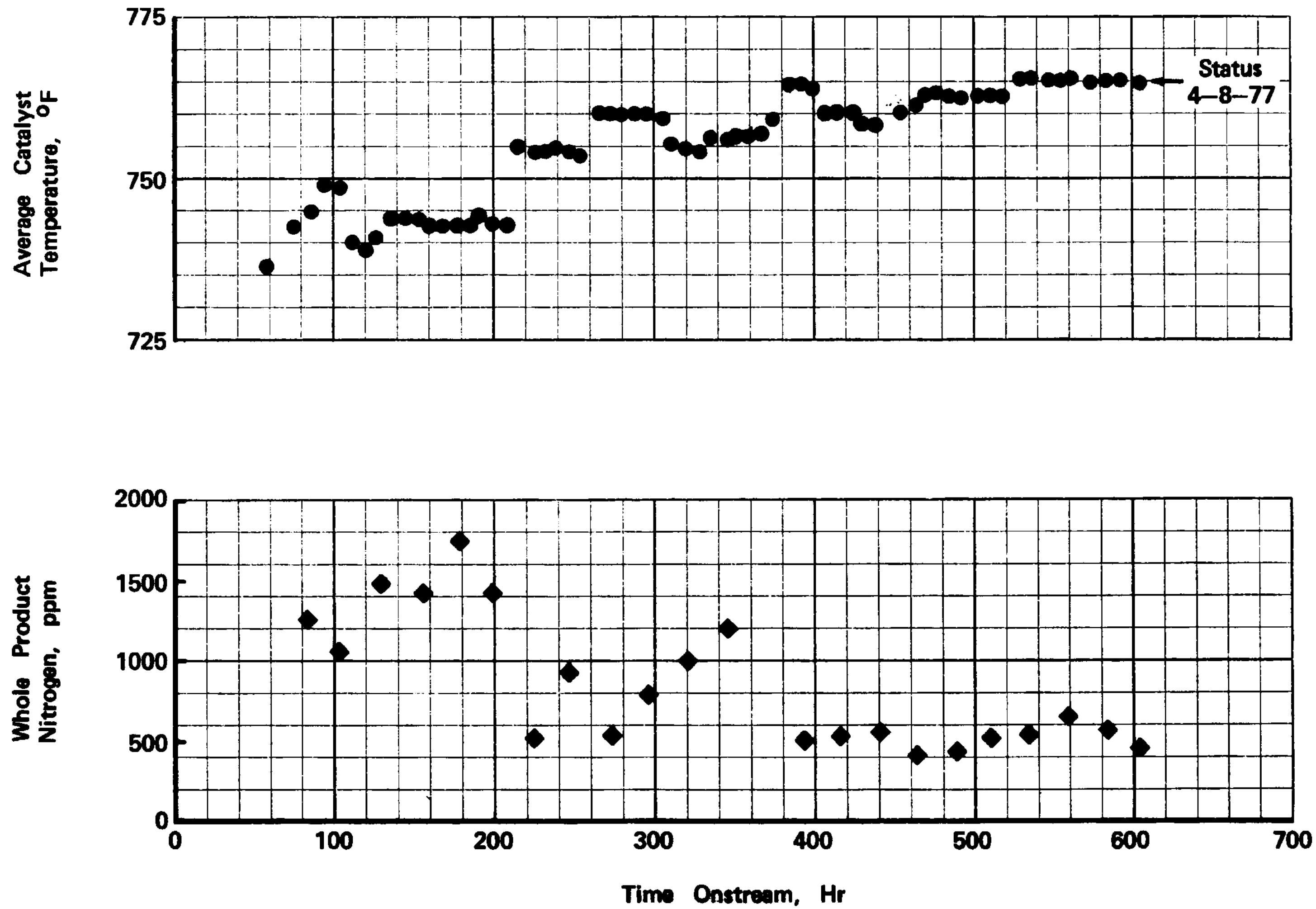
FIGURE 8

PILOT PLANT RUN 86–51

ERDA CONTRACT EF–76–C–01–2315

WHOLE SHALE OIL HYDROFINING WITH ICR 106L

2350 PSIG (~1775 PSIA H_2) – 0.6 LHSV – 8000 SCF/BBL RECYCLE GAS RATE



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