FE 2315-16

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

Monthly Report for the
Period July 1977

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PREPARED FOR THE UNITED STATES
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 Paraho crude shale oil, produced in the indirectly heated mode. The second feedstock is solvent refined coal (SRC), obtained from the Pittsburg and Midway Coal Company pilot plant near Tacoma, Washington. Work on a third feedstock is expected to begin in October of this year.

Encl. - See Index of Enclosures

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/bbl; (d) total inlet pressure is 2350 psig; (e) hydrogen partial pressure has varied between 1200 psia and 2100 psia. The higher hydrogen partial pressures were obtained by use of a recycle gas bleed. The lower hydrogen partial pressures were obtained during periods without recycle gas bleed and with a large pressure drop across the guard bed.

Details of Run 86-51 to 2550 hours on stream were discussed in detail in the April-June Quarterly Report (FE 2315-15). Figure 1 is a run summary from that report that has been extended to show additional results obtained this month.

Normalized catalyst temperature for the whole liquid product nitrogen of 500 ppm remained at 775-780°F until about 3100 hours on stream. The overall catalyst activity at about 2000 psia has remained essentially constant (within the limits of the measurement) for almost 2000 hours. This result is very encouraging and shows that ICR 106 catalyst is extremely stable at these conditions.

Figure 2 shows the temperature profile at 3039 hours. It is quite similar to that observed at 2127 hours. (See Figure 16 of the April-June 1977 Quarterly Report for the temperature profile at 2127 hours and page 8 of that report for a discussion of the relationship between temperature profile and catalyst activity.) The result supports the conclusion that the overall catalyst activity has remained essentially constant between the times these two profiles were taken. (Close examination of the temperature profiles shows that the first catalyst bed has lost some additional activity between the times when the two profiles were taken. Only a relatively small percentage of the overall activity is accounted for by the first catalyst bed at this time; therefore, the total catalyst activity is not appreciably changed by the additional first bed deactivation.

At about 3000 hours on stream, a pressure drop of about 200 psia was noted across the guard bed. At 3200 hours, this pressure drop had increased to 400 psia; and at 3250 hours it had reached 600 psia. As shown previously, a lowering of the hydrogen partial pressure causes an apparent decrease in catalyst activity. This is shown again in Figure 1. During the period between 3100 hours on stream and 3250 hours on stream, the apparent catalyst decreased by about 15°F.

At 3260 hours on stream, the guard bed was removed and is being replaced.

The run will be restarted with a fresh guard bed. It is expected that the catalyst activity will be the same as it was before the pressure drop developed.

We now have additional analytical information of the composition of the guard bed that was replaced at 1950 hours on stream. Figures 3 and 4 show a profile of the composition of the aged guard bed and a typical temperature profile across the guard bed. In studying these figures, remember that the guard bed is operated in an upflow mode.

The figures show that arsenic and iron are found throughout the guard bed, even the portion with a temperature of 200-300°F. The largest amount was deposited in the part of the bed with temperatures ranging from 400-700°F. Plugging occurred when the iron content was 15 wt % and the arsenic content was 7 wt %. The result suggests that if a more gradual temperature is imposed upon the guard bed, more efficient use of the guard bed can be obtained. With more uniform distribution of these metals, a longer period of operation is expected before it is necessary to replace the guard bed.

The shale oil feed contains 70 ppm of iron and 28 ppm of arsenic for an iron/arsenic ratio of 2.5/1. The profile shows that the iron comes out more easily than arsenic. At the bottom of the bed (inlet) which operates at about 200°F, the iron/arsenic ratio is 6/1. At the top of the bed (outlet), which operates at over 750°F, the iron/arsenic ratio is slightly less than one.

Assuming the analyses are correct, virtually all of the arsenic from the feed can be accounted for in the guard bed; 70% of the iron from the feed is accounted for by the iron found in the guard bed.

Figure 4 shows the guard bed is also effective for removing zinc from the feed. Also shown in Figure 4 is the carbon content of the aged guard bed.

The Paraho shale oil also contains a trace of selenium that is removed by the guard bed. The maximum selenium content of the aged guard bed is 0.12%, which occurs about one-third of the way through the bed as shown in Figure 5. Figure 5 also shows the sulfur distribution for the aged guard bed.

Task 2 Alternate--Alternate to Whole Oil Hydrotreating--Coking of Whole Shale Oil; Hydrofinishing of Coker Distillate

(Note: This task is described in the last paragraph of Task 2 of the work statement and is included with the permission of the ERDA Technical Representative.)

A possible alternative processing route for shale oil is the coking of the whole shale oil followed by hydrofining of the coker distillate rather than hydrofining the whole shale oil. In order to evaluate this processing route as compared to whole oil hydrofining, we coked two drums of Paraho shale oil and are starting a pilot plant run (81-10) to evaluate hydrofining of the distillate using ICR 106 catalyst.

Table I gives inspections of the shale oil coker distillate produced. Details and yields from the coking run will be presented in a future report.

Task 4--Extinction Recycle Hydrocracking of 650°F+ Hydrofined Shale Oil

The 650°F+ fraction of the hydrofined shale oil (SGQ-6152, Table IV, April-June Quarterly Report) was hydrocracked to extinction in a single stage in pilot plant catalyst screening runs, 81-8 and 81-9. In the first of these runs, 130 ml of ICR 106 was used as the catalyst; in the second run, 130 ml of ICR 117 was the catalyst.

Target conditions were as follows: 0.6 LHSV, 2300 psig total, ∿2000 psia H_2, 5000 SCF/bbl recycle gas rate, ∿550°F recycle cut point. Lined out per-pass conversion was 40% for ICR 106 at 795°F and about 55% for ICR 117 at 783°F. ICR 117 is more active but gives lower liquid yields than does ICR 106. Average catalyst temperature is somewhat high for optimum yields with the latter catalyst.

Preliminary yield data for these runs are given in Tables II and III. Product inspections are incomplete.

The catalysts are less active than expected, based on correlation with petroleum stocks. It is likely that the small amount of high boiling material (900°F+) is quite refractory and is not converted readily. It may be desirable to remove this material by distillation and use it as refinery fuel rather than crack the entire 650°F+ hydrofined shale oil. Pilot plant runs will be made to determine whether this is true.

Program

During the month of August, the following work is scheduled:

1. Task 2: Continuation of Run 86-51, whole shale oil hydrofining. This run will be resumed using a new guard bed. The run will be continued long enough to determine whether any irreversible catalyst fouling occurred as a result of the recent lower pressure operation and guard bed changeout.

2. Task 2 Alternate: Startup of Run 81-10, hydrofining of shale oil coker distillate. Purpose of this run is to compare coking followed by coker distillate hydrofining to hydrofining of whole shale oil as routes for processing.

3. Task 3: Startup of Run 66-185, hydrofining of the diesel fraction produced by whole shale oil hydrofining. Our initial tests on this diesel fraction indicated that it may be unstable at 500 ppm nitrogen and requires some additional hydrotreating. (Note: The diesel tested was prepared in a batch distillation. Results from testing of petroleum-derived diesels indicate that overheating during batch distillations may cause apparent instability problems even though similar diesel fractions distilled in continuous commercial columns are stable. Therefore, we are also setting up a continuous laboratory distillation of the hydrofined shale oil to obtain a diesel fraction that more closely resembles that distilled commercially. It may be that additional hydrofining is not required.)

4. Task 4: Extinction recycle hydrocracking. We will complete the evaluation of results of the two hydrocracking runs made this month to determine what additional runs are necessary.

5. Task 8: Preparation of 1-1.5 gallons of hydrofined shale oil distillate from two types of feed and at three levels of severity. This task was added by ERDA in Modification No. A004 at the request of Professor V. F. Yesavage of the Colorado

School of Mines and includes hydrofining of distillate fractions from both in situ and ex situ retorting. We expect to complete this task during August.

III. SRC Processing

Task 1

HDF Feed Preparation

The 50/50 SRC/creosote oil feed blend (WOW 3476) has been filtered with Balston 25μ and 2μ filters, respectively. It took three days to filter a barrel of WOW 3476 with the 2μ filter. Tables IV and V show the inspections and metal content of these blends before and after filtration. Analysis shows the filtration reduced the ash from 0.11% to 0.07%. At best, only a small amount of ash was removed; and because of the difficulty in filtration, we do not regard this as a promising pretreatment step.

Task 2

Hydrofining Tests

The first pilot plant test run, 87-67, was shut down at 330 hours due to plugging in the third catalyst bed. A new pilot plant test run, 30-27, is in progress. We are using the filtered feed, WOW 3530-1, and a three-in-series reactor system which has separate temperature controllers in each reactor for isothermal conditions through all the catalyst beds.

We are using the same catalyst, ICR 106, as in Run 87-67. All reactors are downflow; the total volume of catalyst charged is 628 cc: 130 cc in the first reactor and 249 cc in each of the second and the third reactors. In the first reactor, 190 cc of low cost 8-14 mesh material is used upstream from the catalyst bed as a guard bed to protect the catalyst from deposits of metals and ash. The process conditions are identical to Run 87-67: 0.2 LHSV, 2500 psig, and 10,000 SCF/bbl recycle H_2.

Run 30-27 was lined out with creosote oil followed by a feed blend of 25/75 SRC/creosote oil before the feed, WOW 3530-1, was introduced. We also have a standby pumping system to pump creosote oil through the reactor when the feed is discontinued due to a unit upset. The feed left in the reactor can be washed out with creosote oil.

Tables VI, VII, VIII, and IX are the yields and product properties for Run 87-67. The average chemical hydrogen consumption is about 2700 SCF/bbl at 750°F. Note, however, the scatter in hydrogen consumption results for the different yield periods. Table X shows the updated summary of analyses for metal content in the whole liquid product from Run 87-67.

Run 30-27 is now 370 hours on stream with 50/50 SRC/creosote oil. It has already exceeded the run length of Run 87-67. Figure 6 shows the total nitrogen content in the whole liquid product as a function of temperature and run hour. The activity is about the same as that in Run 87-67. Figure 7 shows the D 1160 (2 mm) distillation curves of the feed and a product. Table XI shows the inspections of the feed and the products.

Program

We plan to continue Run 30-27 until we obtain sufficient product of an appropriate boiling range to use as solvent for dissolving SRC for further processing studies.

:bh

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TABLE I

ERDA CONTRACT EF-76-C-01-2315
PROPERTIES OF DEWATERED WHOLE PARAHO SHALE OIL AND SHALE OIL COKER DISTILLATE FROM PARAHO SHALE OIL

Type	Whole Shale Oil	Shale Oil Coker Distillate
Chevron ID No.	WOW 3511	WOW 3524
Inspections		
Gravity, °API	20.5	32.7
Aniline Point, °F		102.5
Sulfur, Wt %	0.63	0.61
Total Nitrogen, Wt %	2.15	1.75
Oxygen, Wt %	1.19	0.94
Arsenic, ppm	26.5	6.3
Pour Point, ASTM, °F	85	25
Carbon, Wt %	84.58	85.00
Hydrogen, Wt %	11.42	12.36
Hydrogen/Carbon	1.61	1.73
Chloride, ppm	0.7	1.7
"Soluble" Iron, ppm	68	0.4
Average Molecular Weight	(277)	228
Ramsbottom Carbon, %	1.85	0.17
Group Type, LV % (FIAM)		
Paraffins and Naphthenes		36
Aromatics		41
Olefins		23
Viscosity, cSt		
At 100°F		2.921
At 122°F	25.45	2.128
Acid Neutralization No., mg KOH/g	2.4	1.1
Base Neutralization No., mg KOH/g	36	31
pH	9.1	9.0
Maleic Anhydride No., mg/g	47.2	27
ASTM Distillation, °F	D 1160 (Run at 2 mm)	D 86/D 1160
St/5	404/506	186/295
10/30	544/670	344/467
50	772	542
70/90	866/1010	595/655
EP	/1088	/684
% Overhead (Excl. Trap)	93	95
% in Trap	2.5	5
% in Flask	4.5	0
TBP Distillation (Simulated by Chromatography)		
St/5		51/236
10/30		298/433
50		514
70/90		584/652
95/99		675/723

TABLE II

ERDA CONTRACT EF-76-C-01-2315
SINGLE STAGE EXTINCTION RECYCLE HYDROCRACKING
OF 650 F+ HYDROFINED SHALE OIL WITH ICR 106

RUN	81- 8		81- 8	
FEED	SGQ 6152		SGQ 6152	
RUN HOURS	215.0- 263.0		263.0- 287.0	
AVG.CAT.TEMP., F.	794.		796.	
LHSV	0.60		0.60	
PER PASS CONVERSION	41.35		36.30	
TOTAL PRESSURE,PSIG	2300.		2304.	
H2 MEAN PRESS.,PSIA	2005.		2010.	
TOTAL GAS IN,SCF/B	5599.		5679.	
RECYCLE GAS, SCF/B	4973.		5122.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.
C1	0.38		0.48	
C2	0.57		0.65	
C3	2.11		2.36	
I-C4	2.14	3.34	2.37	3.70
N-C4	2.38	3.58	2.80	4.20
C5-180 F.	10.39	13.91	10.49	14.09
180-300 F.	22.93	27.37	22.42	26.78
300-550 F.	61.24	67.93	60.53	67.14
TOTAL C5+	94.57	109.22	93.45	108.02
ACT./NO LOSS RECOV.	101.68/102.27		101.57/102.22	
H2 CONS(GROSS),SCF/B	1512.		1533.	
H2 CONS(HCSON),SCF/B	1312.		1287.	

TABLE III

ERDA CONTRACT EF-76-C-01-2315

SINGLE STAGE EXTINCTION RECYCLE HYDROCRACKING OF 650 F+ HYDROFINED SHALE OIL WITH ICR 117

RUN	81- 9		81- 9	
FEED	SGQ 6152		SGQ 6152	
RUN HOURS	169.0- 193.0		217.0- 241.0	
AVG.CAT.TEMP., F.	782.		783.	
LHSV	0.63		0.63	
PER PASS CONVERSION	63.63		48.36	
TOTAL PRESSURE,PSIG	2298.		2299.	
H2 MEAN PRESS.,PSIA	1929.		1960.	
TOTAL GAS IN,SCF/B	6058.		5663.	
RECYCLE GAS, SCF/B	4727.		4643.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.
C1	0.30		0.29	
C2	0.54		0.60	
C3	6.96		6.86	
I-C4	10.19	15.90	9.49	14.80
N-C4	7.55	11.34	6.95	10.44
C5-180 F.	29.08	39.00	28.61	38.33
180-300 F.	28.10	33.52	26.46	31.42
300-550 F.	20.51	22.51	23.99	26.04
TOTAL C5+	77.70	95.04	79.07	95.80
ACT./NO LOSS RECOV.	96.66/103.36		100.01/103.38	
H2 CONS(GROSS),SCF/B	2091.		2108.	
H2 CONS(HCSON),SCF/B	1943.		1954.	

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TABLE IV

ERDA CONTRACT EF-76-C-01-2315

INSPECTIONS OF 50/50 SRC/CREOSOTE OIL FEED BLENDS

Sample No.	WOW 3476	WOW 3521[1]	WOW 3530-1[2]
Specific Gravity at 210°F	1.14	1.15	1.15
Ash, Wt %	0.11	0.08	0.07
H, Wt %	5.70	5.99	5.72
C, Wt %	89.94	89.92	89.61
O, Wt %	2.70	2.81	2.57
S, Wt %	0.90	0.77	0.90
Total N, Wt %	1.46	1.46	1.49
Hot Heptane Insolubles, %	52.2		
Benzene Insolubles, %	30.2		
Ramsbottom Carbon, %	29.0	30.1	
Distillation by D 1160 (2 mm)			
Start	483	473	496
5	516	543	566
10	572	605	586
30	659	674	665
50	725	926	902
End Point	1002	990	1048
Recovered, %	61	53	57

[1]WOW 3476 filtered with 25μ Balston filter.
[2]WOW 3476 filtered with 2μ Balston filter.

TABLE V

ERDA CONTRACT EF-76-C-01-2315
METAL CONTENT OF 50/50
SRC/CREOSOTE OIL FEED BLENDS

Sample No.	WOW 3476	WOW 3521[1]	WOW 3530-1[2]
Metals, ppm			
Al	87	44	41
B	8	6	4
Ca	59	47	56
Cr	1	1	1
Fe	163	134	55
Mg	5	2	1
Mn	-	3	4
Si	50	17	8
Ti	154	134	172
V	3	3	2
Zn	-	7	7

[1]WOW 3476 filtered with 25μ Balston filter.
[2]WOW 3476 filtered with 2μ Balston filter.

TABLE VI-A

ERDA CONTRACT EF-76-C-01-2315

HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	67.0- 79.0	
AVG.CAT.TEMP., F.	723.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2506.	
H2 MEAN PRESS.,PSIA	2128.	
TOTAL GAS IN,SCF/B	13454.	
RECYCLE GAS, SCF/B	10437.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.23	
C2	0.33	
C3	0.28	
I-C4	0.02	0.05
N-C4	0.16	0.32
C5-300 F.	1.99	3.07
300-550 F.	23.47	28.22
550-650 F.	25.88	29.04
650- EP F.	45.80	46.20
TOTAL C5+	97.15	106.54
ACT./NO LOSS RECOV.	101.38/103.92	
H2 CONS(GROSS),SCF/B	3017.	
H2 CONS(HCSON),SCF/B	2956.	

....LIQUID PRODUCT INSPECTIONS....

C5-300 F. PRODUCT

GRAVITY, API	49.0
NITROGEN, PPM.	199.00
LOW MASS,LV.PC.	
PARAFFINS	2.5
NAPHTHENES	86.1
AROMATICS	11.4
TBP DIST., F.	
ST/ 5	153./ 178.
10/30	182./ 220.
50	241.
70/90	280./ 349.
95/99	397./ 447.

TABLE VI-B

ERDA CONTRACT EF-76-C-01-2315

HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106

YIELDS AND PRODUCT INSPECTIONS FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	67.0- 79.0
AVG.CAT.TEMP., F.	723.
....LIQUID PRODUCT INSPECTIONS....	
300-550 F. PRODUCT	
GRAVITY, API	17.1
SULFUR, PPM.	10.0
NITROGEN, PPM.	615.00
MOLECULAR WEIGHT	154.
HIGH MASS,LV.PC.	
PARAFFINS	0
NAPHTHENES	37.7
AROMATICS	62.3
VISCOSITY, CS,100F.	2.591
VISCOSITY, CS,210F.	1.046
TBP DIST., F.	
ST/ 5	259./ 353.
10/30	380./ 446.
50	487.
70/90	519./ 551.
95/99	559./ 577.
550-650 F. PRODUCT	
GRAVITY, API	7.2
SULFUR, PPM.	25.0
NITROGEN, PPM.	700.00
MOLECULAR WEIGHT	193.
VISCOSITY, CS,100F.	10.410
VISCOSITY, CS,210F.	4.743
TBP DIST., F.	
ST/ 5	349./ 539.
10/30	550./ 589.
50	609.
70/90	626./ 650.
95/99	658./ 672.
650- EP F. PRODUCT	
GRAVITY, API	-6.8
SULFUR, WT.PC.	0.067
NITROGEN, PPM.	9900.00
MOLECULAR WEIGHT	484.
22 COMP., LV. PC.	
PARAFFINS	0
NAPHTHENES	18.1
AROMATICS	80.2
SULFUR COMPOUNDS	1.6

TABLE VI-C

ERDA CONTRACT EF-76-C-01-2315
HYDROFINING OF 50/50 SRC/CREOSOTE OIL
WITH ICR 106 YIELDS AND PRODUCT INSPECTIONS
FOR 87-67

Run	87-67
Run Hours	67-79
Whole Liquid Product Properties	
Gravity, °API	4.3
S, Wt %	0.04
Total N, Wt %	0.48
Hot C_7 Insolubles, %	11.50
Ramsbottom Carbon, %	11.65
Oxygen, Wt %	0.64
Benzene Insolubles, %	3.12
Viscosity at 100°F, cSt	86.4
Viscosity at 210°F, cSt	7.4
Molecular Weight	263

TABLE VII-A

ERDA CONTRACT EF-76-C-01-2315
HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	87- 67	87- 67
FEED	WOW 3476	WOW 3476	WOW 3476
RUN HOURS	91.0- 103.0	163.0- 175.0	246.0- 258.0
AVG.CAT.TEMP., F.	724.	749.	751.
LHSV	0.18	0.21	0.20
TOTAL PRESSURE,PSIG	2510.	2496.	2505.
H2 MEAN PRESS.,PSIA	2138.	2027.	2006.
TOTAL GAS IN,SCF/B	13808.	12317.	12989.
RECYCLE GAS, SCF/B	10456.	9773.	9848.

NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.
C1	0.26		0.30		0.25	
C2	0.42		0.51		0.39	
C3	0.34		0.48		0.35	
I-C4	0.03	0.05	0.04	0.08	0.03	0.06
N-C4	0.18	0.35	0.31	0.61	0.22	0.42
C5-850 F.	66.72	76.63	67.42	77.03	70.71	81.47
850- EP F.	30.65	29.88	28.46	27.65	26.38	25.68
TOTAL C5+	97.38	106.51	95.88	104.69	97.10	107.15
ACT./NO LOSS RECOV.	105.42/104.37		100.00/103.28		101.55/104.10	
H2 CONS(GROSS),SCF/B	3352.		2544.		3141.	
H2 CONS(HCSON),SCF/B	3292.		2472.		3092.	

....LIQUID PRODUCT INSPECTIONS....

C5-850 F. PRODUCT

GRAVITY, API	9.7	8.9	10.5
SULFUR, PPM.	115.0	110.0	<300.0
NITROGEN, PPM.	1400.00	1400.00	1200.00
H-C7 AS-TENES,WT.PC.	0.017	0.041	0.015
RAMS. CARBON, WT.PC.	0.310	0.319	0.280
OXYGEN, WT.PC.	0.600	0.540	0.459
BENZENE INSOL.,WT PC	<0.03	<0.03	<0.03
VISCOSITY, CS,100F.	7.641	6.515	5.743
VISCOSITY, CS,210F.	1.945	1.755	1.742

850- EP F. PRODUCT

GRAVITY, API	-11.0	-11.4	-11.2
SULFUR, WT.PC.	0.159	0.130	0.110
NITROGEN, PPM.	13400.00		11500.00
H-C7 AS-TENES,WT.PC.	36.900	31.680	29.989
RAMS. CARBON, WT.PC.	42.490	41.810	40.950
OXYGEN, WT.PC.	1.580	1.140	0.919
BENZENE INSOL.,WT PC	10.50	10.80	7.42

TABLE VII-B

ERDA CONTRACT EF-76-C-01-2315
HYDROFINING OF 50/50 SRC/CREOSOTE OIL
WITH ICR 106 YIELDS AND PRODUCT PROPERTIES
FOR 87-67

Run Run Hours	87-67 91-103	87-67 163-175	87-67 246-258
Whole Liquid Product Properties			
Gravity, °API	2.2	3.5	4.5
S, Wt %	0.06	0.05	0.01
Total N, Wt %	0.59	0.49	0.39
Hot C_7 Insolubles, %	13.3	11.1	7.74
Ramsbottom Carbon, %	12.4	11.3	9.93
Oxygen, Wt %	0.89		0.53
Benzene Insolubles, %	2.58	2.53	1.92
Viscosity at 100°F, cSt	134.2	60.8	35.6
Viscosity at 210°F, cSt	8.4	6.1	4.5

TABLE VIII

ERDA CONTRACT EF-76_c_01_2315

HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106

YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	282.0- 294.0	
AVG.CAT.TEMP., F.	752.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2498.	
H2 MEAN PRESS.,PSIA	1907.	
TOTAL GAS IN,SCF/B	9546.	
RECYCLE GAS, SCF/B	6512.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.35	
C2	0.51	
C3	0.46	
I-C4	0.04	0.07
N-C4	0.30	0.58
C5-643 F.	51.36	60.41
643- EP F.	45.18	48.80
TOTAL C5+	96.54	109.21
ACT./NO LOSS RECOV.	100.82/103.95	
H2 CONS(GROSS),SCF/B	3034.	
H2 CONS(HCSON),SCF/B	2982.	

....LIQUID PRODUCT INSPECTIONS....

C5-643 F. PRODUCT	
GRAVITY, API	13.1
SULFUR, PPM.	65.0
NITROGEN, PPM.	695.00
HYDROGEN, WT. PCT.	10.12
CARBON, WT. PCT.	89.88
OXYGEN, WT.PC.	0.479
HIGH MASS,LV.PC.	
PARAFFINS	9.0
NAPHTHENES	26.7
AROMATICS	64.3
VISCOSITY, CS,100F.	3.804
VISCOSITY, CS,210F.	1.315

643- EP F. PRODUCT	
GRAVITY, API	2.0
SULFUR, WT.PC.	0.180
NITROGEN, PPM.	7800.00
HYDROGEN, WT. PCT.	7.77
CARBON, WT. PCT.	90.88
OXYGEN, WT.PC.	0.670
22 COMP., LV. PC.	
PARAFFINS	0
NAPHTHENES	4.1
AROMATICS	93.3
SULFUR COMPOUNDS	2.6
VISCOSITY, CS,210F.	577.700

TABLE IX-A

ERDA CONTRACT EF-76-C-01-2315
HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	294.0- 306.0	
AVG.CAT.TEMP., F.	750.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2498.	
H2 MEAN PRESS.,PSIA	1929.	
TOTAL GAS IN,SCF/B	12579.	
RECYCLE GAS, SCF/B	10212.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.33	
C2	0.55	
C3	0.50	
I-C4	0.04	0.08
N-C4	0.30	0.58
C5-300 F.	2.18	3.32
300-550 F.	24.46	29.27
550-650 F.	26.42	29.48
650-850 F.	16.07	17.26
850- EP F.	26.42	30.25
TOTAL C5+	95.59	109.60
ACT./NO LOSS RECOV.	104.23/103.06	
H2 CONS(GROSS),SCF/B	2367.	
H2 CONS(HCSON),SCF/B	2310.	

....LIQUID PRODUCT INSPECTIONS....

C5-300 F. PRODUCT

GRAVITY, API	47.6
SULFUR, PPM.	5.1
NITROGEN, PPM.	192.00
MOLECULAR WEIGHT	104.
LOW MASS,LV.PC.	
PARAFFINS	7.5
NAPHTHENES	78.9
AROMATICS	13.7
VISCOSITY, CS,100F.	2.283
VISCOSITY, CS,210F.	0.430
TBP DIST., F.	
ST/ 5	56./ 173.
10/30	180./ 194.
50	225.
70/90	269./ 312.
95/99	330./ 358.

TABLE IX-B

ERDA CONTRACT EF-76-C-01-2315

HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106

YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	294.0- 306.0
AVG.CAT.TEMP., F.	750.
LHSV	0.19

....LIQUID PRODUCT INSPECTIONS....

300-550 F. PRODUCT	
GRAVITY, API	16.4
SULFUR, PPM.	2.2
NITROGEN, PPM.	198.00
MOLECULAR WEIGHT	162.
HIGH MASS,LV.PC.	
PARAFFINS	11.9
NAPHTHENES	24.9
AROMATICS	63.2
VISCOSITY, CS,100F.	2.462
VISCOSITY, CS,210F.	1.015
TBP DIST., F.	
ST/ 5	263./ 360.
10/30	397./ 449.
50	483.
70/90	515./ 543.
95/99	550./ 560.

550-650 F. PRODUCT	
GRAVITY, API	6.4
SULFUR, PPM.	24.0
NITROGEN, PPM.	985.00
MOLECULAR WEIGHT	197.
22 COMP., LV. PC.	
PARAFFINS	0.8
NAPHTHENES	48.9
AROMATICS	50.3
SULFUR COMPOUNDS	0.0
VISCOSITY, CS,100F.	9.991
VISCOSITY, CS,210F.	2.195
TBP DIST., F.	
ST/ 5	510./ 543.
10/30	558./ 590.
50	605.
70/90	620./ 641.
95/99	649./ 660.

TABLE IX-C

ERDA CONTRACT EF-76-C-01-2315

HYDROFINING OF 50/50 SRC/CREOSOTE OIL WITH ICR 106

YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	294.0- 306.0
AVG.CAT.TEMP., F.	750.
LHSV	0.19

....LIQUID PRODUCT INSPECTIONS....

650-850 F. PRODUCT	
GRAVITY, API	1.2
SULFUR, PPM.	110.0
NITROGEN, PPM.	3200.00
MOLECULAR WEIGHT	241.
H-C7 AS-TENES,WT.PC.	0.046
BENZENE INSOL.,WT PC	0.03
RAMS. CARBON, WT.PC.	0.270
22 COMP., LV. PC.	
PARAFFINS	0.2
NAPHTHENES	4.9
AROMATICS	91.8
SULFUR COMPOUNDS	3.1
VISCOSITY, CS,100F.	128.600
VISCOSITY, CS,210F.	6.902
TBP DIST., F.	
ST/ 5	543./ 638.
10/30	650./ 678.
50	700.
70/90	744./ 808.
95/99	828./ 862.

850- EP F. PRODUCT	
GRAVITY, API	-9.8
SULFUR, WT.PC.	0.150
NITROGEN, PPM.	13000.00
H-C7 AS-TENES,WT.PC.	52.600
BENZENE INSOL.,WT PC	9.66
RAMS. CARBON, WT.PC.	42.540

WHOLE LIQUID PRODUCT	
GRAVITY, API	3.6
SULFUR, WT.PC.	0.045
NITROGEN, PPM.	4300.00
H-C7 AS-TENES,WT.PC.	8.010
RAMS. CARBON, WT.PC.	10.500
OXYGEN, WT.PC.	0.570
VISCOSITY, CS,100F.	41.600
VISCOSITY, CS,210F.	5.200

TABLE X

ERDA CONTRACT EF-76-C-01-2315
METALS IN THE WHOLE LIQUID PRODUCT
FROM RUN 87-67

Run Hour	79	151	234
Average Cat. Temp., °F	719	746	725
Average LHSV	0.18	0.20	0.22
Metals, ppm			
Al	7.4	18.7	18.6
B	0.3	0.7	2.3
Ca	5.3	22.1	18.2
Cr	0.1	0.1	0.2
Fe	0.3	1.2	11.4
Mg	0.2	0.4	0.6
Ni	-	0.2	0.9
Si	0.6	1.5	4.7
Ti	27.2	>85	67.3
V	0.3	0.6	0.8
Zn	0.8	-	-

TABLE XI

ERDA CONTRACT EF-76-C-01-2315
HYDROPROCESSING SRC AT 0.2 LHSV, 2000 psig,
AND 10,000 SCF/bbl RECYCLE H_2 WITH ICR 106

	Run 30-27			
	Feed SRC/Creosote 50/50	Product	Product	Product
Sample No.	WOW 3530-1			
Hours On Stream		345	369	417
Hours with Feed		41	65	113
Average Cat. Temp., °F		750	749	765
Average LHSV		0.20	0.20	0.19
Specific Gravity	1.15			
Gravity, °API		5.5	4.7	5.0
Wt %				
H	5.72	9.07	8.92	
C	89.61	89.95	89.84	
O	2.57	0.43		
S	0.90	<0.03	0.04	
Total N	1.49	0.29	0.25	0.33
Ash	0.07	0.02		
Viscosity, cSt				
At 100°F		32.53	22.52	
At 210°F		4.18	8.27	
Hot Heptane Insolubles, %	40.9	5.99	7.10	
Ramsbottom Carbon, %		8.82	9.29	
Benzene Insolubles, %		0.85	1.32	

FIGURE 1

ERDA CONTRACT EF–76–C–01–2315
PILOT PLANT RUN 86–51
WHOLE SHALE OIL HYDROFINING WITH ICR 106L CATALYST
8000 SCF/BBL RECYCLE GAS RATE

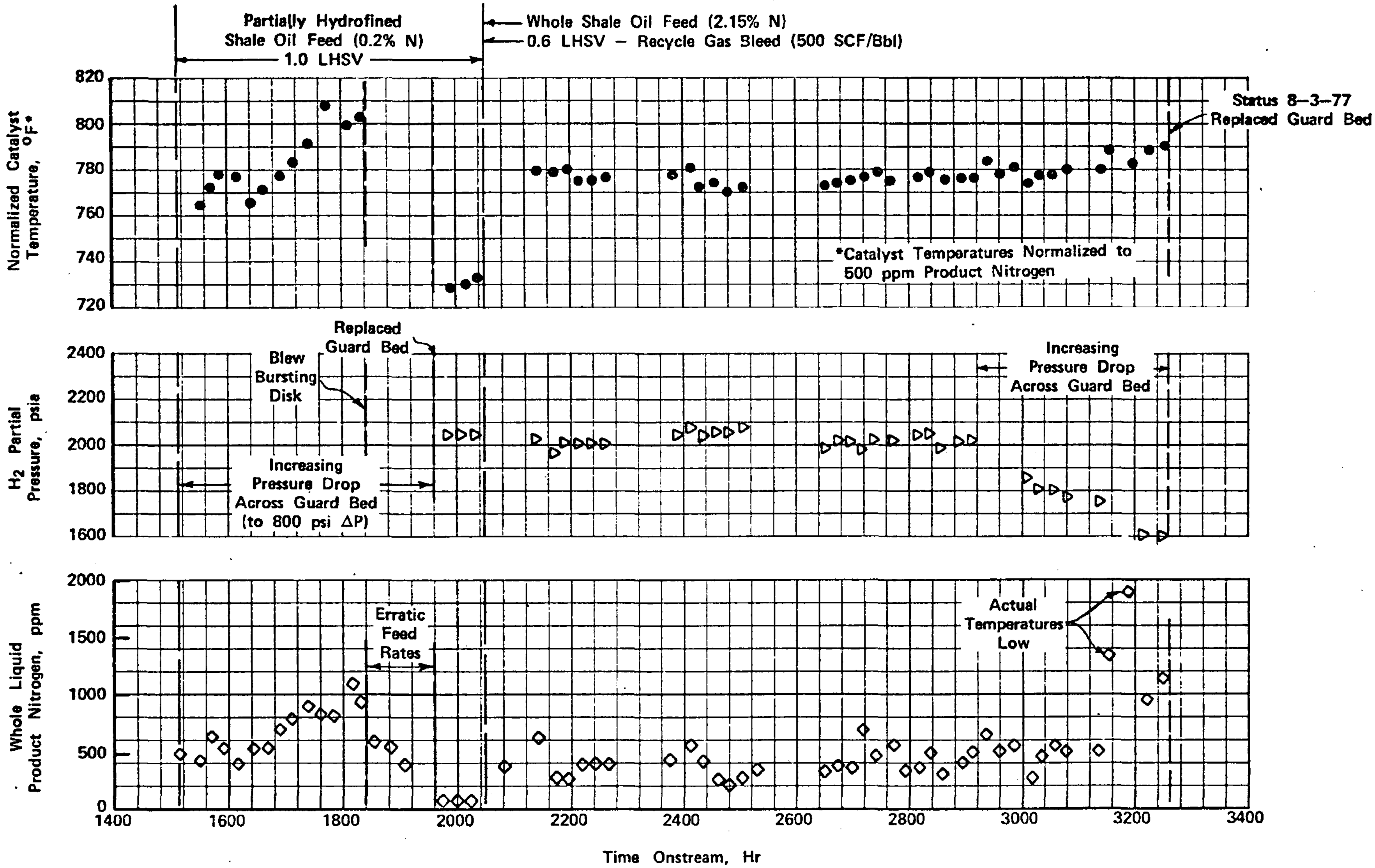

FIGURE 2

ERDA CONTRACT EF-76-C-01-2315

PILOT PLANT RUN 86-51

WHOLE SHALE OIL HYDROFINING WITH ICR 106

CATALYST TEMPERATURE PROFILE AT 3039 HR

AVERAGE CATALYST TEMPERATURE ~777°F

~500 PPM NITROGEN IN WHOLE LIQUID PRODUCT

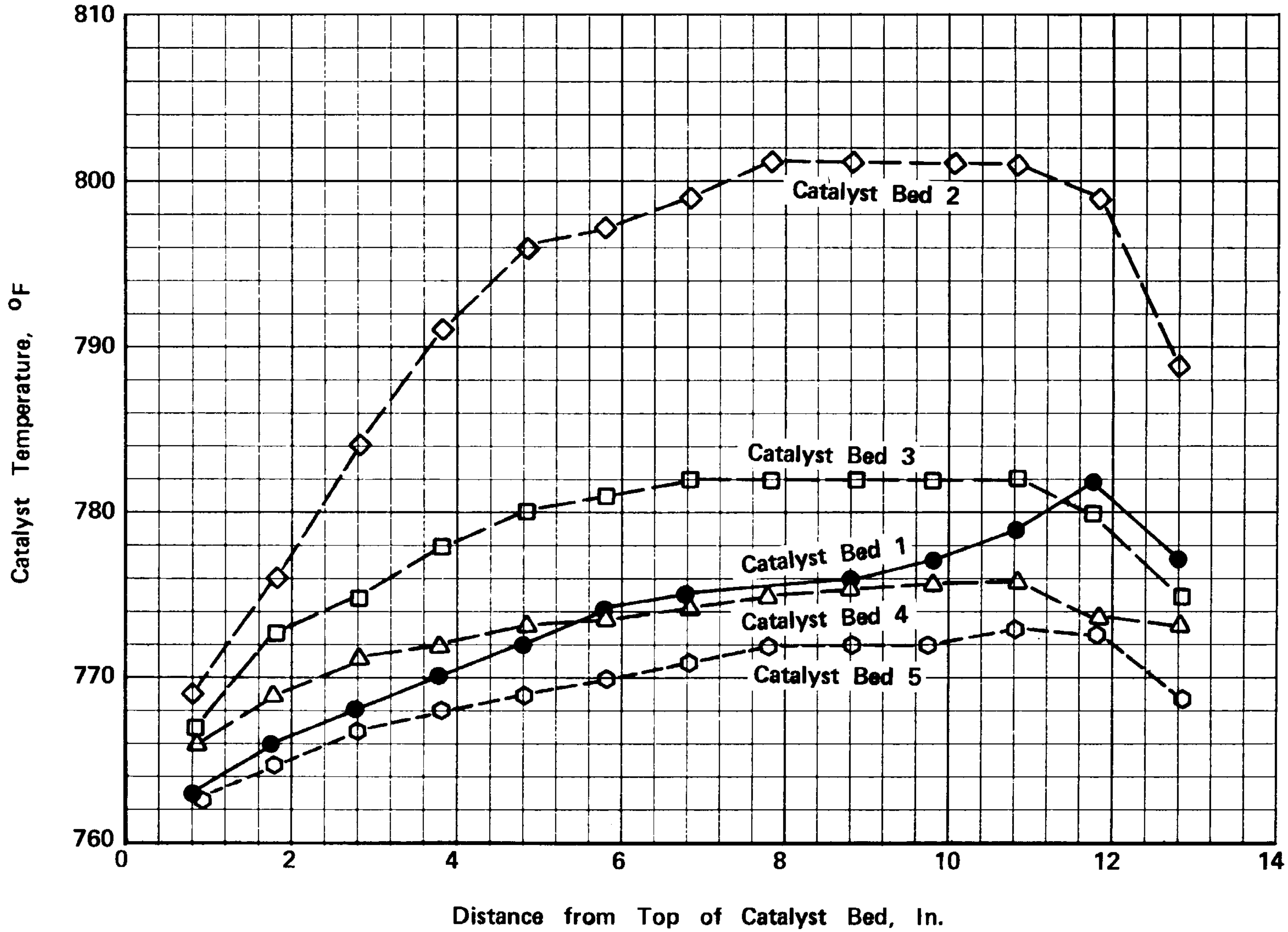

FIGURE 3

ERDA CONTRACT EF-76-C-01-2315

COMPOSITION OF GUARD BED FROM PILOT PLANT RUN 86-51

AFTER 1950 HR ONSTREAM

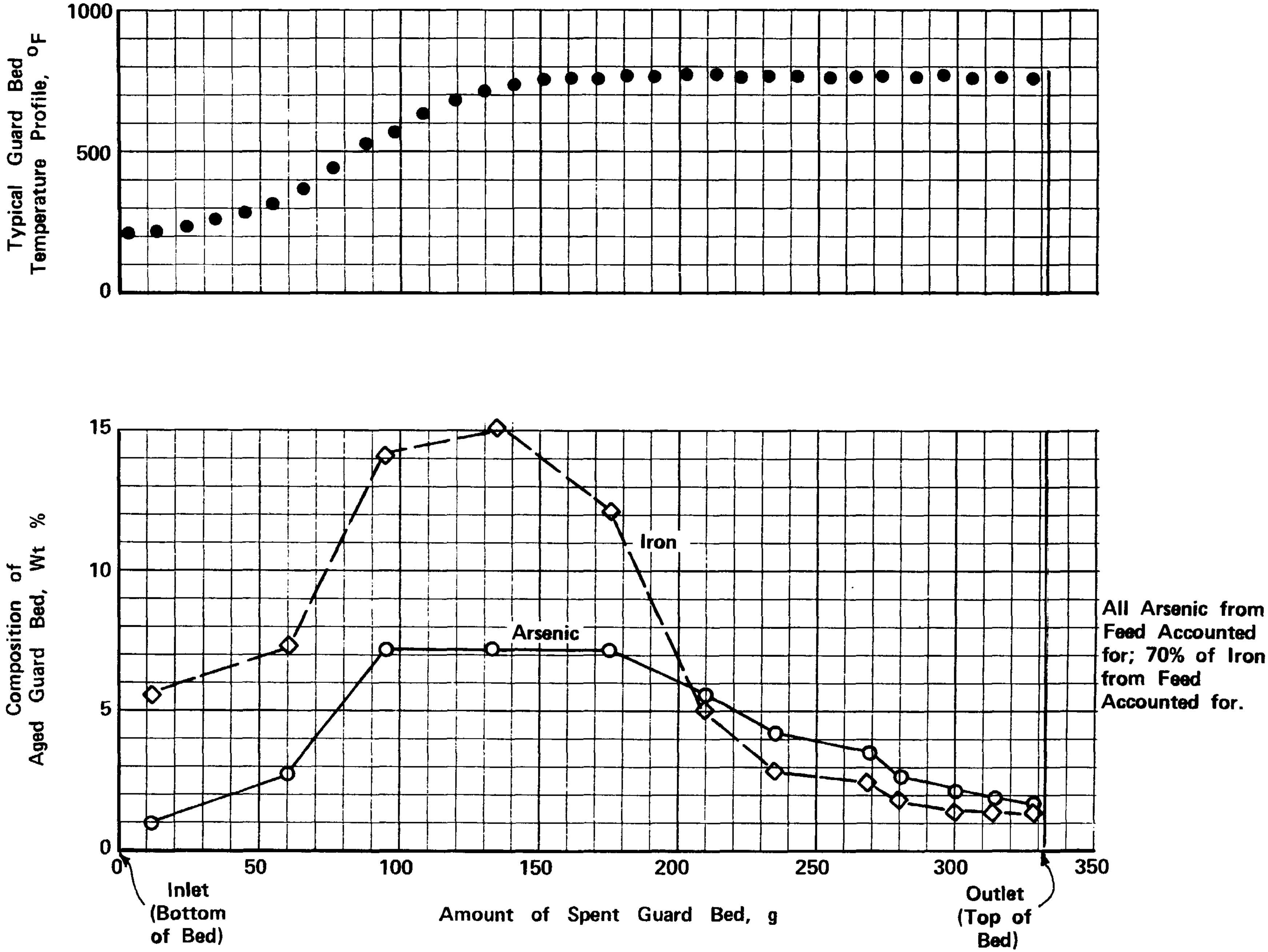

FIGURE 4

ERDA CONTRACT EF–76–C–01–2315
COMPOSITION OF GUARD BED FROM PILOT PLANT RUN 86–51
AFTER 1950 HR ONSTREAM

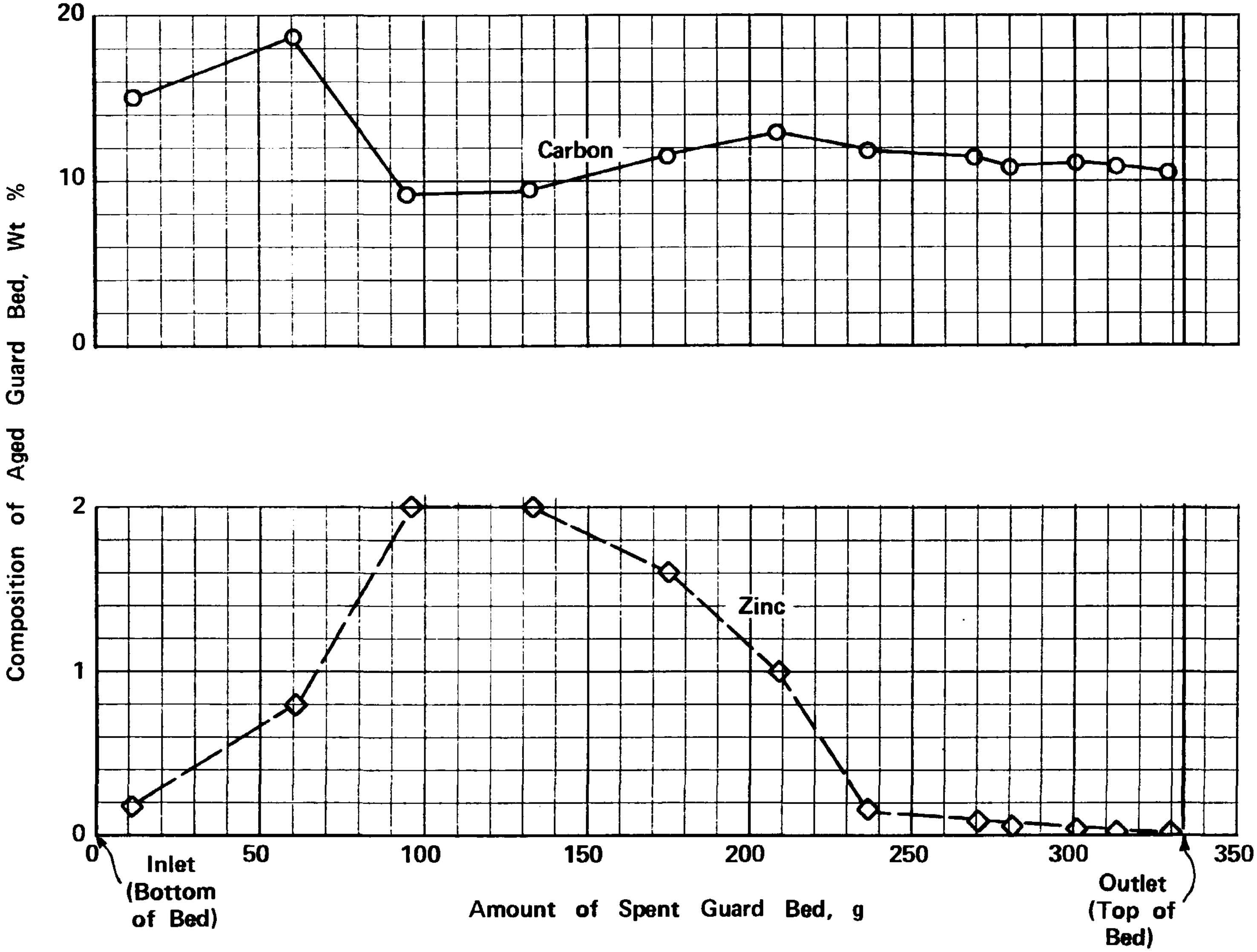

FIGURE 5

ERDA CONTRACT EF–76–C–01–2315
COMPOSITION OF AGED GUARD BED FROM PILOT PLANT RUN 86–51
AFTER 1950 HR ONSTREAM

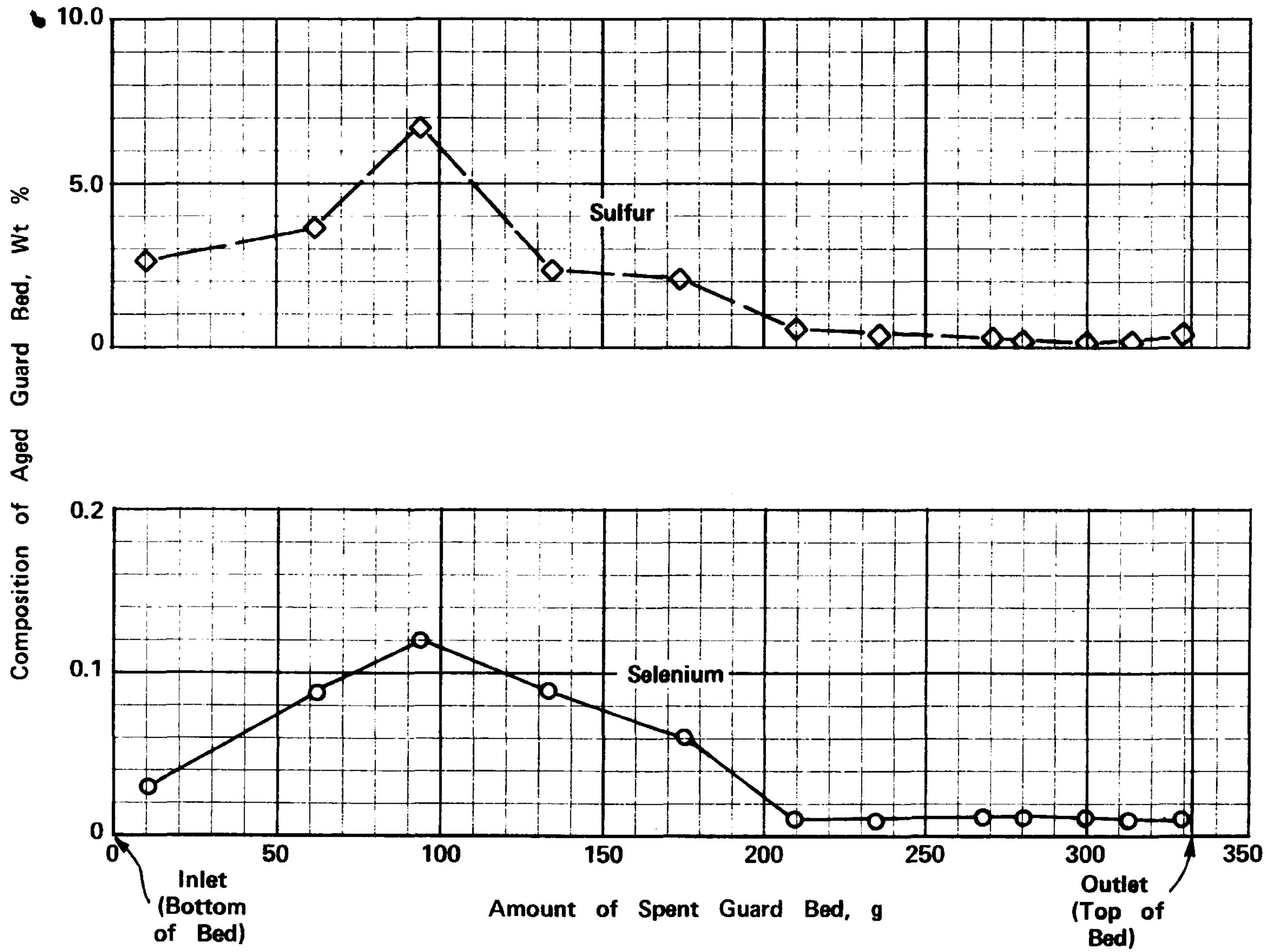

FIGURE 6

ERDA CONTRACT EF-76-C-01-2315
50/50 SRC/CREOSOTE OIL
HYDROPROCESSING WITH ICR 106

RUN 30-27

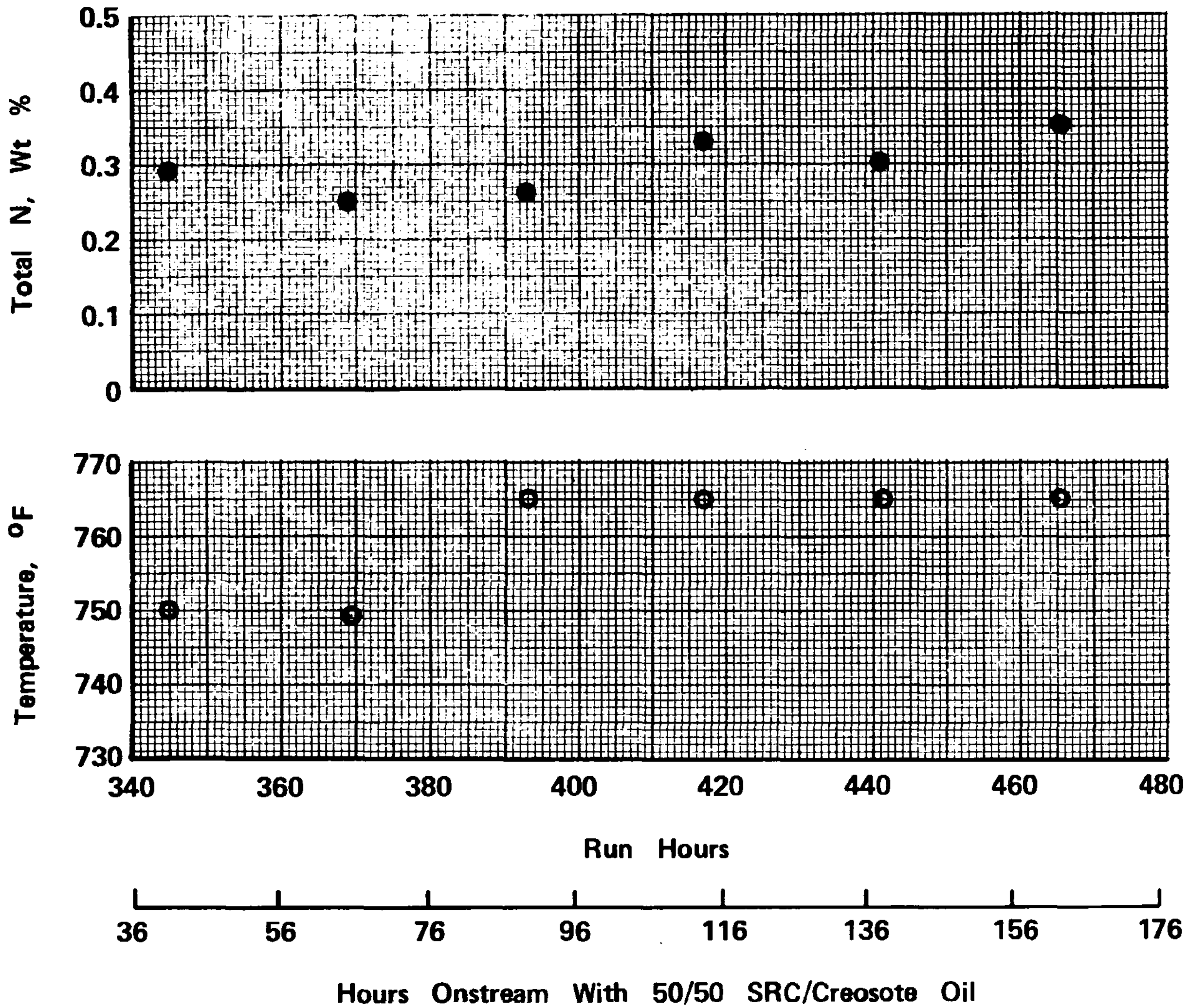

FIGURE 7

ERDA CONTRACT EF–76–C–01–2315
ASTM D 1160 (2 MM) DISTILLATIONS OF
FEED AND PRODUCT OF RUN 30–27

Run Hr	Hr With Feed	Avg. LHSV	Avg. Cat. Temp., °F
O 345	41	0.20	750