

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

Monthly Report for the
Period April 1977

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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 six months. The second feedstock is solvent refined coal (SRC), obtained from the Pittsburgh and Midway Coal Company pilot plant near Tacoma, Washington. Preliminary studies with the SRC started this month.

Encl. - Tables I-IX (RE 772358-RE 772361,
RD 772362, RE 772363, RE 772324-
RE 772326)
Figures 1-4 (RE 772348, RE 772288,
RE 772289, RE 772349)

II. Shale Oil Processing

Task 2--Whole Shale Oil Hydrofining

It was reported last month that 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 hr on-stream, total pressure was 2200 psig; at 210 hr on-stream, the total pressure was increased to 2350 psig to adjust the hydrogen pressure to the target of 1750-1800 psig.

At the time of writing (May 10, 1977), the run has been on-stream for almost 1400 hr. There have been no indications of any plugging problems.

Figure 1 shows the status of the run to date. The following observations are pertinent to the interpretation of the results:

1. The hydrogen pressure decreased as the temperature was increased during the course of the run.
2. Between about 965 and 1200 hr on-stream, a high gas leak caused the hydrogen purity to increase. Apparently as a result of the higher hydrogen pressure, catalyst activity increased. At 2100 psia hydrogen, the catalyst is about 25°F more active than at 1625 psia.
3. After correction of the high gas leak, the hydrogen partial pressure decreased again to 1625 psia, and the activity also decreased by about 25°F.
4. At 1000 hr on-stream (shortly after the high gas leak started), the feed barrel that supplies the unit ran out and we began feeding from a new drum. Because our feed is presumably homogeneous and has been handled with nitrogen blanketing, we assume that the activity improvement is unrelated to the new feed drum but rather the result of the increasing hydrogen partial pressure due to the high gas leak.

5. At 1215 hr on-stream (approximately the same time the high gas leak was corrected), a problem with a temperature controller resulted in a temperature excursion for all the catalyst beds. Temperature in the first catalyst bed reached 915°F; all the other zones reached 850°F. It was noted last month that the temperatures in the first catalyst bed were about 30°F higher than the average catalyst temperature, indicating an exothermic reaction takes place in the first bed (probably largely due to the saturation of olefins). After the temperature excursion, there is less heat release in the first catalyst bed compared to the average than before. Therefore, some deactivation of this bed may have resulted from the excursion. However, the average catalyst does not appear to be affected to any great extent.

The results of this test to date indicate that it may be desirable to maintain a partial bleed of the recycle gas stream in order to increase the hydrogen partial pressure. Presumably, a benefit in both catalyst activity and catalyst fouling rate would be realized as a result of such a bleed. Our Process Design Division is studying these results and may make recommendations for adjustment of operating conditions to take advantage of these observations.

Task 3--Downstream Processing
Studies: Catalytic Cracking of
Hydrofined Paraho Shale Oil--
Preliminary Results

Catalytic cracking is a possible downstream processing step for the refining of the hydrofined shale oil into transportation fuels.

Preliminary small-scale catalytic cracking tests were made on the 650°F+ fractions of two hydrofined Paraho shale oils. The two oils differ in the hydrofining severity, i.e., the extent of denitrification employed in their preparation. Nitrogen contents of the 650°F+ hydrofined shale oil fractions were 385 ppm for the more severely hydrofined and 870 ppm for the less severely hydrofined products used in these tests. Lower boiling material was removed from the hydrofined shale oils by distillation. Table I gives the available inspections for these two oils.

The hydrofined shale oils were cracked in a fixed-fluidized bed test unit using an equilibrium catalyst withdrawn from an operation FCC unit. The quality of the catalyst is shown in Table II. It is a moderately active, moderately metal-contaminated zeolite catalyst.

Catalytic Cracking Conditions

The fixed-fluidized bed test unit is operated in a cyclic manner. Preheated feed enters the bottom of a conical-shaped reactor where it vaporizes and fluidizes the catalyst in the reactor. Steam is added with the feed to aid in its vaporization. Oil is fed for a five-minute period after which a nitrogen purge sweeps the hydrocarbon vapors from the reactor. During the nitrogen purge the temperature of the reactor is raised to 1050°F; and, with completion of the purge, air is introduced into the reactor to burn the accumulated coke from the catalyst. At the end of the regeneration period, a second nitrogen purge removes the air from the reactor thus completing the cracking-regeneration cycle. Multiple cycles are usually made to provide sufficient liquid product for distillation and inspection of the distillate fractions.

The hydrocarbon gas from each cycle is collected in a gas holder, mixed, and sampled. Gas composition is determined chromatographically. The coke deposited on the catalyst is determined for each cycle by passing an aliquot of the regeneration gases over hot cupric oxide to convert carbon monoxide to carbon dioxide and then through an Ascarite absorption bulb.

Nominal reactor conditions for the present study were:

Reactor Temperature, °F	975
Feed Rate, ml/Min.	60
Water (Steam) Rate, ml/Min.	4
Feed Period, Min.	5

Cracking Results

The operating conditions chosen were those used at an earlier date to crack a different "Paraho-type" raw shale oil over the same catalyst and in the same test unit. This permits comparison of crackability and yields between a raw shale oil and hydrofined shale oils. The limited quantities of feeds available permitted only a single six-cycle run to be made with each of the two 650°F+ hydrofined shale oils. Yields and product qualities are discussed below.

Yields

The operating conditions and product yields are presented in Table III. Yields are on a no-loss basis without correction for sulfur and nitrogen compounds in the reaction and regeneration gases. Conversion is defined as 100% minus the percent of 430°F+ liquid product based on feed volume.

The crackability of the hydrofined shale oil is dependent upon its nitrogen content because nitrogen compounds act as temporary catalyst poisons. The 385 ppm nitrogen feed gave 80.1 LV % conversion; whereas, the 870 ppm nitrogen oil gave 75.4 LV % conversion. The raw shale oil cracked earlier at the same conditions was only 38.9% converted. The coke yield is much higher with the raw shale oil (6.21% at 38.9% conversion) than with the hydrofined shale oils (4.41% at 80.9% conversion and 3.87% at 75.4% conversion). Other yield comparisons are not meaningful because of the widely different conversions obtained.

It is interesting to compare yields obtained in cracking hydrofined vacuum gas oils from a typical petroleum source. Two hydrofined gas oils from Arabian Light crude are selected for this comparison¹

1. CCL-4993 27.3°API; 320 ppm Nitrogen
2. CCL-4990 25.3°API; 860 ppm Nitrogen

The nitrogen contents of these two oils are about the same as those of the two hydrofined shale oils of the present study. These oils were cracked at 975°F over the same catalyst (CCL-4914) used in the shale oil study but in a different pilot cracking unit. The pilot plant used to study the cracking of the Arabian gas oils is a continuous catalyst circulation unit. As such, it is a closer approach to commercial FCC unit operation than the cyclic fixed-fluidized bed operation of the present study. The small quantities of hydrofined shale oils available precluded use of the continuous catalyst circulation pilot unit at this time. (However, feed is being prepared for the large-scale tests.) Product workup is similar for both types of units. It has been shown that only minor yield differences are obtained in cracking a given oil in the two units to the same conversion.

¹"A Pilot Plant Comparison of Riser and Dense Bed Cracking of Hydrofined Feedstocks," W. M. Haunschild, D. O. Chessmore, and B. G. Spars. Presented at the 79th AIChE Meeting, Houston, Texas, March 16-20, 1975.

The yields and operating conditions for the cracking runs made on the two hydrofined Arabian gas oils are shown in Table IV. Two runs are shown for each oil. They bracket the conversions obtained with the two hydrogenated shale oils. The hydrofined Arabian gas oils and the hydrofined shale oils of about the same nitrogen content have roughly equivalent crackabilities based on the space velocities (WHSV) and catalyst/oil weight ratios employed in the two pilot plant operations. Coke yields for the shale oils were appreciably lower than those for the Arabian gas oils at constant conversion. C_3 yields were 1-3% on feed higher for the shale oils; C_4 yields were approximately 4% on feed higher for the shale oils. Gasoline yield for the more severely hydrofined shale oil was about 3.5% lower than that for the more severely hydrofined Arabian gas oil. At the higher nitrogen level of about 860 ppm, the shale oil produced about 1% more gasoline than the Arabian gas oil at the same conversion.

Product Qualities

Product inspections for the light and heavy gasolines and for the light and heavy cycle oils are presented in Table V. The gasolines and cycle oils have properties well within the range which we experience for such fractions derived from the cracking of gas oils from petroleum. In this report the only comparison between hydrofined shale oils and hydrofined Arabian gas oils will be that of the gasoline octane numbers. Table VI makes that comparison. The F-1 clear octane numbers for the hydrofined shale oil light gasolines are 0.7-to-1.0 octane number lower than those from the Arabian gas oils. The F-2 octane numbers are, perhaps, about 0.5 octane number lower for the shale oil light gasolines. However, the shale oil heavy gasoline F-1 clear octane numbers are 0.5-0.7 higher than those of the Arabian heavy gasolines, while F-2 clear octane numbers are approximately the same for both types of heavy gasolines.

Conclusion

Shale oil makes an excellent catalytic cracking feedstock provided its nitrogen content is reduced to at least 0.1% by appropriate hydrofining. Gasolines and cycle oils derived from the cracking of hydrofined shale oils are similar to those obtained from the cracking of hydrofined petroleum gas oils.

Program

We plan to continue the current whole shale oil hydrofining pilot plant run to prepare sufficient feed for downstream processing studies and to obtain additional information on the effects of process variables on catalyst activity and fouling rate.

Twenty-three gallons of whole product is currently being distilled to prepare a 650°F+ hydrofined shale oil feed for a larger scale catalytic cracking test scheduled for later this month. An additional distillation will then be made to prepare feed for downstream hydrocracking tests.

We expect to run out of our current supply of whole shale oil sometime in mid-May. We are, however, expecting another shipment of whole shale oil from the ERDA Laramie Laboratory which will allow us to continue these studies.

III. ^C~~SRC~~ Processing

Task 1-A--Preliminary
Feedstock Analysis

We have received three shipments of SRC from the Pittsburgh and Midway Coal Mining Company at Du Pont, Washington, as shown in the following table:

Chevron Identification Number	Quantity Drums	Shipment Date	Type of Material
WOW 3406	6	Dec. 14, 1976	SRC Flakes
WOW 3450	6	March 15, 1976	SRC Fines
WOW 3453	6	March 21, 1976	SRC Flakes

The flaked material was from the stainless steel cooling belt, while the fines were from the product solidification and storage area dust collectors at the SRC pilot plant.

Table VII shows a comparison of analyses of the SRC samples obtained by Chevron and by the Pittsburgh and Midway Coal Mining Company. Table VIII shows the metal content of SRC by emission spectrochemical analyses. Figures 2 and 3 show the qualitative metal analyses of SRC and the ash from SRC, respectively, with energy-dispersive X-ray fluorescence (EDXRF).

Task 1-C--HDF Feed Preparation

To hydroprocess SRC in a fixed bed reactor, the SRC flakes or fines need to be in a form which can be pumped at the operating temperatures of the equipment. The report on SRC handling by Air Products and Chemicals, Incorporated,¹ indicates that a homogeneous mixture of SRC (50 wt %) and creosote oil (50 wt %) can be prepared and handled easily.

The creosote oil used in our tests was obtained from Allied Chemicals (Identification 24-CA). It was washed with water and filtered with a 15- μ filter. Then it was distilled. The creosote oil blend component (WOW 3366) is the 70% over-head from distillation.

¹E. J. Greskovich, "Monthly Technical Progress Report No. 20, Chemical Characterization, Handling, and Refining of SRC to Liquid Fuels," ERDA Contract E(49-18)-2003, Corporate Research Department, Air Products and Chemicals, Incorporated, March 1977.

We prepared a feed blend by charging 440 lb of SRC (WOW 3406) and 440 lb of creosote oil (WOW 3366) to a kettle under a N₂ blanket and heating of 370°F with stirring. A homogeneous mixture was obtained after two-hour stirring at 370°F. Table IX shows the inspections of the feed (WOW 3476) and its components. Figure 4 shows the simulated distillation curves.

Task 2-A--Hydrofining Tests

A. Pumping Experiment

We designed a pumping experiment on the 50/50 wt % mixture of SRC/creosote oil to answer: (1) Can we get a steady pumping rate? (2) To what temperature should we heat the feed? (3) Is stirring needed?

We charge 20 lb of SRC and 20 lb of creosote oil to a feed pot surrounded by band heaters and with an internal stirrer attached to the lid. It was placed on a scale. The rate of weight decrease indicates the feed rate. Pumping began after the feed pot was heated to 370°F with stirring for 2 hr. A Milroyal pump (Model A) was used to pump the feed to an autoclave at 1500 psig. All the transfer lines were heated to 370°F. A steady rate from 85 g/hr to 900 g/hr was achieved.

Then the stirrer was turned off, and the feed pot was cooled to 200°F. We were still able to maintain a steady pumping rate.

Feed samples were taken near the top and the bottom of the feed pot and analyzed for homogeneity. Preliminary analyses indicate that the feed blend is homogeneous at 200°F without stirring. However, additional data are needed to verify this observation.

Once a homogeneous mixture of SRC and creosote oil is obtained by initially heating them to 370°F with stirring, it appears to remain homogeneous. A steady pumping rate is achieved at 200°F without stirring.

Program: Preliminary HDF Test

The preliminary HDF pilot plant test with the 50/50 wt % SRC/creosote oil (WOW 3476) is starting the week of May 2 using ICR 106 catalyst at 0.2 LHSV, 2,500 psig total pressure and 10,000 SCF/B recycle H₂. 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.

This first pilot plant test will serve as a shakedown run to determine whether the pilot plant can handle the SRC-creosote oil blend without modification, to determine catalyst activity, and, if possible, to determine deactivation rate and preliminary yields and product inspections.

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TABLE I

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

Identification No.	CCL-5406	CCL-5407 (SGQ 6115)
<u>Inspections</u>		
Gravity, °API	30.9	30.8
Aniline Point, °F	212.3	210.4
Sulfur, ppm	<10	Incomplete
Nitrogen, ppm	385	870*
Oxygen, ppm	200	200
Molecular Weight (Average)	365	383
Pour Point, °F	Incomplete	+100
Ramsbottom Carbon, %	Incomplete	0.19
Hot Heptane Asphaltenes, ppm	Incomplete	Incomplete
TBP Distillation (Simulated by Chromatography)	Incomplete	Incomplete
Carbon, %	Incomplete	Incomplete
Hydrogen, %	Incomplete	Incomplete
<u>Viscosity, cSt</u>		
at 122°F	Incomplete	
at 210°F	Incomplete	
at 300°F	Incomplete	

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

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TABLE II
CRACKING CATALYST INSPECTIONS

Identification No.	CCL-4914
Catalyst Type	Davison CBZ-1
Source	Equilibrium Catalyst from an FCC Unit
Chevron Research Activity, LV % Conversion*	69.7
<u>Physical Properties</u>	
Surface Area, m/g	107
Apparent Bulk Density, g/cc	0.779
Pore Volume, cc/g	0.38
<u>Particle Size Distribution, Wt %</u>	
0-20 μ	0.0
20-40 μ	0.5
40-80 μ	64.0
80+ μ	35.5
Average Particle Size, μ	72.6
<u>Metals, ppm</u>	
Nickel	266
Vanadium	318
Iron	3020
Copper	9
Chromium	75

*Chevron Research activity is defined as the conversion (LV %) obtained in cracking a light East Texas gas oil (35.4°API; 500-700°F) in a fixed-fluidized test unit (FCTU) operated in a cyclic manner. Conversion is defined as 100- vol %, 430°F+ liquid. Test conditions are:

Reactor Temperature, °F	925
Feed Rate, ml/Min.	30
Feed Period, Min.	10
Catalyst Charge, g	600

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TABLE III

CATALYTIC CRACKING OF HYDROFINED SHALE OILS
OPERATING CONDITIONS AND PRODUCT YIELDS

Run	FCT 5-1038		FCT 5-1039	
Feed	CCL-5406		CCL-5407	
Catalyst	CCL-4914		CCL-4914	
<u>Operating Conditions</u>				
Temperature, °F	975		975	
Catalyst Charge, g	600		600	
WHSV	5.96		5.96	
Cat./Oil Weight Ratio	2.01		2.01	
	Wt %	LV %	Wt %	LV %
Conversion	78.84	80.98	73.45	75.38
<u>Product Yields</u>				
Coke	4.41		3.87	
H ₂	0.06		0.07	
C ₁	1.12		1.07	
Ethane	0.88		0.89	
Ethane	<u>0.78</u>		<u>0.71</u>	
Total C ₂ 's	1.66		1.60	
Propane	3.17	5.43	2.46	4.23
Propane	<u>5.97</u>	<u>9.97</u>	<u>5.15</u>	<u>8.60</u>
Total C ₃ 's	9.14	15.40	7.61	12.83
i-C ₄	7.15	11.06	5.73	8.88
n-C ₄	2.10	3.14	1.69	2.52
C ₄ Olefins	<u>6.45</u>	<u>9.21</u>	<u>6.00</u>	<u>8.57</u>
Total C ₄ 's	15.70	23.41	13.42	19.97
Light Gasoline (C ₅ -250°F)	29.64	38.35	28.70	37.05
Heavy Gasoline (250-430°F)	<u>17.11</u>	<u>17.81</u>	<u>17.11</u>	<u>17.97</u>
Total Gasoline (C ₅ -430°F)	46.75	56.16	45.81	55.02
Light Cycle Oil (430-625°F)	9.68	8.88	10.73	10.04
Heavy Cycle Oil (625°F+)	<u>11.48</u>	<u>10.14</u>	<u>15.82</u>	<u>14.58</u>
Total Cycle Oil (430°F+)	21.16	19.02	26.55	24.62
C ₃ + Liquid Product	92.75	113.99	93.39	112.44

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TABLE IV
CATALYTIC CRACKING OF HYDROFINED ARABIAN LIGHT VACUUM
GAS OILS
OPERATING CONDITIONS AND PRODUCT YIELDS

Run	PP 158-66D		PP 158-67C		PP 158-63B		PP 158-62D	
Feed	CCL-4993				CCL-4990			
Catalyst	CCL-4914							
<u>Operating Conditions</u>								
Reactor Temperature, °F	973		976		974		975	
WHSV	5.01		5.08		5.04		5.03	
Cat./Oil Wt Ratio	3.80		7.81		4.37		7.12	
Conversion	Wt %	LV %	Wt %	LV %	Wt %	LV %	Wt %	LV %
Product Yields	75.91	78.12	79.74	81.93	72.18	74.61	75.38	77.83
Coke	4.67		6.41		6.47		8.27	
H ₂	0.18		0.18		0.24		0.24	
C ₁	1.25		1.19		1.38		1.27	
C ₂	2.22		1.98		2.56		2.18	
Propane	2.32	4.07	2.60	4.57	1.92	3.40	2.07	3.68
Propane	4.88	8.33	4.86	8.30	4.84	8.37	4.89	8.45
Total C ₃ 's	7.20	12.40	7.46	12.86	6.76	11.77	6.96	12.13
1-C ₄	4.95	7.84	6.05	9.58	3.76	6.03	4.54	7.28
n-C ₄	1.76	2.68	1.98	3.03	1.44	2.22	1.59	2.45
C ₄ Olefins	4.94	7.21	4.70	6.86	5.08	7.51	5.07	7.50
Total C ₄ 's	11.65	17.73	12.74	19.47	10.28	15.76	11.20	17.23
Light Gasoline (C ₅ -250°F)	28.49	36.91	30.14	39.13	24.96	32.75	25.71	33.78
Heavy Gasoline (250-430°F)	20.18	21.57	19.62	20.89	19.41	21.11	19.36	20.97
Total Gasoline C ₅ -430°F)	48.66	58.48	49.76	60.02	44.37	53.86	45.06	54.74
Light Cycle Oil (430-625°F	13.37	12.53	11.65	10.77	13.70	13.07	12.29	11.61
Heavy Cycle Oil (625°F+)	10.72	9.36	8.61	7.29	14.12	12.33	12.33	10.56
Total Cycle Oil (430°F+)	24.09	21.88	20.26	18.07	27.82	25.39	24.62	22.17
C ₃ + Liquid Product	91.60	110.49	90.22	110.42	89.23	106.78	87.85	106.27

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TABLE V
CATALYTIC CRACKING OF HYDROFINED SHALE OILS
PRODUCT QUALITY

Run Stock	FCT 5-1038				FCT 5-1039			
	Lt Gaso. ST-250°F	Hvy Gaso. 250-430°F	Lt Cycle Oil 430-625°F	Hvy Cycle Oil 625°F+	Lt Gaso. ST-250°F	Hvy Gaso. 250-430°F	Lt Cycle Oil 430-625°F	Hvy Cycle Oil 625°F+
<u>Inspection</u>								
Gravity, °API	65.2*	37.6	17.5	12.0	63.7*	39.0	20.3	18.3
Aniline Pt, °F	113.2	<30	<30		110.0	<30	<30	
Bromine No.	62	13	20		86	22	28	
Sulfur, %		0.006	0.027	0.28		0.006	0.009	0.15
Nitrogen, ppm	2.6	39	130	726	9.7	141	388	1400
<u>FIAM</u>								
P + N	62	24			56	27	17	
O	31	8			38	13	4	
A	7	68			6	60	79	
			cSt	SUS			SUS	cSt SUS
<u>Viscosity</u>								
at 100°F			2.449	34.23			34.26	21.08 102.3
at 130°F			1.764	-			32.09	11.50 64.57
Pour Pt, °F			0	+70			-20	+85
<u>Octane No.</u>								
F-1, Clear	89.0	95.3			90.3	93.3		
F-2, Clear	79.1	84.6			79.5	82.8		

*Gravity of 140-250°F fraction does not include C₅+ material recovered from the gas.

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TABLE VI
CATALYTIC CRACKING OF HYDROFINED SHALE OILS
GASOLINE OCTANE NUMBERS

Feed Feed Nitrogen, ppm	Hydrofined Shale Oil		Hydrofined Arabian Light Gas Oil			
	385	870	320		860	
Conversion, LV %	80.98	75.38	78.12	81.93	74.61	77.84
<u>Octane Numbers</u>						
<u>Light Gasoline</u> <u>(C₅-250°F)</u>						
F-1 Clear	89.0	90.3	91.2	-	90.2	-
F-2 Clear	79.1	79.5	80.8	-	78.8	79.4
<u>Heavy Gasoline</u> <u>(250-430°F)</u>						
F-1 Clear	95.3	93.3	94.4	94.4	93.0	93.6
F-2 Clear	84.6	82.8	83.3	84.2	82.8	83.5

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ANALYSES ON SRC FROM THE PITTSBURG AND MIDWAY COAL MINING COMPANY

Sample Data Source Sample No.	SRC Flakes ¹	SRC Flakes ¹	SRC Flakes ²	SRC Flakes ²	SRC Dusts ³	SRC Dusts ³
	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway
	WOW 3406		WOW 3453		WOW 3450	
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 (By CS-44)	0.71	0.69 (By CS-44)	0.74	0.67 (By CS-44)	0.66
Total N, Wt %	2.04	2.00	2.21	2.11	2.13	2.24
Basic N, Wt %	0.86		0.78		0.91	
<u>Distillation</u>	TGA		TGA		TGA	
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	

¹Shipped to Chevron Research on December 14, 1976.

²Shipped to Chevron Research on May 21, 1977.

³Shipped to Chevron Research on March 15, 1977

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TABLE VIII
METALS IN SRC BY
EMISSION ANALYSES

Sample	SRC Flakes
Data Source	Chevron Research
Sample No.	WOW 3406
<u>Metals, ppm</u>	
Al	42
B	17
Ca	60
Cr	1
Fe	98
Mg	2
Mn	5
Si	4
Ti	147
V	3
Zn	6

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TABLE IX
THE INSPECTIONS OF HDF FEED
AND ITS COMPONENTS

Sample Sample No.	SRC Flakes WOW 3406	Creosote Oil WOW 3366	SRC/Creosote Oil 50/50 WOW 3476
Specific Gravity		1.11	1.14
Ash, Wt %	0.22		
H, Wt %	6.12	6.14	5.67
C, Wt %	87.78	90.62	88.97
O, Wt %	4.52	1.11	
S, Wt %	0.89	0.64	
Total N, Wt %	2.04	0.78	1.46
Basic N, Wt %	0.86	0.45	0.33
<u>Distillation</u>	TGA	TPG	TGA
Start	159	343	153
5	943	406	573
10	1017	446	648
30	1161	534	802
50	1232	599	977
70		631	1231
90		680	
95		687	
End Point	1281	726	1284
Rec., %	55.2	99.0	72.2

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FIGURE 1

ERDA CONTRACT EF-76-C-01-2315 PILOT PLANT RYN 86-51
WHOLE SHALE OIL HYDROFINING WITH 1CR 10GL CATALYST
0.6 LHSV 8000 SCF/G RECYCLE GAS RATE

NORMALIZED
CATALYST
TEMPERATURE,
°F (°C)

H₂ PARTIAL
PRESSURE, PSIA

WHOLE LIQUID
PRODUCT NITROGEN,
PPM

800
780
760
2200
2000
1800
1600
1500
1000
500

TOTAL
PRESSURE
2200 PSIG

TOTAL PRESSURE
2350 PSIG

NEW
FEED
GRL

HIGH GAS
LEAK

TEMP CONTROLLER
MALFUNCTION;
1ST CAT BED
TEMP TO 915°F

HIGH
FEED
RATE

200 400 600 800 1000 1200 1400

TIME ONSTREAM, HR.

* CATALYST TEMPERATURES NORMALIZED TO
500 PPM PRODUCT NITROGEN

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FIGURE 2
EXDRF ANALYSIS OF SOLVENT-REFINED COAL

WOW 3450
1000 Counts Full Scale

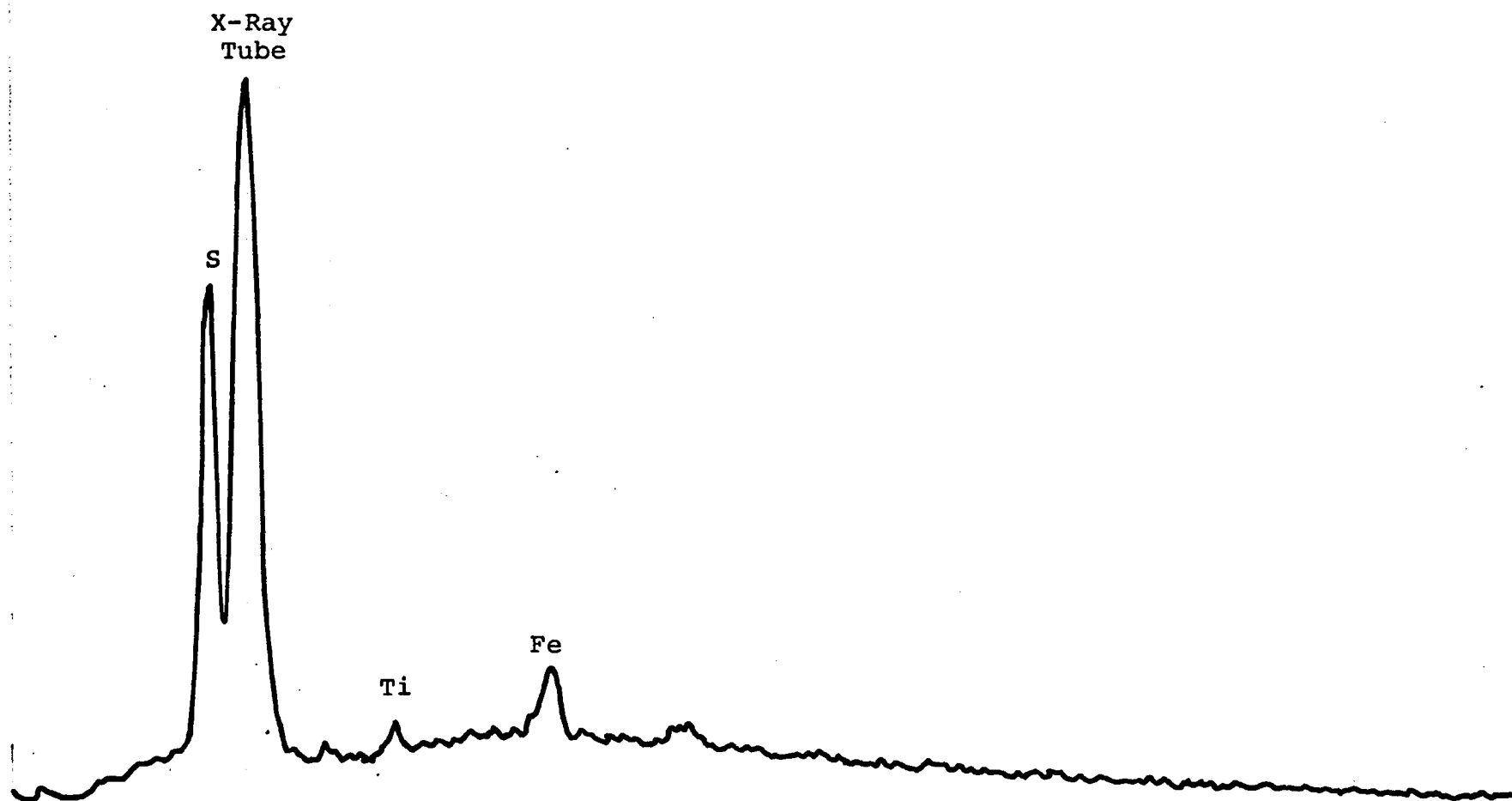
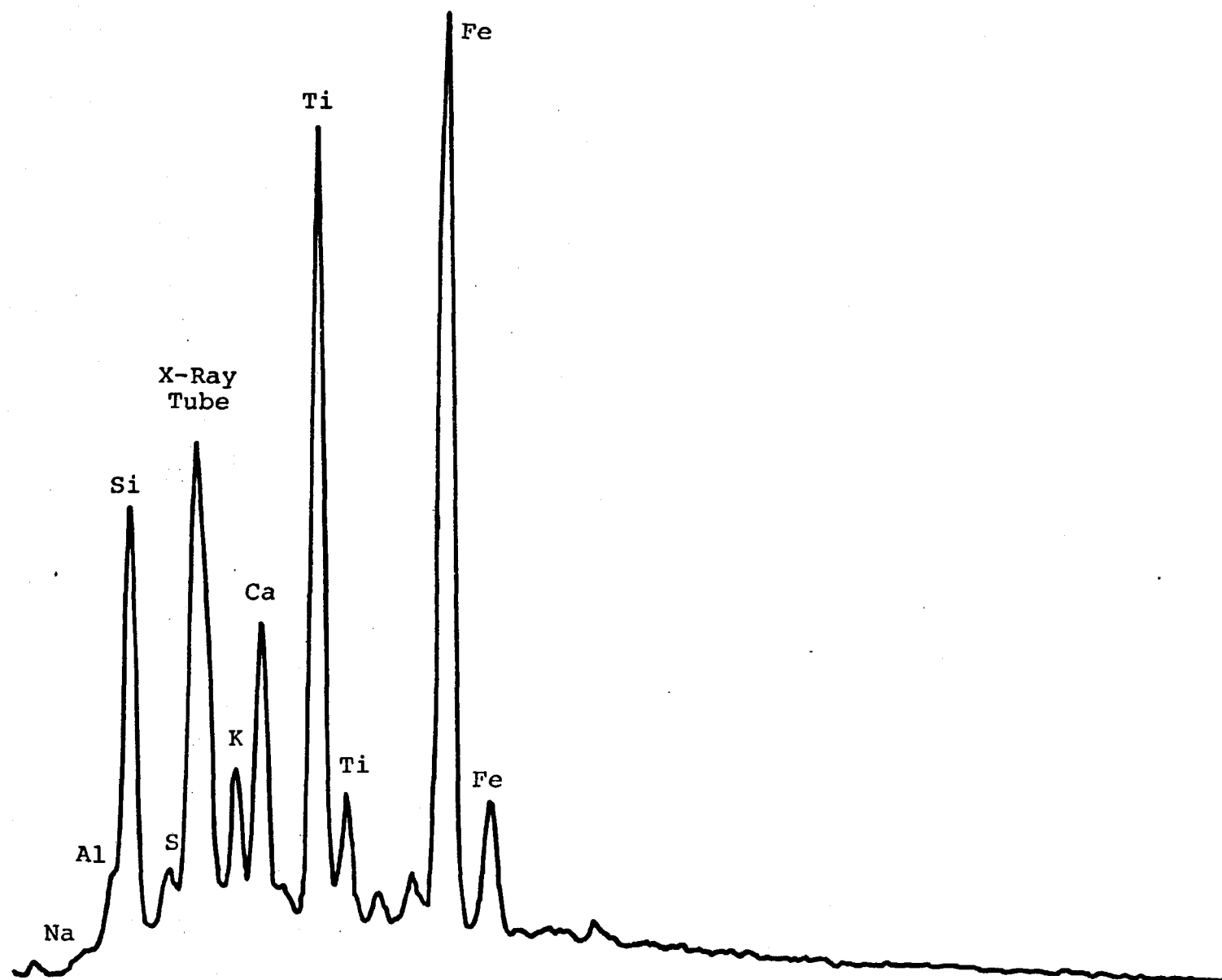


FIGURE 3

EXDRF ANALYSIS OF THE ASH FROM SOLVENT-REFINED COAL

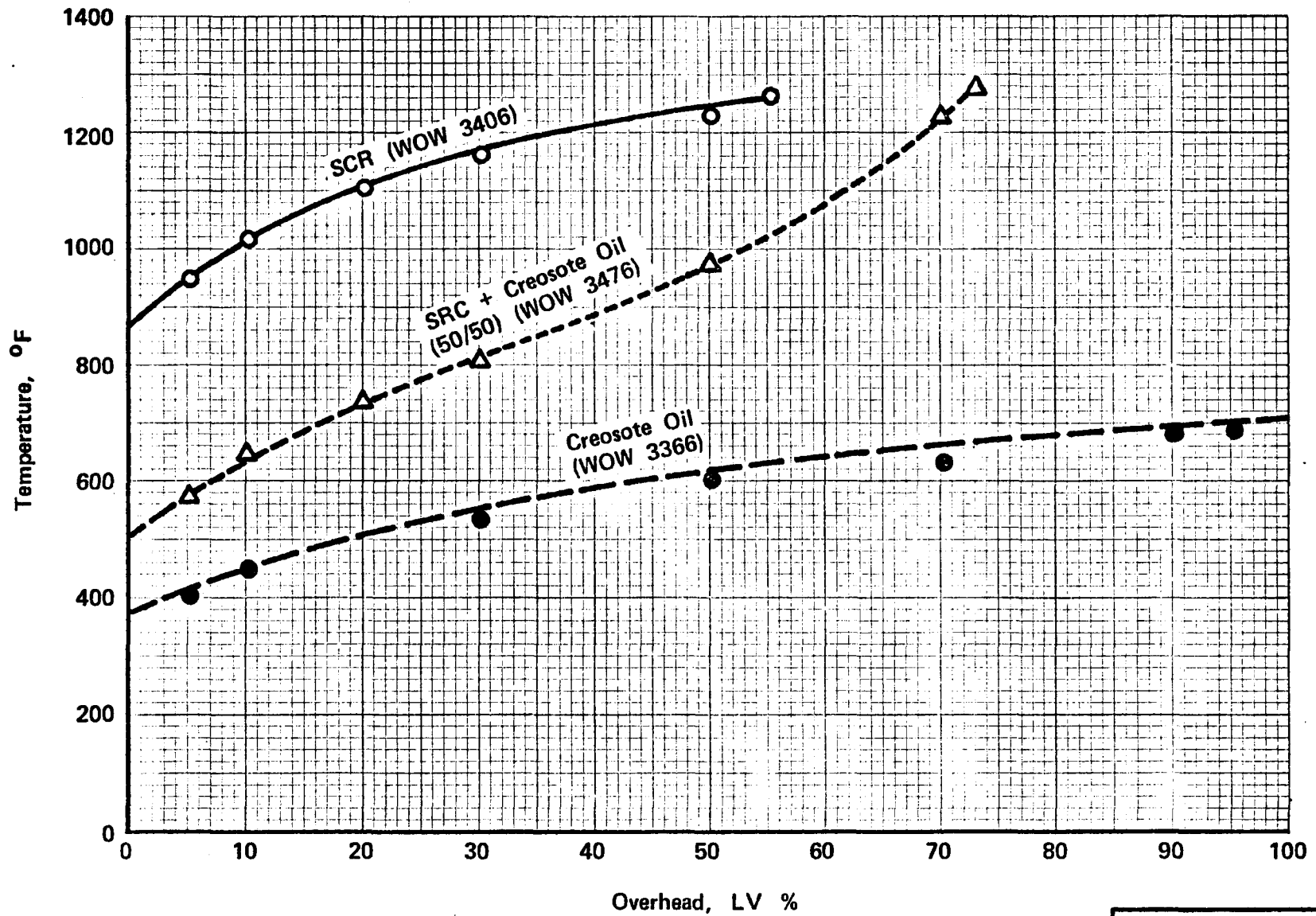
WOW 3450
1000 Counts Full Scale



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FIGURE 4

SIMULATED DISTILLATION CURVES



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