Low Sulfur Synthetic Crude Oil from Coal

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ABSTRACT

Project COED (Char-Oil-Energy Development) was initiated in 1962 when the Office of Coal Research, Department of the Interior, contracted with the FMC Corporation to develop a process for upgrading coal to a synthetic crude oil (syncrude), a salable gaseous product and char. Successful operation of bench scale units led to the design, construction and operation of a 36 ton-per-day pilot plant at the FMC Corporation Chemical Research and Development Center in Princeton, New Jersey. The pilot plant has operated successfully since July, 1970.

The COED process consists of pyrolyzing pulverized, dried coal in a series of fluidized bed reactors. The oil vapors in the pyrolysis off-gases are condensed using a direct water quench. The oils are separated from the water, dried and filtered with a pressurized, rotary-drum precoat filter. Filtration removes carbonaceous dust from the pyrolysis oil prior to hydrotreating in a fixed-bed catalytic reactor containing commercial nickel-molybdenum catalyst. Hydrotreating at reaction conditions of 2500 psig and 750°F produces a low sulfur syncrude from the heavier oils recovered from the pyrolysis of coal. The syncrude can be used as a feedstock to a refinery or burned as a No. 4 fuel oil. Utilization of the syncrude for these purposes is presented here.

PYROLYSIS SECTION

A schematic of the COED process is shown in Figure 1. The dried and crushed coal is heated to successively higher temperatures in a series of fluidized beds. In each bed, a fraction of the volatile matter of the coal is released. The temperature of each bed is selected to be just below the maximum temperature to which the coal can be heated without agglomerating and defluidizing the bed. Typical operating temperatures of the fluidized beds are 600, 850, 1000 and 1600°F respectively. The number of stages and the operating temperature vary with the type of coal processed. Heat for the process is generated by burning some of the char in the fourth stage and then using the hot gases and hot recycle char as the source of heat for the other reactors. These hot fluidizing gases pass countercurrent to the char flow and collect volatile matter from the coal which is released as oil vapors and light hydrocarbon gases.

The pyrolysis recovery system is outlined in Figure 2. The product gas leaving the second stage reactor at 850°F is directly quenched with by-product water from the process. The condensed oil and water are separated in a decanter with the water recycled for cooling operations. The collected COED pyrolysis oil containing char fines is dried and filtered in a pressurized, rotary-drum precoat filter where solids are removed to give a filtrate with less than 0.1 weight percent solids. The filtered oil is the feed to the hydrotreating section of the pilot plant.

HYDROTREATING SECTION

The hydrotreating section is shown in Figure 3. The oil feed system to the hydrotreating section is kept at 250-300°F to keep the oil pumpable. This oil is pumped to 2500-3000 psig with plunger pumps and mixed in a packed vessel (guard chamber) with hot recycle gas. The recycle gas has been superheated in a convective type furnace to a temperature that results in an oil-gas mixture temperature in the guard chamber of 700-750°F. The guard chamber serves several functions:

1. Provides for a direct contact heat transfer zone thereby eliminating the need for preheating the COED pyrolysis oil in small preheater tubes;

2. Allows for the partial removal of contaminants such as metals and coke thereby extending the life of the catalyst beds;

3. Minimizes system pressure drop losses by utilizing a high free volume packing material for the accumulation of coke and metal deposits.

The guard chamber effluents pass downflow through the reactor vessel containing two fixed-beds of nickel molybdate catalyst. The catalyst used in the pilot plant is American Cyanamid's Aero HDS-3 series, which contain 3.0 percent NiO and 15 percent MoO_3 on an alumina support. Both the 1/16-inch and 1/8-inch extrudates are used. The reactor vessel consists of a 12-inch diameter pipe, 25 feet long, with provision for an 8-foot catalyst bed in the upper section and a 12-foot catalyst bed in the lower section. Recycle hydrogen quench is provided between reactor stages to maintain the desired bed temperature profiles. High hydrogen-to-oil ratios (40M to 80M SCF/bbl) are used in the pilot plant for two reasons: (1) as a heat transfer agent to provide the sensible heat necessary to bring the COED oil to reaction temperatures and (2) as a diluent to control the temperature rises in the catalyst beds due to the exothermic heats of reaction. In the commercial unit, the high hydrogen-to-oil ratios can be reduced by using liquid product recycle as the diluent. For example, at 15,000 SCF per barrel fresh COED oil, liquid product recycle rates of 0.7 volumes recycle per volume fresh oil will result in an acceptable reactor temperature profile. Furthermore, this liquid recycle will decrease the concentration of metals in the feed to the reactors and thereby help to disperse the deposition of these metals more uniformly through the catalyst beds. Hydrogen is consumed by reacting with the sulfur, nitrogen, oxygen and the unsaturated hydrocarbons in the oil. Recycle hydrogen purity is controlled by purging a portion of the recycle hydrogen stream. Make-up hydrogen is fed on demand by a pressure controller to maintain the system pressure. The reactor effluents are cooled and liquid products recovered in two stages of heat exchange and flash vaporization. The syncrude is the liquid product mixture from the two flash drums.

Typical properties of the oil feed to hydrotreating are shown in Table 1. The filtered pyrolysis oil must be hydrotreated in order to remove contaminants such as sulfur, nitrogen and oxygen. At the same time that these contaminants are removed, the viscosity, pour point and API gravity of the oil are upgraded. Successful hydrotreating operations have been carried out with pyrolysis oils derived from Colorado Bear, Utah King, Wyoming Big Horn and Illinois No. 6-seam coals. The former two coals are representative of low sulfur, Western, high volatile B bituminous coals. Wyoming Big Horn is representative of a low sulfur, Western, sub-bituminous coal. Illinois No. 6-seam coal is a C bituminous coal. Typical hydrotreating operating conditions are shown in Table 2.

The COED process produces a full distillate range syncrude - 100°F to 900°F - which is suitable as either a refinery feedstock or as a source of various fuel and chemical products. Various synthetic fuels can be derived from fractionation and further processing of syncrude cuts. The properties of the various syncrude products presented are based mostly on one representative sample of a full-range syncrude derived from Illinois No. 6-seam coal.

FUEL TYPES AND YIELDS

Typical yields, distillation data and hydrocarbon type analyses of four syncrudes are shown in Table 3. Syncrudes derived from Western coals result in much higher paraffinic (24% vs. 10%) content and lower aromatic content (34% vs. 48%) than the Illinois type coals. The distillation fractions shown were chosen to simulate the following fuel types:

IBP-180°F	Fuel Gas
180-390°F	Gasoline
390-525°F	Jet Fuel
	Diesel Fuel
390-650°F	No. 2 Oil
390-EP	No. 4 Oil
	Stationary Turbine Oil
650-EP	No. 6 Oil

Although the fuel fractions from COED syncrude do not meet all the ASTM specifications, they do meet the basic requirements. Additional refining and processing will, in most cases, result in a finished product meeting all the required specifications. When the specifications are not met, syncrude fuels can be blended with petroleum fuels to meet the desired specifications. Alternatively, new specifications can be generated for the syncrude fuels such that these fuels can be adapted to existing furnaces and engines. A typical refinery product slate when feeding on an Illinois No. 6-seam syncrude is shown in Table 4. The maximum product yields occur in the middle distillate range which amounts to about 50 volume percent of the COED syncrude.

EXPERIMENTAL EVALUATION OF SYNCRUDE

FMC Corporation subcontracted with AtlanticRichfieldCompany (ARCO) to evaluate a COED syncrude derived from an Illinois No. 6-seam coal as a potential refinery feedstock. The studies were performed in laboratory and pilot plant equipment and included:

1. Fractionation of the full range syncrude
2. Hydrogen pretreatment and reforming of the naphtha fraction of syncrude
3. Evaluation of middle distillate fractions as jet, diesel, home heating and industrial heating fuels
4. Fluid catalytic cracking of the residual fraction of syncrude
5. Hydrocracking of the residual fraction of syncrude.

The detailed processing sequence used by ARCO is outlined in Figure 4. In some cases, particular fuels from syncrude fail to meet some of the ASTM specifications but these fuels are still valuable as blending feedstocks with available specification petroleum-derived fuels. The required blending ratios can be determined from existing correlations when actual experimental data is not available. The major specification not achieved is API gravity owing to the high aromaticity of the coal-derived oil. In this case, an on-specification fuel blend is confidently obtained by volumetric blending of the syncrude fuel with the petroleum fuel. The following is a summary of the experimental evaluations performed by ARCO.

Naphtha Evaluation

Naphthas derived from syncrude contain about 73 percent naphthenes, 7 percent paraffins and 20 percent aromatics with about 100 ppm each of sulfur and nitrogen. The high naphthenic content results in excellent reformer feedstock once the sulfur and nitrogen impurities are removed to prevent poisoning the noble metal reforming catalyst. A hydrogen pretreatment is therefore required to obtain a clean reformer feedstock. The reforming operation with this feedstock results in extremely low aging rates compared to processing conventional petroleum naphthas. The reformate produced had better than 100 Research Octane Number Clear (RONC) and resulted in a material with 80 percent aromatics content.

Middle Distillate Evaluation

The middle distillate range of COED syncrude is highly aromatic and consequently does not have all the desired properties of jet and diesel fuel. However, hydroprocessing of these fractions results in obtaining reasonable product quality, such that middle distillates derived from syncrude can be blended with available petroleum distillates to make a specification fuel. More severe hydrotreatment of the syncrude middle distillates results in acceptable diesel fuels, No. 4 oils, stationary turbine fuels and No. 2 oils with little or no blending necessary.

Cracking of Residuum

Hydrogen pretreatment of the 650°F to end point fraction of syncrude is required prior to fluid catalytic cracking (FCC) and fixed-bed hydrocracking operations. This is necessary to protect the cracking catalysts from deactivators such as nitrogen and polynuclear aromatics. With both the FCC and hydrocracking operations, additional fuel gas, naphthas and middle distillates are produced from the residual fraction of syncrude. The residuum from the cracking operations of the 650°F to end point fraction of syncrude can be used as a very low sulfur No. 6 fuel oil.

PROPERTIES OF SYNCRUDE FUELS

Gasoline

After reducing the sulfur content of the COED naphthas to 1-2 ppm, the catalyst aging rates for reforming the COED naphthas are extremely low compared to those with petroleum-derived naphthas. This should allow reforming operations for producing high octane reformates to be carried out without the need for severe, high pressure pretreatment. Hydrogen production and C_5 to end point (C_5+) yields from COED naphthas were extremely high compared to conventional petroleum reforming. C_5+ reformate yields were in excess of 90 volume percent with Research Octane Number Clear levels between 99 and 102.

Additional naphtha feedstocks for reforming are produced by fluid catalytic cracking and fixed-bed hydrocracking of the 650°F to end point fraction of syncrude. Table 5 shows a summary of yields and type analyses of naphthas derived from syncrude. It can be seen from the table that syncrude is an excellent source of high octane material for either the leaded or unleaded gasoline pool. In a typical refinery feeding on syncrude, approximately 34 volume percent of the syncrude feedstock can be converted to acceptable, low sulfur, high octane gasoline.

Fuel Oils

The full range syncrude, the middle distillate fractions and the residual fraction of syncrude can be used either as straight run fuels or as blending fuels with conventional petroleum fuels. The properties of ASTM No. 2, No. 4 and No. 6 fuel oils are compared with various syncrude fractions in Table 6.

The 350-End Point fraction of syncrude was burned as fuel oil in a laboratory burner test. The stack gas emissions were extremely low and the fuel closely resembled that of a No. 2 grade oil. Particular significance of the emissions tests as outlined in Table 7 is the reduction in particulate emissions compared to that of a typical asphaltic No. 4 residual fuel. SO_x, NO_x and hydrocarbons emissions are also appreciably lower for syncrude. This fuel oil fraction was also determined to be an acceptable No. 4 oil as regards storage stability. Syncrude was stable with regard to the production of insoluble sludges and varnish as shown in Table 8. Even after exposure to air at 150°F for two months, only moderate deterioration occurred. The ASTM compatibility ratings indicate that there is no problem involving precipitation of insoluble sludges when blending with paraffinic straight run diluents such as might be done at dockside when blending to meet marine diesel quality.

Engine Fuel

Engine fuels such as JP-5 jet fuel and No. 4-D diesel fuel can be derived from syncrude but with additional hydroprocessing required. The middle distillate fractions of syncrude, in particular, the 390-525°F fraction and the 390-650°F fractions, are applicable to these types of fuels. One of the key factors regarding the quality of engine fuels is the degree of aromaticity which adversely affects such properties as smoke point, luminometer number, specific gravity and cetane number. Middle distillate fractions of syncrude are higher in aromatics than petroleum distillates and are, therefore, unsuitable as engine fuels. However, severe hydrotreatment can result in enough saturation to decrease the aromatics level to less than a few percent. Comparisons of ASTM specification jet and diesel fuels respectively with various middle distillates derived from an Illinois No. 6 syncrude are shown in Tables 9 and 10. The last column on each table lists properties of a middle distillate fraction derived from syncrude that was hydroprocessed in a bench-scale unit. Although all specifications were not achieved, other hydroprocessing conditions might result in a satisfactory JP-5 jet fuel or diesel fuel product. In either case, the syncrude products, with their exceedingly low sulfur content, should serve as suitable blending fuels with conventional petroleum fuels. Yields and aromatics levels for syncrudes from four different coals are shown in Table 11. Western coals, such as Utah King, result in syncrude middle distillate fractions with less than half the aromatics content of middle distillates derived from Illinois No. 6 syncrude. These oils should indeed be more suitable for engine fuel production and would most assuredly result in less severe hydroprocessing conditions necessary to meet fuel specifications.

CONCLUSIONS

The COED process has been successfully demonstrated on a pilot-plant scale resulting in the upgrading of coal to three salable products--gas, char and a low sulfur synthetic crude oil. The syncrude can be further processed in a refinery to produce more valuable products such as gasoline and chemicals. Alternately, the raw syncrude can be fractionated into a range of fuel products suitable for straight run usage or as blending fuels with conventional petroleum fuels. The extremely low sulfur content of syncrude products, when the latter are used as blending stocks, has the resultant effect of upgrading the value of available high sulfur petroleum fuels. The value of syncrude on a dollar per barrel basis when utilized in a petroleum refinery is presently being determined by Chem Systems, Inc.

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FIGURE 1

COED COAL PYROLYSIS

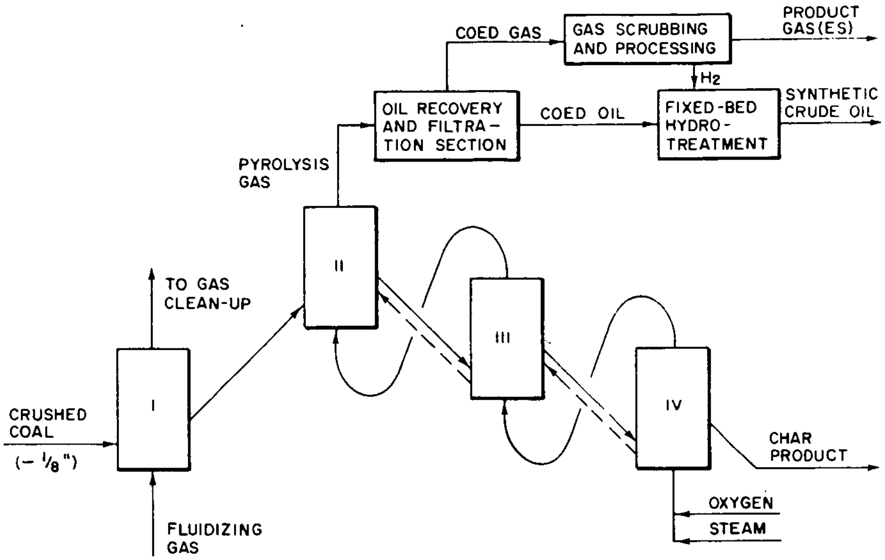

FIGURE 2

COED OIL RECOVERY SYSTEM

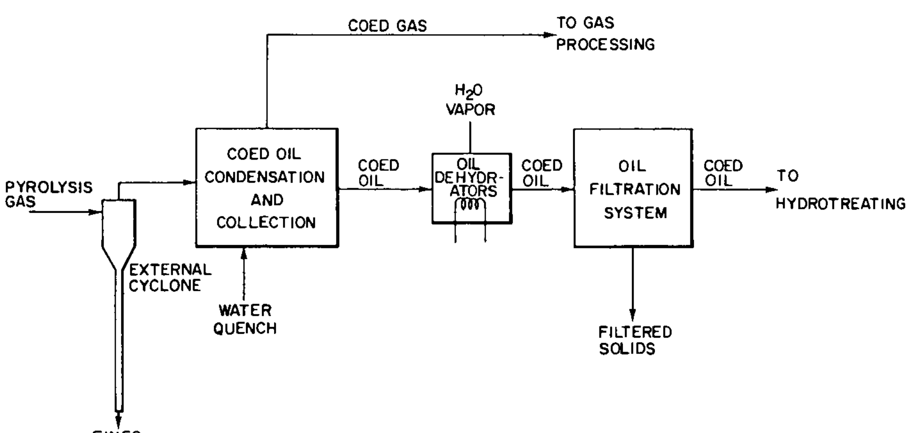

FIGURE 3
SCHEMATIC OF HYDROTREATING SECTION

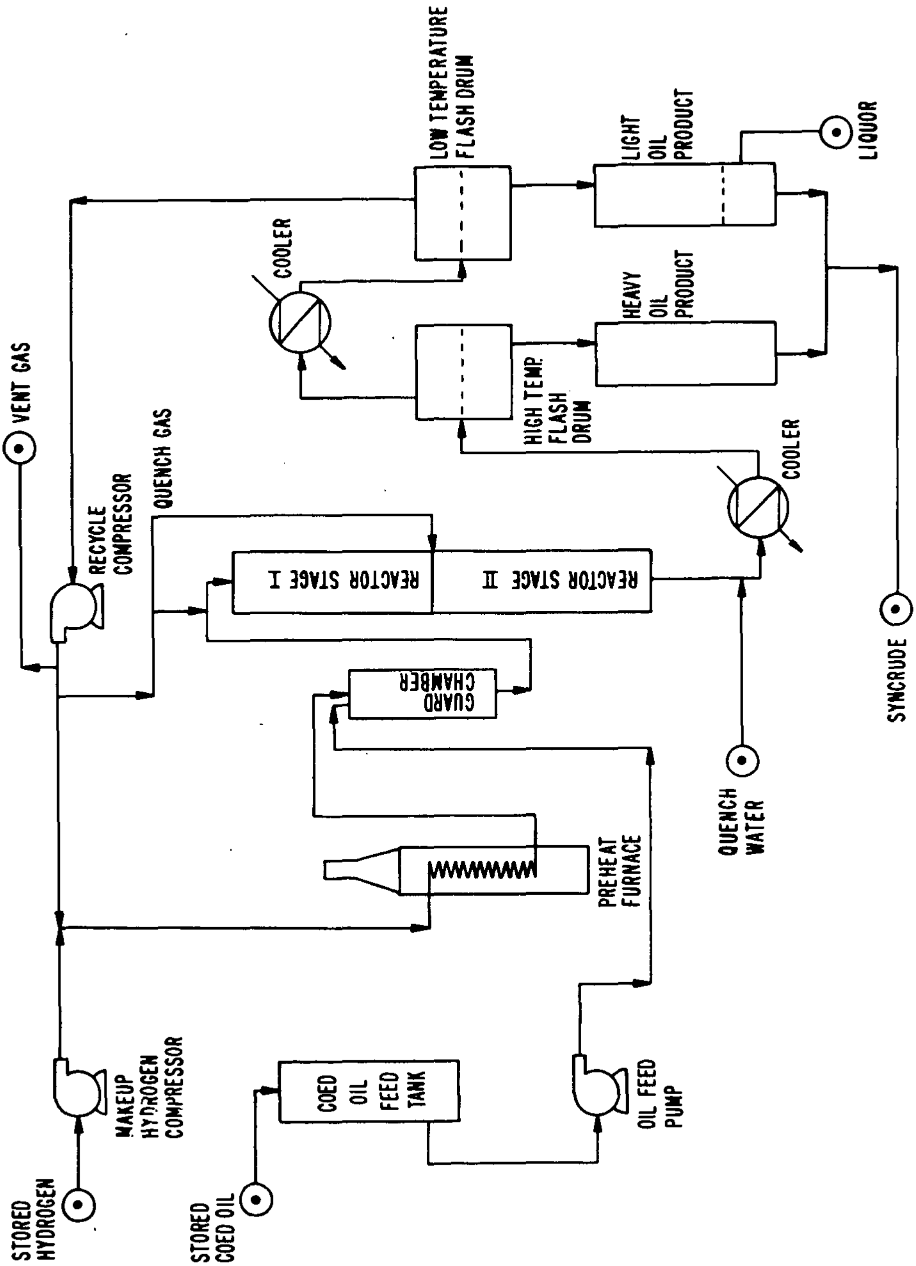

FIGURE 4

PROCESSING SEQUENCE

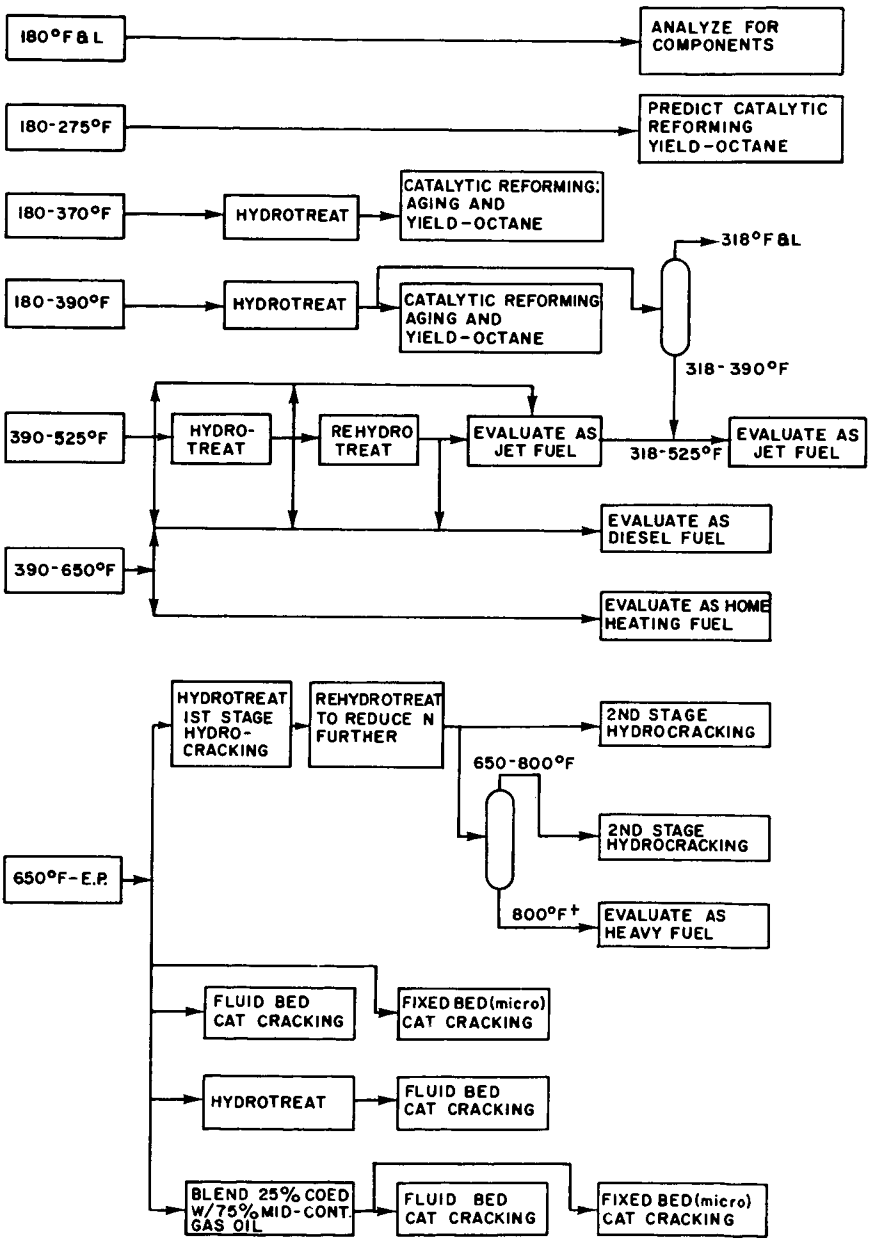

TABLE 1

Typical Properties of Hydrotreating Feed Oils

Coal Source	Colorado Bear	Utah King	Wyoming Big Horn	Illinois No. 6-Seam
Properties of Derived Oil				
Elemental Analysis				
Weight % dry				
Carbon	83.6	83.7	82.7	80.5
Hydrogen	8.3	8.6	8.0	7.0
Nitrogen	1.1	1.0	1.0	1.2
Sulfur	0.4	0.2	0.6	2.0
Oxygen	6.6	6.5	7.5	9.2
Ash	0.0	0.0	0.2	0.1
API Gravity, 60°F	-4	-3.5	-4	-4
Moisture, Weight %	0.2	0.1	0.4	0.2
Pour Point, °F	108	130	120	115
Viscosity, SUS 210°F	1090	390	228	1333

TABLE 2

TYPICAL PILOT PLANT HYDROTREATING OPERATING CONDITIONS

Illinois No. 6-seam Coal Derived Oil

Catalyst:	NiMo on Alumina extrudates
Pressure:	1750-2500 psig
Temperature:	700-800°F
Space Velocity:	0.3-0.6 lb. oil/hr./lb catalyst
Gas Recycle Rate:	40M to 80M SCF/bbl
Gas Recycle Concentration:	90-95% H_2
Hydrogen Consumption:	3500 SCF/bbl

TABLE 3

COED Syncrudes

Typical Yield, Distillation Data and Hydrocarbon Type Analyses

Coal Source	Illinois No. 6	Colorado Bear	Utah King
Hydrocarbon Type Analysis, Lvol %			
Paraffins	10.4	(2)	23.7
Olefins	NIL	(2)	NIL
Naphthenes	41.4	(2)	42.2
Aromatics	48.2	(2)	34.1
API Gravity, 60°F	28.6	24.4	28.5
ASTM Distillation, °F			
Initial Boiling Point (IBP)	108	200	260
50% distilled	465	540	562
End Point (EP) (1)	746	840	868
Fractionation Yields, wt. %			
IBP-180°F	2.5	0	0
180-390°F	30.2	28.0	5.0
390-525°F	26.7	22.0	35.0
390-650°F	51.0	50.0	65.0
650-EP	16.2	22.0	30.0
390-EP	67.2	72.0	95.0

(1) 95% except for Illinois No. 6 which is 98%

(2) Componential analyses not available

TABLE 4

COED SYNCRUDE

Typical Refinery Product Slate

Basis: 100 bbl. of Syncrude from Illinois No. 6-seam coal

Fraction	Yields, bbl. FCC (1)	Yields, bbl. HC (2)	End Use
Hydrogen, SCF	18000	6000	Hydrocracking
Initial Boiling Point-180°F	10.0	8.5	Liquefied Petroleum Gas, Refinery Gas
180-390°F	33.8	34.5	Gasoline
390-650°F	51.6	52.1	Middle Distillate Fuels (Jet, Diesel, No. 2)
650-End Point	2.8	3.1	Residual Fuel (No. 6)

(1) With Fluid Catalytic Cracking operation on 650-end point fraction of syncrude.

(2) With Hydrocracking operation on 650-800°F fraction of syncrude.

TABLE 5

COED NAPHTHAS

Source: Syncrude From Illinois No. 6-Seam Coal

Hydrocarbon Type Analysis, Liquid Volume %, Basis COED Syncrude	Raw Naphtha	Pretreated Reformer Feedstock	Reformate (1) (C_5+)	Cat (2) Naphthas	Hydrocracked (3) Naphthas
Paraffins	2.3	2.3	3.3	2.3	1.1
Naphthenes	23.7	24.4	1.6	2.3	4.5
BTX (6) and Alkylbenzenes	6.6	6.3	25.7	1.6	0.2
Indans/Tetralins	0.7	0.3	NIL	NIL	0
Olefins	NIL	0	0	2.0	0
API Gravity, 60°F	44	44	34	50	50.6
Sulfur, ppm	93	<1	<1	10	<10
Research Octane No. clear	70	--	99.5	87.4	100 (4)
Research Octane No. leaded	--	--	--	96.4	---
Yields, Basis COED Syncrude					
H_2 (SCF/bbl. Syncrude)	--	-100	+1500	--	--
C_5+ (Liquid Volume %)	33.3	33.3	30.6	8.0	4.9 (5)

(1) Gasoline produced by reforming 180-390°F fraction of syncrude.

(2) Gasoline produced by fluid catalytic cracking of 650-end point fraction of syncrude.

(3) Naphthas produced by hydrocracking 650-end point fraction of syncrude.

(4) Predicted RONC for hydrocracked naphtha after reforming over platinum catalyst.

(5) Predicted yield of hydrocracked naphtha reformate product.

(6) Benzene, Toluene, Xylene

TABLE 6

SYNCRUDE FUEL OILS

Comparisons with ASTM Specifications

Test	350-End Point Fraction	ASTM No.4	390-650°F Fraction	ASTM No.2	650-End Point Fraction	ASTM No.6
Flash Point, °F	160	130 min.	215	100 min.	400	150 min.
Pour Point, °F	+25	20 max.	-70	20 max.	70	---
Ash, Weight %	0.007	0.10 max.	0.0	---	---	---
Sulfur, Weight %	0.16	0.30 max.	0.004	0.30 max.	0.07	---
Water & Sediment, %	0.10	0.5 max.	---	0.05 max.	---	2.0 max.
Gravity, °API, 60°F	18.4	---	22.5	30 min.	11.2	---
Viscosity, SUS 100°F	52.5	45-125	39.3	32.6-37.9	---	900-9000
Carbon Residue (1)	0.4	---	---	0.35 max.	1.13	---
ASTM Distillation, °F						
Initial Boiling Point	354	---	436	---	557	---
10% distilled	409	---	459	460 max.	705	---
90% distilled	780	---	586	540-640 max.	870	---
End Point	---	---	613	660 max.	---	---

(1) Conradson Carbon Residue Test, ASTM D-189

TABLE 7

COED SYNCRUDE

Fuel Oil Stability Test

	Fresh	Stored (1)
ASTM D-2781 Compatibility Rating (2)	2	2-3
0.8μ Millipore Filter Residue, Weight %	0.01	0.01, 0.03
ASTM D-1661 Thermal Stability Rating	1-2	2

(1) Two months at 150°F, vented to air.

(2) As mixed with equal volumes of 350-650°F petroleum distillate. Same rating was observed with 100% coal liquid. Spot No. 2 is defined as "Faint or poorly defined inner ring"; No. 3 is "Well defined, thin, inner ring, only slightly darker than the background." A No. 3 rating or above indicates that a fuel may cause problems in field applications.

TABLE 8

SYNCRUDE FUEL OILS

390-End Point Fraction of Syncrude

Emissions Levels Measurements

Test	Result	Typical No. 4 Residual Fuel
Particulates, lbs./100 gal.	4.6	19.2
Gaseous Contaminants, ppm		
SO_x	60	490
NO_x	53	95
CO	4.5	6.0
HC	3.0	7.1

Test Conditions

Fuel was burned in a small industrial burner firing a 1.5 million Btu/hr. boiler. Stack gas is controlled at 12.5% CO_2, approximately equivalent to 25% excess air and stack temperature is held at 500°F to 510°F.

TABLE 9

COED SYNCRUDE

Comparison of JP-5 Jet Fuel Specifications with Syncrude

Properties	JP-5	390-525°F Fractions As Distilled	2 Stage HDS (1)
ASTM 10% Distillation point, °F	400 maximum	436	412
ASTM Distillation End point, °F	550 maximum	530	526
Gravity, °API, 60°F	36-48	25.7	33.2
Sulfur, Weight %	0.4 maximum	0.007	0.001
Freezing Point, °F	-51 maximum	<-70 (2)	<-80 (2)
Net Heating Value, Btu/lb.	18300 minimum	18070 (2)	18390 (2)
Aniline Gravity Product (3)	4500 minimum	1490	4501
Aromatics, Liquid Volume %	25 maximum	60.7	0 - 5
Olefins, Liquid Volume %	5 maximum	0	0
Smoke Point (4)	19 minimum	10	22
Luminometer Number (5)	50 minimum	20.4 (2)	43.6 (2)
Flash Point	140 minimum	186	156
Copper Corrosion Test (6)	1	1	---

(1) After hydroprocessing of 390-525°F syncrude fraction.

(2) Calculated

(3) A measure of the aromaticity of an oil.

(4) A measure of the smoke and soot-producing characteristics of jet fuels.

(5) A measure of the flame temperature of jet fuels.

(6) A measure of the corrosiveness of oil to copper, a "1" designation indicates slight tarnish.

TABLE 10

COED SYNCRUDE

Comparison of Diesel Fuel Specifications with Syncrude Middle Distillates

Properties	ASTM-No. 4-D Specifications	390-650°F (As Distilled)	525-650°F (As Distilled)	390-525°F (As Distilled)	390-525°F Fraction 2 Stage HDS (1)
Pour Point, °F	0 Maximum	-70	-20	<-80	<-80
Flash Point, °F	130 Minimum	215	300	186	156
Sulfur, Weight %	Legal	<.001	<.001	.007	<.001
ASTM Distillation, °F					
10% distilled	460 Maximum	459	562	436	412
50% distilled	--	(5)	588	455	437
90% distilled	620 Maximum	586	626	494	483
100% distilled	660 Maximum	613	(5)	530	(5)
Gravity, °API, 60°F	33 - 37	22.5	19	25.7	33.2
Cetane Number (3)	30 Minimum	18	24	23.0	>30 (2)
Aniline Point, °C (4)	--	(5)	(5)	14.3	58.2
Aromatics, Liquid Volume %	--	(5)	(5)	58.3	5.2

(1) After hydroprocessing of 390-525°F fraction of syncrude.

(2) Estimated

(3) A measure of the ignition quality of diesel fuel.

(4) A measure of the aromaticity of oil.

(5) Not measured.

TABLE 11

COED SYNCRUDE

Middle Distillate Fractions

Syncrude Source	Yields, Liquid Volume % Basis Syncrude 390-525°F Fraction	Yields, Liquid Volume % Basis Syncrude 390-650°F Fraction	Gravity °API,60°F(1)	Liquid Volume % Aromatics (1)
Colorado Bear	25	47	30.1	45.1
Utah King	40	75	33.0	29.8
Illinois No. 6-seam	24	51	25.7	60.7

(1) For 390-525°F fraction of syncrude only.