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(12) Patent:

(54) HIGH QUALITY LUBE OIL AND/OR JET FUEL FROM WAXY PETROLEUM FRACTIONS

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799453

The present invention relates to the hydroconversion of waxy petroleum fractions comprising hydrocarbons having at least twenty-one carbon atoms to lubricating oils having a Viscosity Index (VI) of at least 110 and/or jet fuel having a luminometer number of at least 100. More particularly, the invention relates to the hydroisomerization of waxy petroleum fractions comprising hydrocarbons having at least twenty-six carbon atoms at hydroisomerization conditions dependent upon the concentration of oil in the waxy petroleum fraction.

The influx of plastic coatings into the milk carton and wrapping paper business may be expected to reduce the demand for paraffin wax and microcrystalline wax for such purposes. Of course, both paraffin wax and microcrystalline wax can be catalytically cracked into gasoline and naphtha to <sup>decrease</sup>~~increase~~ excess of supply over demand. On the other hand, the demand for "Hi-Mach" jet fuel and the demand for premium, high viscosity petroleum fractions suitable for use as base material for high Viscosity Index lubricating oils and premium hydraulic fluids exceeds the supply. In addition, the product value of the latter products compared to the value of the gasoline or naphtha which could be produced from the waxy fractions is in the ratio of 2.2 to 1.

In accordance with the present invention, there is provided a process for hydroisomerizing waxy hydrocarbon mixtures boiling above 204°C. and containing less than 50



percent of oil, by weight, wherein the mixtures are hydroisomerized in the presence of hydrogen and a catalyst composition promoting isomerization under hydroisomerizing conditions, comprising regulating the severity of the conditions by temperature in the range of 232-510°C. and by liquid hourly space velocity in the range of 0.25-5.0 dependent upon the concentration of oil in the mixture. As indicated, it has now been found that the product quality of both produced lube oil and jet fuel is dependent upon (1) the type of charge stock, that is paraffinic charge stock from paraffinic wax and/or paraffinic distillate or non-paraffinic charge stock from microcrystalline wax, such as petrolatum, (2) the amount of liquid hydrocarbon in the waxy charge stock and (3) the degree of conversion to products boiling below 343°C. Thus, the greater the amount of charge stock converted to material boiling below 343°C. per pass, the higher the quality of the jet fuel (boiling range, 190°C. to 260°C.) and the dewaxed 343°C. lube oil. The foregoing is established by the data presented in Tables I and II.

799453

Table I  
(Charge Stock Inspection)

Stock Inspections	A	B	C	D	E
Viscosity					
KV@ 134°C.	3.87	3.91	7.66	8.99	3.85
SSU@ 134°C.	39	39.1	51.3	55.9	39
Color ASTM	10.5	10.5	3.5	3.0	10.5
Melting Point °C.	88	-	103	95	91
Oil Content, % wt.	8.3	19.8	2.0	28.2	0.2
Hydrogen, % wt.	14.59	14.20	14.25	13.74	14.37
Vacuum Distillation					
IBP, °C	380	359	475	482	379
5% vol.	403	386	516	522	424
10% vol.	411	394	529	533	431
20% vol.	419	402	537	541	439
30% vol.	424	407	542	546	444
40% vol.	429	412	547	549	453
50% vol.	435	418	553	554	461
60% vol.	441	423	558	560	469
70% vol.	447	428	562	567	477
80% c	453	434	569	577	487
90% vol.	462	442	579	590	497
95% vol.	469	448	589	601	515
High molecular weight Mass Spectrometer Analysis					
+2 paraffins, mol %	90	-	74	54	96
0 monocyclic paraf fins mol %	7.9	-	22	30	4.2
-2, -4, condensed cyclic paraffins, mol %	1.3	-	2.1	11	-
-6 alkylbenzenes mol %	0.8	-	1.7	4	-
-8, -10 tetralines, indenes, mol %	0.2	-	0.4	1.2	-
-12 naphthalenes mol %	-	-	-	0.2	-
Carbon Range, C-atoms	21-35	-	30-52	26-54	21-36
Average Carbon number	26	-	39	40	26

799453

Table II  
Section A  
(Feed: Stock A - Paraffin Wax - 8.3% Oil)

Run No.	A-1	A-2	A-3
Pressure, psig.	2000	2000	2000
Temperature, °C.	351	331	383
Space Velocity, v/hr/v	2.0	0.5	2.0
5 H <sub>2</sub> -Circulation, SCF/B	2000	2000	2000
Conversion, vol %†	8.7	15.9	44.1
Product, % wt. of charge			
IBP to 191°C.	3.0	6.8	22.1
191°C. to 260°C.	2.1	3.5	10.4
10 260°C. to 343°C.	3.0	4.5	8.9
Conversion wt. %*	8.1	14.8	41.4
343°C. + Bottoms			
Lube Oil Fraction	33.1	30.8	30.8
Unconverted Wax Fraction	58.8	54.4	27.8
15 Total	100.0	100.0	100.0
Inspections			
191° to 260°C. Jet Fuel			
Gravity, °API	51.3	51.8	55.2
Aniline No. °C.	80.4	81.4	79.7
20 Freeze Point, °C.	<-60	<-60	<-60
Luminometer No.	95	100	115
Dewaxed Lube			
Four Point °C.	-4°C.		-4°C
Viscosity, SSU@ 134°C.	37.8	36.8	36.8
25 Viscosity Index (VI)	144		138

†Conversion, vol. % - per cent by volume of charge stock converted to products boiling below 343°C. in one pass.

\*Conversion, wt % - per cent of charge stock to products boiling below 343°C. in one pass.

799453

Table II  
Section B  
(Feed: Stock B - Paraffin Wax - 19.8% Oil)

Run No.	B-1	B-2	B-3	B-4	B-5
Pressure, psig.	500	2000	500	2000	2000
Temperature, °C.	374	398	392	385	409
Space Velocity, 5 V/hr/v	2	2	2	2	2
H <sub>2</sub> -Circulation, SCF/B	2000	2000	2000	2000	2000
Conversion, vol. % <sup>+</sup>	28.2	28.0	59.9	51.5	70
Product, % wt. of Charge					
10 IBP to 191°C.	10.7	9.9	26.9	26.0	38.5
191° to 260°C.	7.3	7.7	13.4	12.9	16.6
260° to 343°C.	8.6	10.0	17.4	10.0	12.7
Conversion wt. %* 343°C. + Bottoms	26.6	27.6	57.7	48.9	67.8
15 Lube Oil Fraction	44.8	55.2	33.9	30.9	25.0
Unconverted Wax Fraction	28.6	17.2	8.4	20.2	7.2
Total	100.0	100.0	100.0	100.0	100.0
Inspections					
20 191° to 260°C. Jet Fuel					
Gravity, °API	49.5	50.3	50.5	52.7	53.5
Aniline No., °C.	70.6	78	73.6	80	80.7
Freeze Point, °C.	<-60	<-60	<-60	<-60	<-60
25 Luminometer No.	62	93	69	107	111
Dewaxed 343°C. + Lube Pour Point, °C.	-9	-12	-12	-12	-9
Viscosity, SSU@ 134°C.	37.2	36.9	36.5	37.4	36.9
30 Viscosity Index (VI)	134	138	126	140	137

<sup>+</sup>Conversion, vol. % - per cent by volume of charge  
stock converted to products boiling below 343°C. in one pass.

35 <sup>\*</sup>Conversion, wt. % - per cent of charge stock converted to  
products boiling below 343°C. in one pass.

799453

Table II  
Section C  
(Feed: Stock C - Petrolatum - 2.0% Oil)

Run No.	C-1	C-2	C-3	C-4
	2000	2000	2000	2000
	289	285	407	418
5	2.0	0.5	2.0	2.0
	2000	2000	2000	2000
	0.5	28.8	34.6	52.0
		15.7	13.0	24.1
		6.4	10.2	12.2
10		4.2	9.2	13.0
	0.5	26.3	32.4	49.3
	2.9	10.1	39.7	29.8
	96.6	63.6	27.9	20.9
15	100.0	100.0	100.0	100.0
<b>Inspections</b>				
	-	52.9	53.5	54.8
	-	79	81	79
20	-	<-60	<-60	<-60
	-	97X	106X	110X
	18.3	15.6	-1	-1
	51.3	43.8	43.4	41.8
25	132	143	150	153

+Conversion, vol. % - per cent by volume of charge stock converted to products boiling below 343°C. in one pass.

29 \*Conversion, wt. % - per cent of charge stock converted to products boiling below 343°C. in one pass.

799453

Table II  
Section D  
(Feed: Stock D - Petrolatum - 28.2% Oil)

Run No.	D-1	D-2	D-3
	2000	2000	2000
Pressure, psig	398	412	424
Temperature, °C.	2.0	2.0	2.0
Space Velocity, v/hr/v	2000	2000	2000
H <sub>2</sub> -Circulation, SCF/B	14.2	26.5	52.7
Conversion, vol %+			
Product, % wt. charge			
IBP to 191°C.	4.0	9.3	21.2
191° to 260°C.	4.2	7.9	13.9
260°C. to 343°C.	5.3	8.0	15.4
Conversion wt. %*	13.5	25.2	50.5
343°C. + Bottoms			
Lube Oil Fraction	44.2	44.6	34.4
Unconverted Wax Fraction	42.3	30.2	15.1
Total	100.0	100.0	100.0
<b>Inspections</b>			
191° to 260°C. Jet Fuel			
	44.2	44.4	48.1
Gravity, °API	63	67	70
Aniline No. °C.	<-60	<-60	<-60
Freeze Point, °C.	55	55	56
Luminometer No.			
Dewaxed 343°C. + Lube			
Pour Point, °C	-3.9	-6.7	-6.7
Viscosity, SSU@ 134°C.	51.5	46.7	43.8
Viscosity Index	121	131	136

+Conversion, vol % - per cent by volume of charge stock converted to products boiling below 343°C. in one pass.

\*Conversion wt. % - per cent of charge converted to liquid products boiling below 343°C. in one pass.



799453

Table II  
Section E  
(Feed: Stock E - Paraffin Wax - 0.2% Oil)

Run No.	E-1	E-2	E-3	E-4	E-5
Pressure, psig.	250	250	250	2000	2000
Temperature, °C.	260	299	319	357	373
Space velocity, 5 V/hr/v	2	2	2	2	2
H <sub>2</sub> -Circulation, SCF/B	2000	2000	2000	2000	2000
Conversion, vol. % <sup>+</sup>	12.2	30.0	52.1	17.2	53.1
Product, %wt. Charge					
10 IBP to 191°C.	3.2	8.6	21.4	2.6	20.3
191°C. to 260°C.	3.5	6.6	13.9	6.1	16.0
260°C. to 343°C.	4.9	13.3	14.6	7.7	14.7
Conversion, wt.% <sup>*</sup>	11.6	28.5	49.9	16.4	51.0
343°C. + Bottoms					
15 Lube Oil Fraction	44.6	46.6	38.4	53.8	39.8
Unconverted Wax					
Fraction	43.8	24.9	11.7	29.8	9.2
Total	100.0	100.0	100.0	100.0	100.0
Inspections, 191-260°C.					
20 Jet Fuel Gravity, °API	53.7	55.0	54.4	55.0	55.1
Aniline No. °C.	84	82	82	83.5	82.2
Freeze Point °C.	←60	←60	←60	←60	←60
Luminometer No.	123	116	112	130	122
25 Inspections: Dewaxed 343°C. + Lube Oil					
Four Point, °C.	-6.7	-6.7	-6.7	-6.7	-6.7
Color, ASTM	2.5	LI.0	LI.0	LO.5	LO.5
30 Viscosity SSU@ 134°C.	37.8	37.3	37.3	37.4	37
VI	149	138	138	142	137

<sup>+</sup>Conversion, vol.% - per cent by volume of charge stock converted to products boiling below 343°C. in one pass.

34 <sup>\*</sup>Conversion, wt. % - per cent of charge stock converted to products boiling below 343°C. in one pass.

The data presented in Table II, Sections A, B, C, D and E for luminometer number have been plotted in Figure I. It will be observed that, for paraffinic and non-paraffinic charge stocks containing eight per cent or more of oil, the luminometer number increases with increased conversion of charge stock to products boiling below  $343^{\circ}\text{C}$ . However, the data for the luminometer for charge stock D containing 28.2 per cent of oil establishes that when the charge stock contains in excess of 23 per cent of oil the luminometer number of the 191-260 $^{\circ}\text{C}$ . jet fuel cannot be raised to 100. The data plotted in Figure I for charge stock containing practically no oil (as 0.2 per cent) establishes that at a pressure of 250 psig or at a pressure of 2000 psig the more severe the reaction conditions (that is, the greater the conversion to products boiling below  $343^{\circ}\text{C}$ ), the lower the luminometer number of the 191-260 $^{\circ}\text{C}$  jet fuel. However, it will be observed that at a given conversion the higher the pressure the higher the luminometer number for stocks containing substantially no oil, (0.2 per cent oil).

From the curves in Figure I for charge stocks containing 2 per cent to 19.8 per cent of oil curves have been drawn and presented in Figure II to more clearly illustrate the anomalous effect of the increasing concentration of oil. With increasing concentration of oil from 2 per cent to 8 per cent, the conversion must be reduced to maintain a target luminometer number.

As the concentration of oil in the charge stock increases from 8 per cent to 23 per cent, the conversion must

be increased to maintain the target luminometer number of the  
191-260°C. jet fuel. The curves for constant luminometer  
number presented in Figure II establish that, while the  
conditions to produce jet fuel having higher luminometer  
5 number must be more severe the higher the luminometer number,  
to maintain a target luminometer number, the severity of the  
hydroisomerization conditions must be reduced when the charge  
stock contains between 0.2 to 7.0 per cent of oil and the  
severity of hydroisomerization conditions increased as the  
10 concentration of oil (as defined herein) increases from 7  
per cent to 23 per cent. (Severity being measured by the  
conversion to products boiling below 343°C).

With a consideration of the curves for the correlation  
of Viscosity Index and per cent conversion to products boiling  
15 below 343°C. presented in Figure III, other controlling  
factors are visualized. Pressure during hydroisomerization and  
the character of the charge stock are recognized as effective  
variables.

With a paraffinic charge stock containing 0.2 per cent  
20 of oil (as defined herein), it will be observed that pressure  
has little, if any, effect upon the Viscosity Index. Thus,  
at a hydroisomerization pressure of 250 psi and at a hydro-  
isomerization pressure of 2000 psi and conversions of 52 and  
53 per cent of the charge stock to products boiling below  
25 343°C. the Viscosity Index is 137 and 138. On the other hand,  
while at 2000 psi the oil content in the range of 8 to 20  
per cent has little, if any, effect upon the Viscosity Index  
when the hydroisomerization reaction is carried out at 2000

psi, at an operating pressure of 500 psi the oil content of the charge stock has a marked effect upon the Viscosity Index. It will also be observed that the Viscosity Index of the Lube Oil Fraction of the hydroisomerization products boiling above 343°C. when the hydroisomerization operation is carried out at 2000 psi, is substantially the same when a paraffinic stock containing 0.2 to 20 per cent of oil is treated. This is established by the numerical values for Viscosity Index read from the curves of Figure III.

Charge Stock Type	Oil Content %	Conversion to Products Boiling <343°C.	Viscosity Index Lube Oil Fraction Products Boiling >343°C.
Paraffinic	0.2	30	138
Paraffinic	19.8	29	138
Paraffinic	8.3	30	138

A paraffinic charge stock containing about 20 per cent of oil illustrates the effect of operating pressure on the Viscosity Index of the Lube Oil fraction of the conversion products boiling above 343°C. as established by the data presented in the following tabulation:

	Charge Stock Type	Oil Content %	Pressure psi	Conversion to Products Boiling < 343°C.	Viscosity Index Lube Oil Fraction Products Boiling > 343°C.
5	Paraffinic	19.8	2000	28	138
	Paraffinic	19.8	500	28	134
	Paraffinic	19.8	2000	60	137
10	Paraffinic	19.8	500	60	126

With a non-paraffinic charge stock, such as petrolatum containing 2 per cent of oil and a non-paraffinic charge stock, illustrated by petrolatum containing 28 per cent of oil, it will be observed that, at an operating pressure of 2000 psi and at a conversion of about 25 per cent to products boiling below 343°C., the Viscosity Index of the Lube Oil Fraction of the products boiling above 343°C. varies; the Viscosity Index of the Lube Oil Fraction produced from the charge stock containing 2 per cent of oil is 144.5, while that of the Lube Oil Fraction produced from the charge stock containing 28 per cent of oil is 130. At a conversion of 50 per cent to products boiling below 343°C., the Viscosity Index of the Lube Oil Fraction produced from the charge stock containing 2 per cent oil is 152.5, while the Viscosity Index of the Lube Oil fraction produced from charge stock containing 28 per cent of oil is 135.5. These statements are summarized in the following tabulation:

799453

	<u>Stock Type</u>	<u>% Oil</u>	<u>% Conversion</u>	<u>VI of Lube Oil Fraction</u>
	Non-paraffinic	2	25	144.5
	"	28	25	130.0
5	"	2	50	152.5
	"	28	50	135.5

From the foregoing, the following general characteristics of the effect of charge stock type and the oil content thereof can be obtained: (1) for paraffinic charge stocks  
10 containing 0.2 to 7 per cent of oil, both luminometer number and Viscosity Index decrease with increased severity as measured by the per cent of the charge stock converted to products boiling below 343°C; (2) for paraffinic stocks  
15 containing oil in the range of 8 to 23 per cent, the luminometer number increases and the Viscosity Index decreases with increased severity as measured by the per cent of the charge stock converted to products boiling below 343°C.;  
20 (3) for non-paraffinic charge stocks containing oil in the range of 2 to 23 per cent of oil, both Luminometer number and Viscosity Index increase with increased severity; (4)  
for non-paraffinic stocks containing oil in excess of 23 per cent, as 28 per cent, the luminometer number is static, while the Viscosity Index increases with increased severity;  
25 and (5) operating pressure in the range of 100 to 2000 psi has little or no effect upon the Viscosity Index of the Lube Oil Fraction of conversion products boiling above 343°C.  
produced from paraffinic stocks containing less than 8 per cent of oil, but has marked effect upon the Viscosity Index

of the Lube Oil Fraction of products produced from paraffinic stocks containing 8 per cent or more of oil at conversions in excess of 25 per cent.

It follows that the severity of hydroisomerization conditions must be coordinated with: (1) the type of charge stock, that is, paraffinic and non-paraffinic; (2) the quantity of oil in the charge stock, that is the concentration of hydrocarbon boiling above 343°C. which is liquid after solvent dewaxing at -17.8°C.; (3) the quality of the jet fuel, 191-260°C. fraction of the liquid products boiling below 343°C.; and (4) the quality of the lube oil fraction, the fraction of the bottoms product (boiling above 343°C.) liquid after solvent dewaxing at -17.8°C. or lower, as -53.9°C. or lower.

Charge stocks are characterized as paraffinic or non-paraffinic. Charge stocks A, B, and E are illustrative of paraffinic stocks, while charge stocks C and D are illustrative of non-paraffinic stocks. Thus, paraffinic stocks contain at least 80 mol per cent of +2 paraffins and not more than 20 mol per cent of monocyclic paraffins. On the other hand, non-paraffinic charge stocks contain less than 80 mol per cent of +2 paraffins and 20 mol per cent or more of monocyclic paraffins. The hydrogen or hydrogen-containing gas can be used in a once-through manner, that is no recycle of the reaction products boiling below C<sub>4</sub> hydrocarbons. Yet, in many operations local conditions will require recycle of the gas comprising hydrogen and C<sub>1</sub> to C<sub>3</sub> hydrocarbons for economic industrial operation. Accordingly, the flowsheets diagrammed in Figures IV and V provide the recycle of the C<sub>3</sub>

and lighter hydrocarbons and hydrogen separated from the C<sub>4</sub> and heavier hydroisomerization products. It is to be understood that, although not illustrated in flowsheets Figures IV and V, the C<sub>3</sub> and C<sub>4</sub> hydrocarbons present in the effluent  
5 of the hydroisomerization zone can be recovered for use as "LPG" (liquid petroleum gas) or the C<sub>1</sub> to C<sub>3</sub> hydrocarbons can be upgraded as by conversion into unsaturated hydrocarbons such as acetylene, ethylene, and propylene, respectively.

10           The flowsheets, Figures IV and V illustrate three embodiments of the present invention to wit: "Embodiment A" in which a charge stock containing less than 25 per cent of hydrocarbon liquid after solvent dewaxing at -17.8°C., that is having a pour point of -6.7°C., is converted to "Hi-Mach"  
15 jet fuel (luminometer number at least 100), and naphtha having an EBP (end boiling point) of 191°C. The bottoms boiling above 260°C. are recycled to extinction. The feed or charge stock in "Embodiment A" is at least one of slack wax, petrolatum, or other waxy hydrocarbon mixtures having  
20 an IBP (initial boiling point) higher than 204°C. and containing less than 25 per cent of oil. "Embodiment B" is illustrative of the hydroisomerization of waxy charge stock containing less than 50 percent of oil having an IBP of at least 343°C., as slack wax petrolatum and the like,  
25 to "Hi-Mach" jet fuel, premium Diesel fuel, and high Viscosity Index (HI-VI) automotive lubricating oils having a pour point not higher than -6.7°C. and a VI above presently commercially available automotive lube oils



of usual quality as measured by the VI. In "Embodiment B" the wax separated from the 343°C. + bottoms in a conventional dewaxing operation at -17.8°C. is recycled to extinction.

In "Embodiment C" waxy hydrocarbon mixtures containing less than 5 per cent of oil (as finished paraffin wax) is hydroisomerized; the waxy 343°C + bottoms is dewaxed at -17.8°C., and the dewaxed oil is dewaxed a second time at considerably lower temperature to provide twice-dewaxed oil having a pour point not higher than -40°C. The twice-dewaxed oil is  
10 suitable for use as fluid in supersonic aircraft hydraulic systems and a waxy "Hi-VI" automotive lube.

Embodiment A  
(maximizing jet fuel)

A charge stock containing not more than 25 per cent oil, as slack wax, petrolatum, or other waxy hydrocarbon mixture having an initial boiling point (IBP) above 204°C is drawn as shown in Figure IV from a source (not shown) through pipe 10 by pump 11. Pump 11 discharges the charge stock into pipe 12 through which the charge stock flows to  
20 heat exchanger 13. When sufficient charge stock has been hydroisomerized to produce distillation bottoms boiling above 260°C., the bottoms, flowing from distillation tower 25 through pipe 45 to the suction side of pump 47 (valve 46 open, valve 56 closed), is discharged by pump 47 into pipe 48 at a pressure in excess of that in pipe 12, and is admixed with the charge stock at some point in pipe 12

intermediate to pump 11 and indirect heat exchanger 13 to  
form hydroisomerization feed. The hydroisomerization feed,  
hereinafter designated feed, flows through pipe 12 to  
indirect heat exchanger 13 where the feed is in heat transfer  
5 relation with the total effluent of the hydroisomerization  
reaction zone 20 flowing from indirect heat exchanger 15  
through conduit 22. From indirect heat exchanger 13 the feed  
flows through pipe 14 to indirect heat exchanger 15 where  
the feed is in heat transfer relation with the total  
10 effluent of the hydroisomerization reaction zone 20 flowing  
therefrom through conduit 21.

From indirect heat exchanger 15, the feed flows  
through pipe 16 to coil 17 in heater 18. In heater 18 the  
feed is heated to a temperature sufficient to provide at  
15 the inlet of hydroisomerization reaction zone 20, a hydro-  
isomerization temperature in the range of 260°C. to 482°C.  
From heater 18 the heated feed flows through conduit 19 to  
the inlet of hydroisomerization reaction zone 20. At a  
point in conduit 19 intermediate to heater 18 and reaction  
20 zone 20, hydrogen flowing from a source not shown through  
conduit 49 under control of valve 50 (valves 32 and 33  
closed) to conduit 31, or hydrogen-containing gas flowing  
from accumulator 30 or as otherwise described hereinbefore  
through conduit 31 (valves 32 or 33 open, valve 50 closed)  
25 and thence to the suction side of compressor 51 and thence  
through conduit 52 to conduit 19, is admixed with the  
aforesaid feed to form charge mixture comprising 1 to 25 mols  
of hydrogen per mol of feed.

The charge mixture flows through conduit 19 to reaction zone 20. Generally, one reactor is sufficient since the hydroisomerization reaction is exothermic. However, more than one reactor can be used in a hydroisomerization reaction zone.

The conditions in the hydroisomerization zone are in the limits set forth hereinafter; the severity of the hydroisomerization conditions being regulated in accordance with the oil content and type (paraffinic or naphthenic) of the feed and the quality of the major product.

	<u>Broad</u>	<u>Preferred</u>
Hydrogen Pressure, psi	at least 100	200 to 2000
Temperature, °C.	232-510	260-482
Liquid Hourly Space		
Velocity, v/hr/v	0.25 to 5.0	1 - 3
H <sub>2</sub> -Circulation SCF/B	300 to 10,000	1000-5000
Catalyst	Particle form solid hydroisomerization	0.6 wt.% Pt. 4.5 wt.% F. 0.36 wt. % Cl, on alumina

It is preferred to use platinum-group metal hydroisomerizing catalysts. Such catalysts comprise one or more of platinum, palladium, and nickel, preferably on a support comprising eta alumina and especially gamma alumina or a non-alkaline base including bentonite, barite and faujasite containing chlorine and/or fluorine. Particularly preferred is a platinum group metal hydroisomerizing catalyst comprising 0.1 to 1.0 per cent by weight of platinum, and halogen in the range of 0.1 to 10 per cent by weight, on an alumina support. Halogen as used herein is limited to fluorine and chlorine; and, dependent upon the method of preparation with

either hydrofluoric acid or hydrochloric acid or a combination of the two acids, the catalyst contains fluorine or chlorine or both in the indicated percentage range.

5 The charge mixture flows downwardly through reaction zone 20 in contact with the particle-form solid hydroisomerization catalyst to the reaction zone outlet and conduit 21. The reaction zone effluent flows through conduit 21 to indirect heat exchanger 15, thence through conduit 23 (valve 24 open, valve 53 closed) to fractionation tower 25.

10 As illustrated, separation of the reaction zone effluent takes place at pressures in the range of 25 to 50 psig. The reaction zone pressure is reduced to distillation pressure (25 to 50 psig) by pressure reducing valve 24. The single fractionation tower 25 can be replaced with a plurality  
15 of fractionation towers. In addition, a high-pressure separator or flash drum can be used up-stream of the distillation zone to separate C<sub>4</sub> and lighter hydrocarbons and hydrogen from the C<sub>5</sub> and heavier hydrocarbons of the reaction zone effluent. The pressure on the C<sub>5</sub> and heavier fraction  
20 of the reaction zone effluent can be reduced, and only the C<sub>5</sub> and heavier reaction zone product can be introduced into the distillation zone.

As illustrated, the fractionation zone comprises fractionating tower 25, a reboiler represented by pump and  
25 heater 26, coolers and receivers. In "Embodiment A" the reaction zone effluent flows through conduit 23 from reducing valve 24 at a pressure up to 50 psig and a temperature at which hydrocarbons boiling below 260°C. at atmospheric

pressure are volatile. The reaction zone effluent is fractionated into (1) hydrogen and C<sub>1</sub> to C<sub>4</sub> hydrocarbons, (2) C<sub>5</sub> to 191°C. end boiling point (EBP) naphtha, (3) 191-260°C. "Hi-Mach" jet fuel, and (4) bottoms boiling above 260°C.

5           The C<sub>4</sub> and lighter, including hydrogen, is taken overhead through conduit 27 to cooler 28 where the overhead is cooled to a temperature at which, at the existing pressure, the C<sub>4</sub> hydrocarbons condense. The cooled overhead flows from cooler 28 through conduit 29 to accumulator 30.  
10       In accumulator 30 the uncondensed C<sub>3</sub> and lighter hydrocarbons separate and flow therefrom through conduit 31 and conduit 54 (valve 32 closed; valve 55 open) to the refinery fuel system, recovery of hydrogen, or other hydro processes; or, with valve 55 closed and valve 32 open (valve 33  
15       closed), to compressor 51 for recycle to reaction zone 20.

          The condensed overhead C<sub>4</sub> and heavier flows from accumulator 30 through pipe 34 to recovery of C<sub>4</sub> and heavier. A portion of the C<sub>4</sub> and heavier flowing through pipe 34 is diverted under control of valve 36 through pipe 35 to the  
20       suction of pump 37. Pump 37 discharges the C<sub>4</sub> and heavier into pipe 38, through which the C<sub>4</sub> and heavier flows to fractionation tower 25 for use as a reflux.

          A fraction boiling in the range of C<sub>5</sub> to 191°C. and designated hydronaphtha, is taken in the usual manner (with  
25       or as illustrated without reflux) through pipe 39 to cooler 40 where the C<sub>5</sub> to 191°C. is cooled to a temperature at which, under the existing pressure, the C<sub>5</sub> to 191°C.

hydronaphtha is condensed. The condensed hydronaphtha flows from cooler 40 through pipe 41 to hydronaphtha storage.

5 A "Hi-Mach" jet fuel (luminometer number at least 100), is taken in the usual manner (with or as illustrated without reflux) through pipe 42. The "Hi-Mach" jet fuel flows through pipe 42 to cooler 43 where the "Hi-Mach" jet fuel is condensed. The condensed "Hi-Mach" jet fuel flows from condenser 43 through pipe 44 to "Hi-Mach" jet fuel storage.

10 The bottoms (reaction zone effluent boiling above 260°C.) flows from fractionator 25 through pipe 45 under control of valve 46 (valve 56 closed) to the suction side of pump 47. Pump 47 discharges the unconverted waxy bottoms (recycle bottoms) into pipe 48 for admixture with the charge  
15 stock to form the hydroisomerization feed.

"Embodiment A" provides a means for producing "Hi-Mach" jet fuel, but no lube oil with recycle of the waxy 260°C. + bottoms to extinction. Consequently, there is no need to compromise on the severity of the conditions in the  
20 hydroisomerization reaction zone. The temperature and space velocity are regulated to provide the maximum yield of jet fuel having a luminometer number of at least 100. The temperature and space velocity accordingly are regulated dependent upon the oil content of the feed and the lumino-  
25 meter number of the produced "Hi-Mach" jet fuel.

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EMBODIMENT B  
(Maximize "Hi-Mach" Jet Fuel or Maximize  
"Hi-VI" Automotive Lube Oils)

As illustrated in Figures IV and V, the combination of operations in "Embodiment B" are coordinated to product (1) "Hi-Mach" jet fuel, (2) premium Diesel fuel and (3) above conventional quality, "Hi-VI" automotive lubricating oil.

The charge stock is a waxy mixture of hydrocarbons containing less than 50 per cent of oil, hydrocarbon liquid at -17.8°C. after conventional dewaxing as to a pour point of -6.7°C. The charge stock flows as described in "Embodiment A" through the heat exchangers, heater and reaction zone at a temperature, a pressure, a space velocity and hydrogen circulation in the limits set forth hereinbefore. However, the recycle bottoms is the unconverted wax separated in a conventional manner, as by using methyl-ethyl ketone and benzol at -17.8°C. The total hydroisomerization reaction zone effluent flows from reactor 20 through conduit 21 to indirect heat exchanger 15, thence through conduit 22 to indirect heat exchanger 13 and from exchanger 13 through conduits 23 and H-57 and F 57 (valve 24 closed; valve 53 open) to fractionation tower 58.

Fractionation tower 58 and a reboiler of any suitable construction (indicated by pipes 59 and 60, pump 61, and heater 62) can be replaced by a plurality of fractionation towers and auxiliary equipment, or a high-pressure separator can be interposed between heat exchanger 13 and fractionation tower or towers 58 as described hereinbefore. As illustrated, the total reactor effluent at a reduced pressure in the range of 10 to 50 psig enters fractionator 58 at a tempera-

ture at which hydrocarbons boiling below 343°C. under normal conditions are volatile.

An overhead comprising C<sub>4</sub> and lighter hydrocarbons and hydrogen is taken through conduit 63. The overhead  
5 flows through conduit 63 to cooler 64. In cooler 64 the overhead is cooled to condense C<sub>4</sub> and heavier hydrocarbons. The cooled overhead flows from cooler 64 through conduit 65 to accumulator 66. In accumulator 66 the hydrogen and C<sub>1</sub>  
10 to C<sub>3</sub> hydrocarbons, uncondensed overhead, separate and flow therefrom through conduit F-67 and H-67 [(Fig. IV) valve 33 open; valve 32 closed] to compressor 51 (Fig. IV) for use as recycle gas. The separated C<sub>4</sub> and heavier flow from accumulator 66 through pipe 68 to storage. A portion  
15 of the C<sub>4</sub> and heavier is diverted through pipe 69 under control of valve 70 to the suction side of pump 70a. Pump 70a discharges the diverted portion of the C<sub>4</sub> and heavier into pipe 71, through which the aforesaid diverted portion flows to fractionator 58 for use as reflux.

In a conventional manner without reflux as illustrated  
20 or with reflux, a C<sub>5</sub> to 191°C. hydronaphtha flows from fractionator 58 through pipe 72 to cooler 73 where the C<sub>5</sub> to 191°C. hydronaphtha is condensed. The condensed hydronaphtha flows from cooler 73 through pipe 74 to storage.

In the usual manner without reflux as illustrated  
25 or with reflux, a "Hi-Mach" jet fuel fraction, boiling range 191-260°C. flows from fractionator 58 through pipe 75 to cooler 76. In cooler 76 the "Hi-Mach" jet fuel is condensed, the condensed "Hi-Mach" jet fuel flows therefrom to storage through pipe 77.



A third intermediate fraction is taken in the usual manner. The third intermediate fraction is a premium Diesel fuel boiling in the range of 260-343°C. and has a cetane number in the range of 60 to 90; this flows from fractionator 58 through pipe 78 to cooler 79. The premium Diesel fuel is condensed in cooler 79 and flows therefrom through pipe 80 to storage.

The bottoms (a waxy mixture of hydrocarbons boiling above 343°C.) flows from fractionator 58 through pipe 81 to means such as cooler 82 and other heat exchangers. In the heat exchanger system indicated by cooler 82, the 343°C.+ bottoms is cooled to provide, when mixed with suitable dewaxing solvent (as methyl-ethyl ketone-benzol mixture or methyisobutyl ketone mixture), a dewaxing temperature to provide a -6.7°C. pour point oil for the purpose of removing unconverted wax. The cooled 343°C. bottoms are dewaxed in a suitable manner, for example, at a temperature not higher than -6.7°C. in a dewaxing unit indicated by 84. The dewaxing operation is conventional. Under the normal dewaxing conditions to produce a lube oil having a pour point of -6.7°C. or lower, "Hi-VI" lubricating oil suitable as a base for premium automotive lubricating oil flows from dewaxing unit 84 through pipe 85 (valve 86 open; valve 88 closed) to storage, and/or compounding.

The unconverted wax flows from dewaxing unit 84 through pipe F-87 and H-87 to pipe 45 (valve 56 open; valve 46 closed; Fig. IV) and thence to the suction side of pump 47

for use as recycle in the feed to the hydrosomerization reactor 20. The unconverted wax preferably is recycled to extinction.

In "Embodiment B", dependent upon local marketing conditions, the severity of the hydroisomerization conditions -- as oil content of the charge stock, hydrogen operating pressure, space velocity and reaction temperature as measured at the inlet of the reaction zone -- is regulated as described herein to maximize the production of "Hi-Mach" jet fuel depending upon the luminometer of the jet fuel produced and/or to maximize the production of "Hi-VI" automotive lube oils.

EMBODIMENT C  
(Maximizing Premium Product)

The severity of hydroisomerization conditions as measured by hydrogen pressure, space velocity and temperature in the ranges set forth hereinbefore is regulated to maximize "Hi-Mach" jet fuel or hydraulic fluid particularly for supersonic aircraft or aircraft turbine engines dependent upon the luminometer numbers, the stability in the range of -53,9°C. to 371°C. and viscosity index, respectively, and dependent upon the oil content of the charge stock.

The feed is substantially any waxy hydrocarbon mixture containing less than 5 per cent of oil, such as microcrystalline wax, partially refined or refined paraffin wax.

As described in conjunction with the discussion of "Embodiments A and B" hereinbefore, the charge stock flows together with unconverted wax through the heat exchangers,

heater and reaction zone 20. The reaction zone effluent flows through conduit 21, heat exchanger 15, conduit 22, and heat exchanger 13 to conduit 23. With valve 24 closed and valve 53 open, the total reactor effluent flows through  
5 conduit H-57 and F-57 to one or more fractionators 58, or to a high pressure separator and one or more fractionators as described hereinbefore.

In "Embodiment C" as illustrated, the total effluent enters the separation system at a temperature at which hydro-  
10 carbons boiling up to 343°C.+ are volatile. Separation is as described in "Embodiment B" except that, after dewaxing the 343°C.+ bottoms in a conventional manner to produce an oil having a pour point not higher than -6.7°C., the partially dewaxed oil is deep dewaxed in a suitable manner,  
15 for example by using methyl-ethyl ketone-benzene or preferably 100 per cent methyl-isobutyl ketone as the solvents, at a temperature not higher than about -53.9°C.

Thus, the total reactor effluent flows from heat exchanger 13 (Fig. IV) through conduits 23, H-57, F-57 to  
20 fractionator 58 (Fig. V). In fractionator 58 an overhead is recovered and treated as previously described in "Embodiment B." Three intermediate fractions or side streams, namely a C<sub>5</sub> to 191°C. hydronaphtha, a 191-260°C. "Hi-Mach" jet fuel, and a premium Diesel fuel, are recovered as previously  
25 described in "Embodiment B."

In "Embodiment C" however, the 343°C.+ bottoms flowing from fractionator 58 through pipe 81 are dewaxed as stated hereinbefore to produce partially dewaxed oil and

unconverted wax. The unconverted wax is recycled through pipe F-87 and H-87 (valve 56 open; valve 46 closed) to pipe 45 as described in "Embodiment B." The partially dewaxed oil flows from the dewaxing unit indicated by 84 through pipe 89 (valve 88 open; valve 86 closed) to a suitable dewaxing operation indicated by 90. In the dewaxing unit indicated by 90 the partially dewaxed oil is again dewaxed using the described solvents at a temperature not higher than -53.9°C. Two products are obtained from this "deep" dewaxing, to wit: (1) an oil suitable for use as the fluid in the hydraulic systems of supersonic aircraft and being stable in the range of -45.6°C. to 371°C. or for use as an aircraft turbine engine oil, and (2) "high VI" automotive oils and turbine oils far superior to the presently available "Hi-VI" automotive and turbine oils, as is manifest by the following comparison of the properties of the novel "Hi-VI" oils produced as hereindescribed and conventional "Hi-VI" automotive oils.

Conventional Automotive Base Stocks and Turbine Lubes	Present Novel Base Stocks
Viscosity Index 90 - 105	Viscosity Index 125 - 155

As in "Embodiments A and B", the severity of the hydroisomerization reaction conditions is regulated dependent upon the concentration of oil in the waxy charge and the luminometer number of the jet fuel or the VI of the fractions recovered from the 343°C.+ bottoms.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. The process for hydroisomerizing waxy hydrocarbon mixtures boiling above  $204^{\circ}\text{C}$ . and containing less than 50 percent of oil, by weight, wherein the mixtures are hydroisomerized in the presence of hydrogen and a catalyst composition promoting isomerization under hydroisomerizing conditions, comprising regulating the severity of the conditions by temperature in the range of  $232-510^{\circ}\text{C}$ . and by liquid hourly space velocity in the range of 0.25-5.0 dependent upon the concentration of oil in the mixture.

2. The process in accordance with Claim 1 wherein the severity is regulated to produce a jet fuel fraction boiling in the range of  $191-260^{\circ}\text{C}$ . and having a luminometer number of at least 100.

3. The process in accordance with either Claim 1 or 2 wherein the severity of the conditions is increased with the increase in oil content of mixtures containing in excess of 8 percent of oil.

4. The process in accordance with either Claim 1 or 2 wherein the severity of the conditions is not increased with the increase in oil content of mixtures containing not more than 8 percent of oil.

5. The process in accordance with claim 1 wherein liquid product is recovered and is separated into jet fuel boiling  $191-260^{\circ}\text{C}$ ., diesel fuel boiling  $260-343^{\circ}\text{C}$ . and bottoms boiling above  $343^{\circ}\text{C}$ .

6. The process in accordance with Claim 5 wherein bottoms are recycled to extinction.

7. The process in accordance with Claim 5 wherein the bottoms are cooled to  $-6.7^{\circ}\text{C}$ . and a high viscosity index lubricant is recovered.

8. The process in accordance with Claim 1 wherein the severity is regulated to produce a lubricating oil having a Viscosity Index of at least 110.

9. The process in accordance with Claim 1 wherein the severity is regulated to produce a Diesel fuel boiling in the range of 260-343°C. and having a cetane number of at least 60.

10. The process in accordance with claim 1 wherein the severity is regulated to product (1) a jet fuel boiling in the range of 191-260°C, and having a luminometer number of at least 100, (2) a diesel fuel boiling in the range of 260-343°C. and having a cetane number of at least 60, (3) a hydraulic oil stable in the range of -18°C. to 371°C., boiling above 343°C, and having a Viscosity Index of at least 100, and (4) an automotive lubricating oil boiling above 343°C. and having a Viscosity Index of at least 125.

11. The process in accordance with claim 1 or claim 10 wherein the catalyst is a halogen-promoted platinum group metal supported on alumina.

12. The process in accordance with claim 1 or claim 10 wherein the catalyst is halogen-promoted platinum

supported on alumina, in which the platinum content is 0.6 percent and the halogen content is 5-6 percent of which 90 percent is fluorine.

13. The process in accordance with claim 1 or claim 10 wherein the catalyst is halogen-promoted platinum supported on alumina, in which the platinum content is 0.6 percent, fluorine content is 4.5 percent and chlorine content is 0.36.

14. A process for the production of a liquid product comprising jet fuel which comprises selectively cracking and hydroisomerizing a waxy hydrocarbon mixture containing less than about 50 weight percent oil combined therewith in the presence of hydrogen and a catalyst composition promoting isomerization at a temperature in the range of from about 450° to about 950° F., to obtain a conversion of said waxy hydrocarbon mixture to products boiling below about 650°F. of from about 25 percent to about 70 percent, recovering a liquid product from said selective cracking step, separating said liquid product into a jet fuel boiling in the range of from about 375° to about 500° F., a diesel fuel boiling in the range of from about 500° to about 650°F. and a bottoms fraction boiling above about 650°F. and recycling said bottoms fraction to said selective cracking step.

15. A process for converting waxy constituents containing less than 50 percent weight of a distillate oil combined therewith which comprises contacting the waxy constituents with hydrogen in the presence of a halogen promoted platinum containing catalyst under temperature and pressure conditions to obtain greater than 25 percent conversion of said waxy constituents to product material boiling below about 650°F. and recycling to extinction product material boiling above about 650°F.

16. A process for upgrading a waxy hydrocarbon mixture containing less than about 50 percent of distillate oil to lower boiling products comprising jet fuels and high VI lube oils which comprises contacting the waxy material combined with a hydrogen in the presence of a hydroisomerizing catalyst under hydroisomerizing conditions to obtain a conversion of said

waxy hydrocarbon to products boiling below about 650°F. of from about 25 percent to about 70 percent, separating the hydroisomerized product under conditions to obtain a jet fuel boiling range product and a liquid product boiling above about 650°F., cooling the liquid product boiling above about 650°F. to a temperature below about 20°F. and recovering a high viscosity index lube oil from the product of the cooling step.

17. A process for converting a paraffin wax containing from about 8 to about 50 percent oil combined therewith which comprises contacting said paraffin wax with hydrogen in the presence of a halogen promoted platinum type catalyst under hydroisomerizing conditions sufficient to obtain a conversion of said combined paraffin wax and oil to products boiling below about 650°F. of from about 25 percent to about 70 percent to obtain jet fuel boiling range materials and a liquid product boiling above about 650°F. and continuously recycling the liquid product to said hydroisomerizing.

18. In the hydroisomerization of waxy hydrocarbon mixtures boiling above 400°F. selected from a member of the group consisting of petrolatum and microcrystalline wax and containing less than 50 percent of oil at a pressure in the range of 200 to 2000 psig in the presence of about 300 to 10,000 s.c.f. of hydrogen per barrel of said waxy hydrocarbon mixture and in the presence of catalyst composition promoting isomerization the improvement which comprises regulating the severity of reaction conditions as measured by temperature in the range of about 450° to about 950°F. and the liquid hourly space velocity in the range of .25 to 5.0 to convert said waxy hydrocarbon to products boiling below about 650°F. in an amount of between about 25 percent and about 70 percent to produce jet fuel fraction boiling in the range of about 375° to about 500°F. and having a luminometer number of at least 100.

19. The process set forth in claim 18 wherein the catalyst comprises halogen promoted platinum-group metal on alumina support.

20. The process set forth in claim 19 wherein the catalyst



comprises about 0.6 weight percent of platinum, about 4.5 weight percent of fluorine, and about 0.36 weight percent of chlorine on alumina.

21. The process set forth in claim 19 wherein the catalyst comprises about 0.6 weight percent of platinum and about 5 to 6 weight percent of halogen of which at least about 90 percent is fluorine on alumina.

22. In the hydroisomerization of waxy hydrocarbon mixtures boiling above  $400^{\circ}\text{F}$ . and containing less than 50 percent of oil at a pressure in the range of 200 to 2000 psig in the presence of about 1000 to 5000 s.c.f. of hydrogen per barrel of said waxy hydrocarbon mixture and in the presence of catalyst composition promoting isomerization the improvement which comprises regulating the severity of reaction conditions as measured by temperature in the range of about 500 to about  $900^{\circ}\text{F}$ . and the liquid hourly space velocity in the range of 1.0 to 3.0 to produce products boiling below  $650^{\circ}\text{F}$ . in amount at least 25 percent of the feed and dependent upon the concentration of oil in the aforesaid waxy hydrocarbon mixture to produce jet fuel boiling in the range of about  $375^{\circ}$  to about  $500^{\circ}\text{F}$ . and having a luminometer number of at least 100.

23. The process set forth in claim 22 wherein the isomerization catalyst comprises about 0.6 weight percent of platinum and about 5 to 6 percent by weight of halogen of which at least 90 percent is fluorine, wherein for a jet fuel having a predetermined luminometer number of at least 100, and a feed containing in excess of 8 percent of oil, the severity of reaction conditions is increased with the increase in oil content.

24. In the hydroisomerization of waxy hydrocarbon mixtures boiling above  $400^{\circ}\text{F}$ . selected from a member of the group consisting of petrolatum and microcrystalline wax and containing less than 50 percent of oil at a pressure in the range of 200 to 2000 psig in the presence of about 300 to 5000 s.c.f. of hydrogen per barrel of said waxy hydrocarbon mixture and in the presence of catalyst composition promoting isomerization the improvement which comprises regulating the severity of reaction conditions

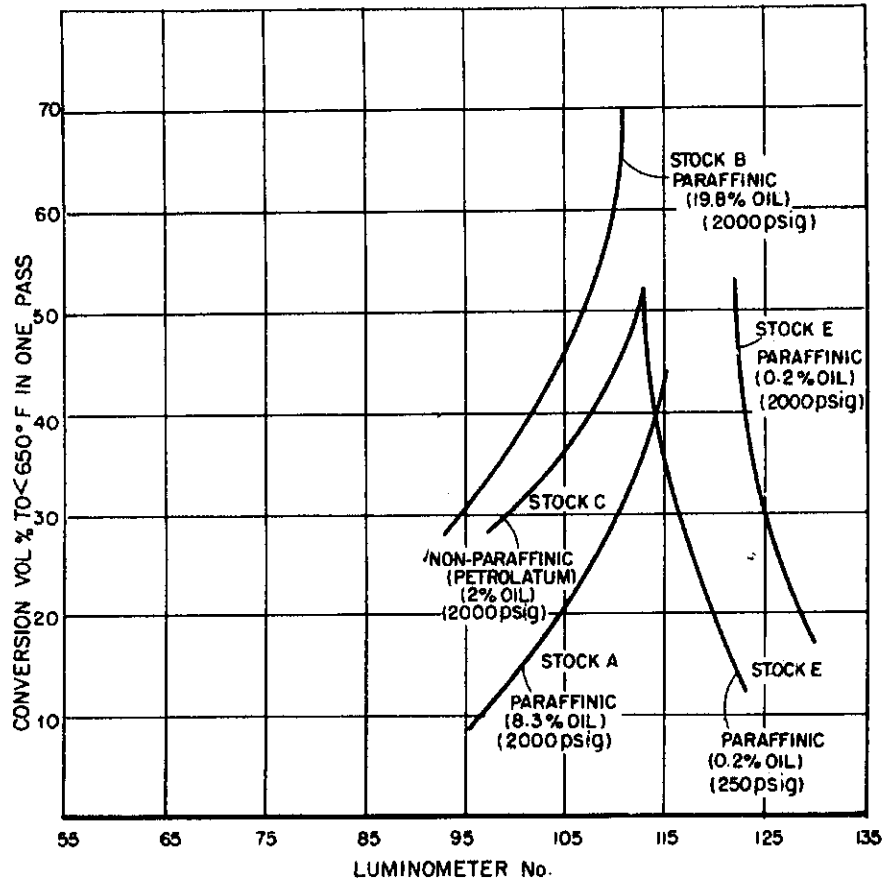
as measured by temperature in the range of about 500° to about 900°F. and the liquid hourly space velocity in the range of 0.25 to 3.0 to convert from 25 percent to 70 percent of the waxy hydrocarbon mixture to products boiling below about 650°F. to produce jet fuel fraction boiling in the range of about 375° to about 500°F. and having a luminometer number of at least 100 and a lubricating oil having a viscosity index of at least 120.

25. The process set forth in claim 24 wherein the isomerization catalyst comprises about 0.6 weight percent of platinum and about 5 to 6 weight percent of halogen of which at least about 90 percent is fluorine on alumina.

26. The process set forth in claim 24 wherein a diesel fuel boiling in the range of about 500° to about 650°F. and having a cetane number of at least 60 is produced.

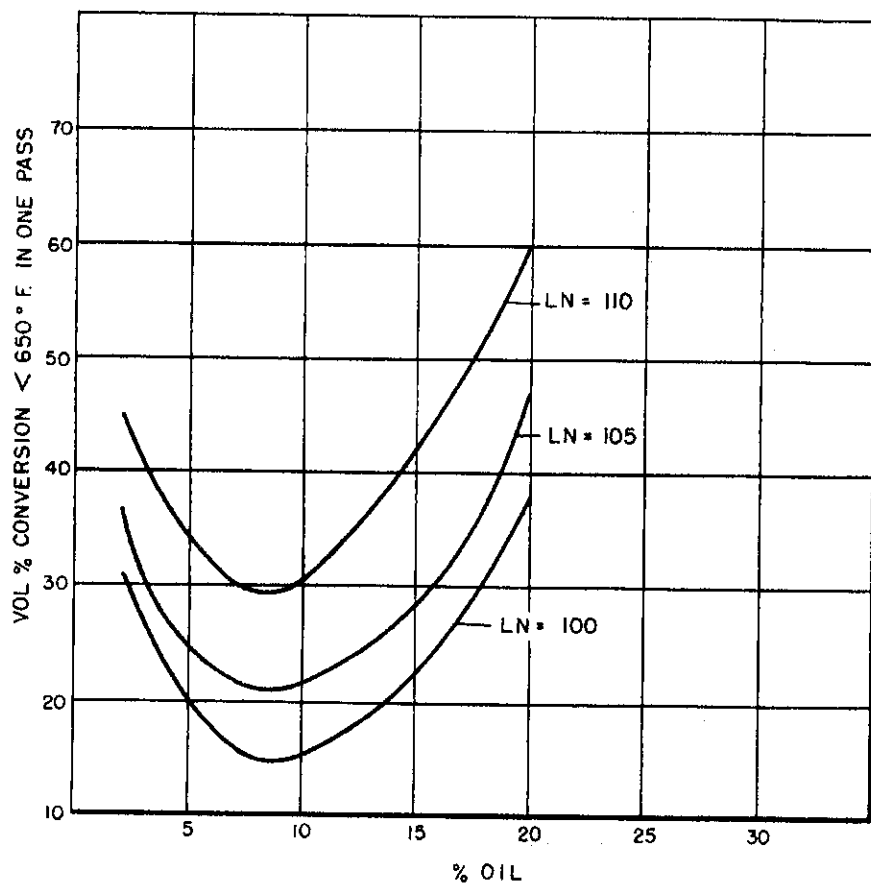
27. The process set forth in claim 24 wherein (1) a jet fuel boiling in the range of about 375° to about 500°F. and having a luminometer number of at least 100, (2) a diesel fuel boiling in the range of about 500° to about 650°F. and having a cetane number of at least 60, (3) an oil suitable for the hydraulic systems of supersonic aircraft stable in the range of 0° to 700°F. boiling above 650°F. and having a viscosity index of at least 100, and (4) an automotive lube oil boiling above 650°F. and having a viscosity index of at least 125 are produced.

FIG. 1



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FIG. II



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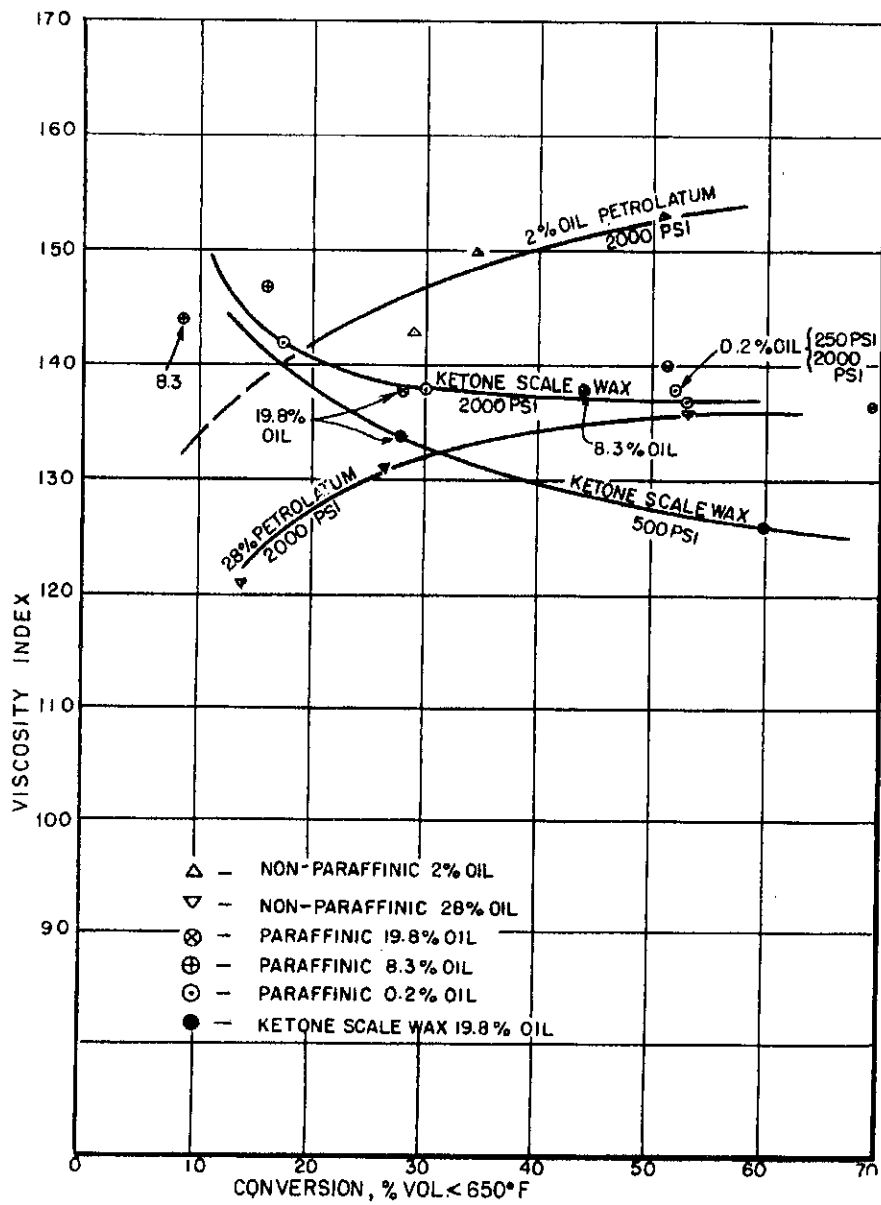


FIG. III

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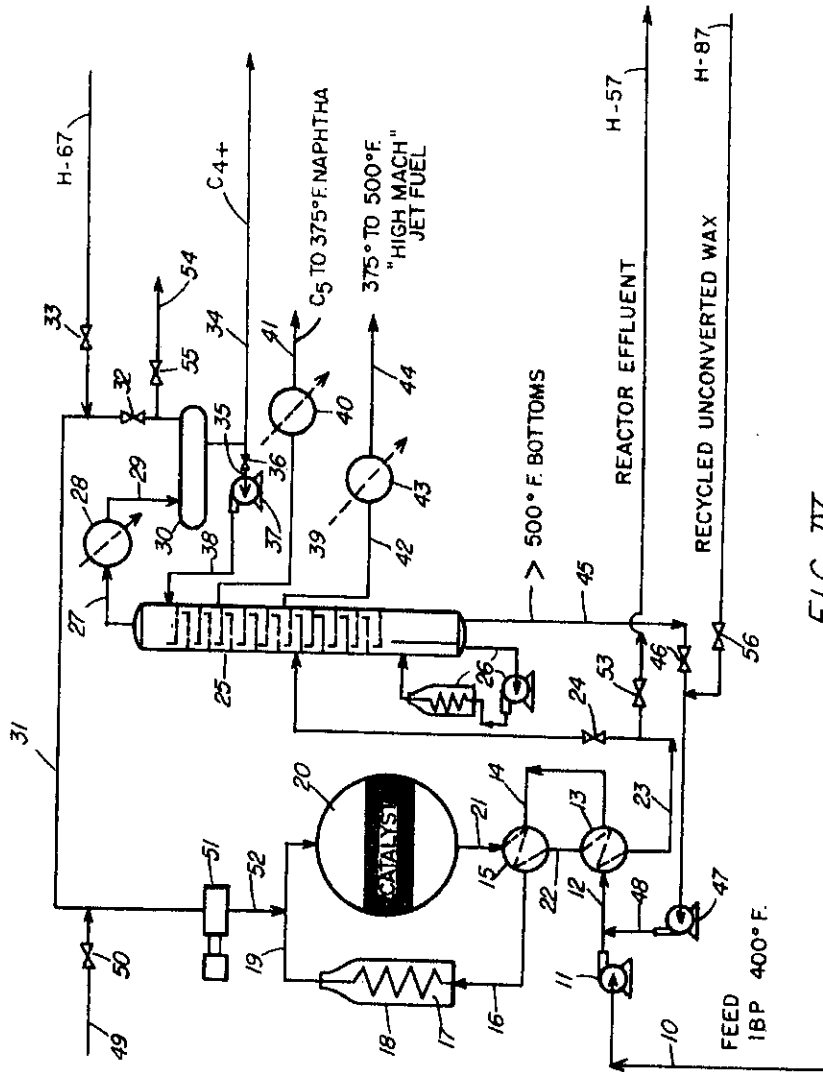


FIG. IV

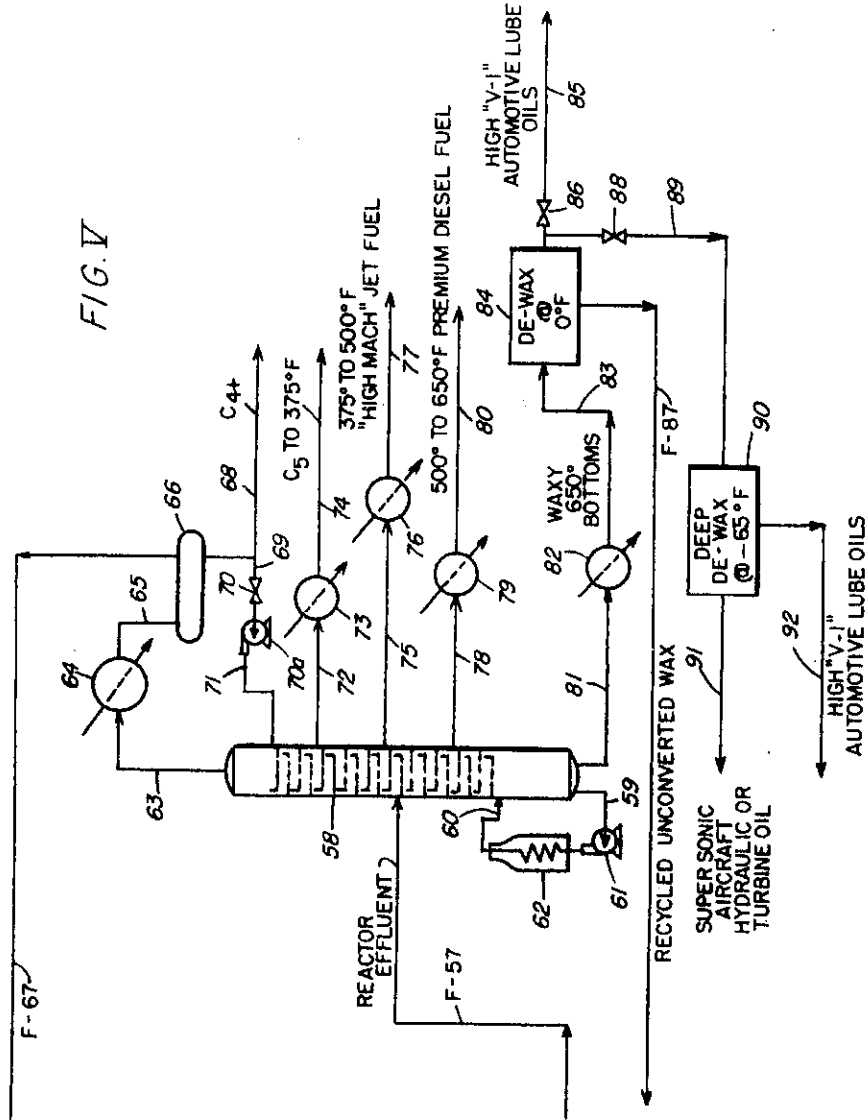
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FIG. V



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