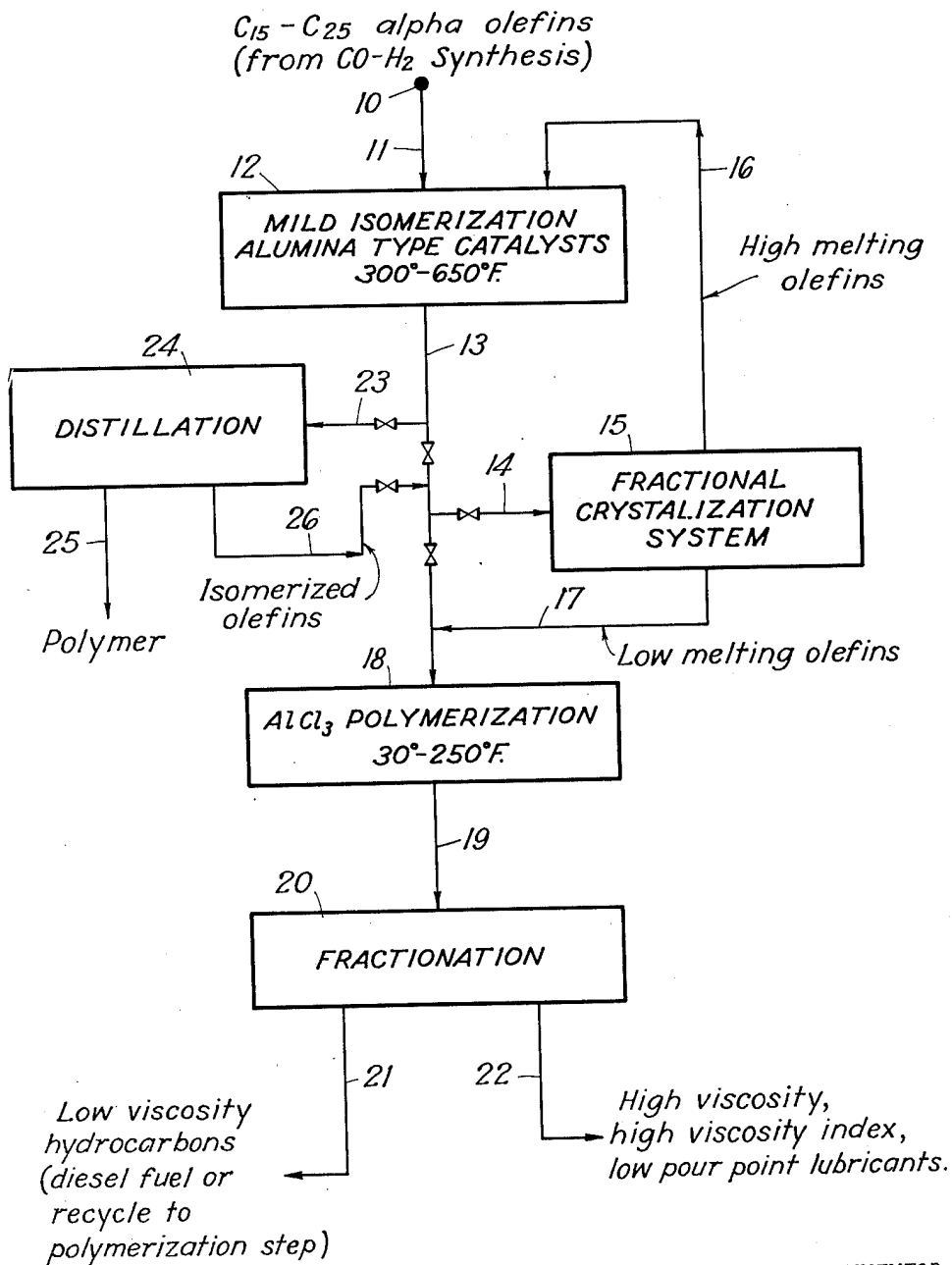


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SYNTHETIC LUBRICATING OIL

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## SYNTHETIC LUBRICATING OIL

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This invention relates to an improved two-step process for the conversion of high boiling alpha olefins into lubricating oils characterized by a high viscosity, a high viscosity index and a low pour point. In a preferred embodiment the invention relates to synthetic lubricating oil production from alpha olefins higher than C<sub>14</sub> and preferably in the range of about C<sub>15</sub> to C<sub>25</sub> and to such olefins which are obtained by hydrocarbon synthesis with iron type catalyst.

The production of lubricating oils from low boiling olefins has long been known to the art and is described for example in U. S. Patents 1,374,277, 1,798,288 and 1,955,260 (also note Ind. Eng. Chem. 23 (1931) page 607 et seq.). Lubricating oils have also been prepared from relatively high boiling alpha olefins but the viscous oils of high viscosity index thus prepared have been characterized by a high pour point. An object of this invention is to provide an improved method and means whereby low pour point synthetic oils can be obtained from alpha olefins having from about 15 to about 25 carbon atoms per molecule. A further object is to provide a method and means for treating high boiling alpha olefins for effecting mild isomerization and lowering of their pour point without changing their molecular weight and without isomerizing them to such an extent as to impair their subsequent polymerizability. Another object is to provide an improved method and means for converting to more valuable materials the high boiling hydrocarbon fractions obtained by carbon monoxide-hydrogen synthesis with iron type catalysts. Other objects will be apparent as the detailed description of the invention proceeds.

I have discovered that when a high boiling alpha olefin, such for example as 1-octadecene, is treated with an active silica-alumina catalyst (of the type conventionally employed in catalytic cracking operations) at a temperature in the range of about 300 to 650° F., its melting point (which may be considered as its pour point) may be lowered by as much as 50° F. or more, depending upon the severity of the treatment. It appears that this lowering of the melting point is due to a shifting of the double bond from the alpha position toward the center of the molecule. I have also discovered that when the high melting point 1-octadecene is polymerized with aluminum chloride the resulting lubricating oil has a pour test of about 55° F. and that when the isomerized, low melting point octadecene is polymerized with aluminum chloride, a lubricant with a pour point as low as -30° F. is obtained. In accordance

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with my invention, therefore, I first effect a mild isomerization of a high boiling alpha olefin with a solid alumina type catalyst at a temperature in the range of about 300 to 650° F. at a pressure of about atmospheric to about 50 pounds per square inch gauge and with a space velocity in the range of about 1 to 100 volumes of oil per hour per volume of catalyst employed. In some cases it may be desirable to distil the product. The mildly isomerized olefin thus obtained is then polymerized with a Friedel-Crafts type catalyst such as aluminum chloride under conventional polymerizing conditions.

The mildly isomerized olefins may be fractionated by crystallization methods prior to the polymerization step so that only the low melting point olefins are polymerized while the high melting point olefins are recycled to the isomerization step.

The resulting polymerization product may be vacuum distilled into fractions of various boiling ranges and/or subjected to conventional dewaxing processes for obtaining oils of still lower pour point.

When the invention is applied to high boiling paraffinic fractions of the product obtained by carbon monoxide-hydrogen synthesis with iron catalyst, the mild isomerization step accomplishes the further function of converting some of the oxygenated materials contained in the charge into olefins with the elimination of water. This reduces the amount of catalyst required for effecting polymerization. The unpolymerized portions of the product from the aluminum halide treatment are superior components for use in diesel or burner fuels. These are of higher cetane number and better burning quality than the original synthesis fraction.

Heretofore it has been the belief of those skilled in the art that synthetic oils of low pour point, high viscosity index and high yield could not be prepared by the polymerization of olefins containing more than about 12 to 14 carbon atoms per molecule. The oil obtained by polymerizing 1-heneicosene (C<sub>21</sub>), for example, has been reported to have a pour test of 92° F. By polymerizing 1-octadecene with aluminum chloride I obtained a viscous oil having a pour test of 55° F. 1-octadecene itself has a pour test of about 60° F. depending on the purity. Treatment of 1-octadecene at about 375° F. at atmospheric pressure in a batch process at various space velocities (V<sub>o</sub>/Hr./V<sub>c</sub>) with a very active silica-alumina resulted in a material lowering of the pour point

or melting point thereof as shown by the following tabulation:

$V_0/\text{Hr.}/V_c$	Pour Test, ° F.
12	+60
4.8	+45
2.7	+25
2.0	+15
	+10

A small amount of polymer was formed but the lowering in pour test is due principally to shifting of the double bond. Polymerization of the low pour test isomate gives still lower pour test polymer oil.

The invention will be more clearly understood from the following detailed description of specific examples and from the accompanying drawing which is a schematic flow diagram representing the sequence of steps employed.

#### Example 1

When 1-octadecene was initially isomerized with active silica-alumina catalyst at 375° F. and atmospheric pressure in a batch reactor at about 1  $V_0/\text{Hr.}/V_c$ , and the entire resulting product was polymerized at room temperature with aluminum chloride, the resulting synthetic oil was found to have a pour test of -5° F., compared with +55° F. pour test on polymer oil from 1-octadecene. Raising the polymerization temperature to 140° F. gave a better yield. Data with respect to the polymerization and polymer properties in these cases are as follows:

Charge	1-Octadecene	Mildly isomerized 1-octadecene	
		(a)	(b)
Polymerization Step:			
Temp. ° F.	(1)	(1)	140
Wt. percent AlCl <sub>3</sub>	5	5	2
Time, Hrs.	6	6	6
Wt. Percent Polymer Oil	82	55	82
Polymer Properties:			
Mol. Wt., Av.	1004	622	626
Pour Test, ASTM, ° F.	55	-5	0
Say. Vis. at 210° F.	138.7	62	56.3
Vis. Index	130	126	129
Color, ASTM		1-1.5	1.5

<sup>1</sup> Room.

It will be noted that the isomerization step results in a remarkable improvement in the pour test of the polymer oil without any substantial reduction in the high viscosity index. The preliminary mild isomerization materially alters the polymerization as indicated by the differences in average molecular weights and Saybolt viscosities at 210° F. However, the oil obtained through the mild preliminary isomerization step has a Saybolt viscosity at 210° F. of 62 seconds, a viscosity index of 126 and an ASTM color of 1 to 1½ and it is most remarkable that the oil is obtained with a pour test as low as -5° F.

#### Example 2

To determine the effect of the preliminary mild isomerization step on a large scale source of the higher olefins, runs were made on a C<sub>18</sub>-C<sub>22</sub> fraction of the product which was obtained from a carbon monoxide-hydrogen synthesis operation over iron type catalyst at a temperature of the order of about 540° F. to 660° F. and a pressure in the range of about 150 to 650 p. s. i. g. It is known that such high boiling synthesis product is about 50% olefinic and contains substantial

amounts of alpha olefins. Data on batch runs made with this particular charging stock are as follows:

5	Isomerization Step:		
	$V_0/\text{Hr.}/V_c$	None	1.0.
	Isomerization Temperature	do.	400° F.
	Isomerization Catalyst	do.	Silica-Alumina.
10	Polymerization Step:		
	Wt. Percent AlCl <sub>3</sub>	10%	10%.
	Temperature	Room	Room.
	Time, Hrs.	6	6.
	Wt. percent polymer yield	41	36.
15	Polymer Properties—		
	Molecular weight, average	718	563.
	Four point, ASTM	+25° F.	-5° F.
	Say. viscosity at 210° F.	157	103.
	Viscosity Index	101	97.

Here again it will be noted that the preliminary isomerization step markedly alters the nature of the subsequent polymerization and with the initial mild isomerization step the lubricating oil polymer had a Saybolt viscosity at 210° F. of 103 seconds, a viscosity index of 97 and an ASTM pour point of -5° F. It should be noted that the AlCl<sub>3</sub> requirement of 10% will be less if the polymerization temperature is raised to 140° F. or if a longer reaction time is employed.

#### Example 3

The following example demonstrates that a partially isomerized alpha olefin can be fractionated by crystallization methods to obtain a more completely isomerized portion for the polymerization step, and to recycle the cake fraction to the isomerization step. 1-octadecene was partially isomerized by contacting it in the liquid phase at 375° F. with active silica-alumina at  $V_0/\text{Hr.}/V_c$  of 6. This treatment reduced the pour test from 60° F. to 25° F. The product was dissolved in 3.5 vols. of acetone and 2.5 vols. of toluol, chilled to -25° F., and filtered. The filtrate and cake fractions after removal of diluent were approximately equal in weight and had pour tests of 20° F. and 35° F., respectively. Upon polymerization of each at room temperature with 10% AlCl<sub>3</sub> for 6 hours followed by removal of AlCl<sub>3</sub> and unreacted monomer, the following polymer oils were obtained:

	Filtrate Fraction	Cake Fraction
50		
	Polymer yield, wt. percent	58
	Pour test, ASTM ° F.	-5
	Mol. wt.	656
	Say. Vis. at 210° F.	64.1
	Vis. Index	127
55		
		55
		+5
		690
		65.5
		128

#### Example 4

Silica-alumina catalyst was used to isomerize 1-octadecene in a batch type reaction at 500° F. The higher temperature permitted a smaller amount of catalyst to be used. 225 volumes of octadecene were stirred 0.84 hour with 3 volumes of catalyst. The  $V_0/\text{Hr.}/V_c$  ratio was 89. The isomate was distilled to eliminate 13% of polymer of +50° F. pour test formed by competing reaction. The distillate, which now had a pour test of -10° F. was stirred for nine hours with 3% of AlCl<sub>3</sub> at 60° F. and finished in conventional manner to give 45% of C<sub>18</sub> cut suitable for recycle to the polymerizer, 5% loss to AlCl<sub>3</sub>, and a 50% yield of polymer with the following properties:

70	Pour test of	° F.	-20
	Say. vis. at 210° F.		56.8
	Vis. index		126
	Color, ASTM		2
75	Mol. wt.		580

## Example 5

Alpha octadecene was isomerized in the presence of two volumes of diluent, dodecane, in order to suppress the formation of the minor amount of high pour polymer from alpha olefin which otherwise occurs simultaneously with the isomerization. The reaction was batch-type at 375° F. with a ratio of  $V_0/Hr./V_c$  of 7.5. For comparison the results from a comparable reaction in the absence of diluent are given below:

	Diluent used	No diluent
Percent polymer formed.....	2.7	8.
Pour test of feed.....	60	60
Pour test of polymer free isomate, °F.....	+20	+15

$AlCl_3$  polymerization of the above isomates produced polymer oil of 0° F. pour test.

## Example 6

In this example the isomerization catalyst was alumina gel prepared as described by Llewellyn Heard in U. S. 2,449,847. This catalyst is milder than the silica-aluminas. Twenty volumes of alpha octadecene were stirred with three volumes of the freshly activated catalyst for six and one-half hours at 500° F. in an inert atmosphere, thus making a  $V_0/Hr./V_c$  ratio of 1. The isomate was distilled to eliminate five per cent of high pour polymer and then had a pour test of 25° F. It was polymerized with 2% of  $AlCl_3$  in an HCl atmosphere at 140° F. for seven hours. The catalyst complex layer was removed, product washed, 18% of monomer recovered for recycle purposes, and a yield of 69 weight per cent of polymer obtained (based on isomate charged) with the following properties:

Pour test.....°F.....	+5
ASTM color.....	2
Mol. wt.....	670
Say. vis. at 210° F. ....	59.3
Vis. index.....	130

If desired the pour test could be made lower by extending the isomerization somewhat.

## Example 7

Five volumes of alpha octadecene were stirred for two and one-half hours at 375° F. with two volumes of montmorillonite clay which had been acid leached, washed, and dried at about 1000° F. The entire product from this reaction was contacted at room temperature for six hours with 5% of  $AlCl_3$  and finished in a conventional manner. This procedure gave a 47% yield, based on alpha octadecene, of polymer oil of high viscosity index, with a pour test of +20° F., an average molecular weight of 672 and a color of 2½ ASTM.

Active clays of this type are principally silica-aluminas containing several per cent of magnesia and other oxides. Catalysts containing magnesia, including synthetic silica-magnesia, have been found to cause considerable polymerization of alpha olefin before the isomerization is complete. More satisfactory isomerization can be obtained with these catalysts by lowering the olefin concentration; for example, by operating with diluent present or with the oil charge in the vapor phase.

## Example 8

The improvement in the fuel characteristics of the unpolymersizable portion of a carbon

monoxide-hydrogen synthesis fraction is shown in the following example. A  $C_{18}-C_{22}$  fraction from the synthesis product from iron type catalyst was isomerized in a batch operation with synthetic silica-alumina at 375° F. and a ratio of  $V_0/Hr./V_c$  of five, and subsequently polymerized with ten percent of aluminum chloride to give thirty-seven weight percent of polymer oil having a molecular weight of 643, a Saybolt viscosity at 210° F. of 104 and a viscosity index of 100. Unpolymerized  $C_{18}-C_{22}$  fraction was recovered in thirty-six weight per cent yield and had the improved fuel characteristics shown in the following table:

	Original $C_{18-22}$	Recovered $C_{18-22}$
Cetane Index <sup>1</sup> .....	65	74
Watson Characterization Factor <sup>2</sup> .....	12.1	12.4
Gms. of Carbon Deposit per 10 gal. Oil Consumed (Calc.).....	13.6	5.
Percent Oxygen.....	1.25	0.82

<sup>1</sup> Sub-committee 4, Gas, Diesel and Fuel Oils, J. Inst. Pet. Tech., 30, 193, 1944.

<sup>2</sup> Ind. Eng. Chem., 27, 1460, 1935.

The severity of the preliminary isomerization step must be within certain defined limits in order to obtain the beneficial results hereinabove described and illustrated. Generally speaking, while any olefin polymerization catalyst of the alumina type may be employed, active silica-alumina catalysts (of the type used in catalytic cracking) require the use of milder conditions than catalysts of the activated alumina or alumina-gel types. Alumina-gel or activated alumina catalysts require considerably higher temperatures and/or longer reaction periods than are employed for silica-alumina catalyst for obtaining equivalent results. With the weaker alumina-gel catalysts at  $V_0/Hr./V_c$  around 10 I may employ temperatures as high as about 700° F. whereas with very active silica-alumina catalysts at such space velocity temperature required is about 375° F., the preferred temperatures being in the range of about 375 to 500° F. in the case of silica-alumina catalysts and about 500 to 700° F. in the case of activated alumina or alumina-gel. Space velocities may be in the range of about 0.1 to 200  $V_0/Hr./V_c$ , the higher space velocities being employed with the higher temperatures and more active catalysts. The isomerization may be effected in any conventional manner in fixed bed, moving bed or fluidized catalyst systems, and in liquid or vapor phase.

I have found that when the olefin treatment is effected at temperatures upwards of about 700° F. the reaction is so drastic at customary contact times that little or no polymerization can subsequently be effected with the aluminum chloride. Thus the  $C_{17}-C_{21}$  cut from a hydrocarbon synthesis fraction treated with a silica-alumina catalyst at 800° F. with a weight space velocity of 13.3, in a continuous fluid reactor gave only 3 weight percent of polymer in the subsequent aluminum chloride polymerization step. A  $C_{18}-C_{22}$  hydrocarbon fraction from synthesis product isomerized at 712° F. with alumina gel at a weight space velocity of 2 in a fixed bed reactor gave an isomerized product which, upon subsequent polymerization, yielded only 14 weight percent polymer. Thus the conditions heretofore normally employed for finishing and/or deoxygenating hydrocarbon synthesis products are too drastic when the treated prod-

uct is to be polymerized with aluminum chloride for the production of low pour point lubricating oils of high viscosity and high viscosity index.

Referring to the drawing, alpha olefins in the range of C<sub>15</sub> to C<sub>25</sub> are introduced from source 10 through line 11 to isomerization system 12. When such olefins are those contained in a hydrocarbon product fraction from carbon monoxide-hydrogen synthesis over an iron type catalyst they may contain oxygenated compounds as impurities or they may be diluted with other hydrocarbon components.

The isomerization must be milder than that heretofore employed in the refining of synthesis hydrocarbons for improving their color, odor and knock rating. The alumina type catalysts may be silica-alumina, activated alumina or alumina-gel catalysts all of which are well known to those skilled in the art and require no detailed description. The important feature in this step is to insure the use of sufficiently mild conditions to effect a lowering of the pour point or melting point of the alpha olefins without seriously interfering with their ability to be polymerized by aluminum chloride or other known polymerization catalysts. Small amounts of water decrease the activity of these catalysts. For this reason they should be conditioned or activated by heating, preferably to 1000° F. The isomerization should be effected within the range of about 300 to 650° F., with excellent results being obtainable at about 400° F. and V<sub>0</sub>/Hr./V<sub>0</sub> of about 5 with silica-alumina catalysts and about 500° F. with V<sub>0</sub>/Hr./V<sub>0</sub> of .5 with alumina gel catalysts. The isomerized olefins may be withdrawn by lines 13 and 14 to a frictional crystallization systems 15 wherein separation of the olefins according to their melting points may be effected by any known method, the high melting olefins being recycled to the isomerization step by line 16 and the low melting olefins being introduced by lines 17 and 18 to polymerization system 18.

The polymerization may be effected with any known catalyst of the Friedel-Crafts type, such for example as aluminum chloride, the preferred catalysts being the chlorides, bromides and fluorides of aluminum, zinc and boron. The catalyst may be activated by hydrogen halides. Conventional operating conditions may be employed for effecting the polymerization and with aluminum chloride temperatures may be in the range of about 30 to 250° F. Usually the amount of aluminum chloride required for treating high boiling hydrocarbon synthesis products is somewhat higher than that required for polymerizing pure olefins unless the isomerized olefins are given a preliminary treatment to remove certain of the impurities which tend to modify the catalyst or unless a high polymerization temperature is employed. From the previous examples it will be noted that 5% aluminum chloride was adequate for polymerization in all cases at a temperature of 140° F. Some form of countercurrent reaction with the AlCl<sub>3</sub> is desirable for efficient use.

After polymerization the products are withdrawn through line 19 to a conventional fractionation system 20 from which low viscosity hydrocarbons may be removed by line 21 and the synthetic lubricating oils may be removed by line 22. Synthetic lubricating oils may be fractionated by vacuum distillation and/or subjected to other processes of lubricating oil refining. If desired, at least a part of the product from the isomerization process may be passed via line 23 to a distillative fractionation zone 24 wherein any

polymer in the isomerized product is separated and withdrawn through line 25. Isomerized product, substantially free of polymer, is returned to line 13 via line 26.

It should be emphasized that the process hereinabove described is particularly advantageous with alpha olefins having at least about 15 carbon atoms per molecule. The initial isomerization step is unnecessary with lower boiling olefins. Thus a C<sub>12</sub>-C<sub>13</sub> hydrocarbon product fraction obtained by carbon monoxide-hydrogen synthesis over iron catalyst can be polymerized without the initial isomerization step to give a synthetic oil having a Saybolt viscosity at 210° F. of 76.2 seconds, a viscosity index of 108 and a pour test of -30° F.

The synthetic polymer oils may be hydrogenated or alkylated by conventional methods to increase the stability to oxidation. Oxidation inhibitors may also be used.

I claim:

1. The method of producing lubricating oils of high viscosity, high viscosity index and low pour point from alpha olefins in the range of C<sub>15</sub>-C<sub>25</sub> which method comprises isomerizing said alpha olefins by contacting them with a solid catalyst having alumina as its active component at a temperature in the range of about 300 to about 650° F. under a pressure of about atmospheric to about 50 pounds per square inch with a space velocity in the range of 1 to 100 volumes of oil per hour per volume of catalyst employed and sufficient to effect isomerization to such an extent that the pour point thereof is materially lowered without destroying their polymerizability and subsequently polymerizing the isomerized olefins with a Friedel-Crafts catalyst.

2. The method of producing a synthetic lubricating oil of high viscosity, high viscosity index and low pour point which method comprises isomerizing an alpha olefin in the range of C<sub>15</sub> to C<sub>25</sub> with an alumina catalyst at a temperature in the range of about 300 to about 650° F. to obtain the lowering of the pour point of at least a substantial portion of said olefins and contacting said olefins of lower pour point with a Friedel-Crafts polymerization catalyst under conditions for effecting polymerization thereof.

3. The method of claim 2 which includes the step of separating high melting point from low melting point olefins from products leaving the isomerization step and recycling high melting point olefins to said isomerization step.

4. The method of claim 2 wherein the isomerization catalyst is a silica-alumina catalyst and wherein the isomerization temperature is below about 550° F.

5. The method of claim 2 wherein the isomerization catalyst consists essentially of alumina and wherein the isomerization temperature is at least about 500° F.

6. The method of claim 2 in which the alpha olefin is a component of a high boiling oxygen containing hydrocarbon product fraction obtained by carbon monoxide-hydrogen synthesis over iron catalyst at a temperature in the range of 540° F. to 660° F., and a pressure in the range of 150 to 650 pounds per square inch.

7. The method of claim 2 which includes the step of employing a diluent for suppressing polymer formation while the isomerization is being effected.

8. The method of claim 2 wherein the pour point of at least a substantial portion of said olefins is lowered by at least 20° F.

9. The method of claim 8 which includes the step of separating the portion of said olefins of lowered pour point from other olefins prior to the polymerization step.

10. The method of producing a synthetic lubricating oil of high viscosity, high viscosity index and low pour point, which method comprises isomerizing an alpha olefin in the range of C<sub>15</sub> to C<sub>25</sub> with an alumina catalyst at a temperature in the range of about 300 to about 650° F. to obtain a lowering of the pour point of a substantial portion of said olefins to the extent of at least 20° F., separating the portion of lower pour point olefins from other olefins in the isomerization product by fractional crystallization, recycling said other olefins from the fractional crystallization step to the isomerization step, and contacting the portion of separated olefins of low pour point with a Friedel-Crafts polymerization catalyst under conditions for effecting polymerization thereof.

11. The method of obtaining a high quality lubricating oil and a high quality diesel fuel from an oxygen-containing, high-boiling hydrocarbon product of carbon monoxide-hydrogen synthesis over iron catalyst which product contains alpha olefins having at least 15 carbon atoms per molecule, which method comprises contacting said product with a solid isomerization catalyst containing alumina as its active component under non-cracking conditions including a temperature in the range of about 300 to 650° F. and a space velocity sufficient to give isomerization to an isomerized material of lower pour point as the predominant reaction, contacting the products of the isomerization step with a polymerization catalyst under polymerization conditions and fractionating the products from the last named contacting step into a low viscosity hydrocarbon fraction of high cetane index and a high viscosity lubricating oil fraction of low pour point and high viscosity index.

12. The method of producing lubricating oils of high viscosity index and low pour point from a C<sub>15</sub> plus hydrocarbon product produced by carbon monoxide-hydrogen synthesis over iron catalyst and containing a substantial amount of alpha olefins which method comprises contacting said product with a solid isomerization catalyst containing alumina as its active component under non-cracking conditions including a temperature in the range of 300 to 650° F., and a space velocity sufficient to effect isomerization to an isomerized material of lower pour point as the predominant reaction with polymerization as a minor side reaction, distilling the products of said contacting step to remove polymerization products produced as a side reaction and poly-

merizing the isomerized product fraction to obtain a high viscosity lubricating oil of low pour point and high viscosity index.

13. The method of claim 12 which includes the further step of separating a low pour point isomerized olefin from a higher pour point olefin by fractional crystallization so that only the low pour point isomerization product is subjected to polymerization.

14. The method of obtaining high quality diesel fuels and lubricants from high boiling oxygen containing hydrocarbon products of carbon monoxide-hydrogen synthesis over iron catalyst and containing alpha olefins in the range of 15 to 25 carbon atoms per molecule, which method comprises contacting said products with a silica-alumina catalyst at a temperature in the range of about 300° F. to 550° F. at a space velocity sufficient to obtain olefin isomerization to an isomerized material of lower pour point as the predominant reaction, together with a minor amount of polymerization, separating the products from the contacting step to obtain an isomerized olefin fraction in which the olefins have a substantially lower melting point than the olefins contained in the original synthesis product, polymerizing said isomerized olefin fraction with an aluminum chloride catalyst at a temperature in the range of 130 to 250° F., with a sufficient amount of catalyst and time of contact to polymerize a substantial amount of the olefins into polymers of high viscosity and fractionating products from the polymerization step to obtain a low viscosity hydrocarbon fraction of high diesel index and a high viscosity fraction of low pour point and high viscosity index.

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