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3,663,423

PRODUCTION OF LUBRICATING OILS

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U.S. Cl. 208—59

6 Claims

ABSTRACT OF THE DISCLOSURE

A wholly catalytic route for the production of lubricating oils for petroleum feedstocks boiling above 350° C. comprises the steps of VI improvement over a catalyst of a Group VIa and/or Group VIII metal on a refractory oxide support and catalytic dewaxing over a catalyst of a Group VIa and/or Group VIII metal on low alkali metal content mordenite. The steps may be in either order but the VI improvement step is preferably first.

The VI improvement step is at 650–850° F. and 1000–3000 p.s.i.g. using preferably, Co-Mo or silica alumina and the catalytic dewaxing step is at 450–950° F. and 100–3000 p.s.i.g. using, preferably, Pt on decationised mordenite having a SiO₂:Al₂O₃ ratio of 14:1–50:1. Depending on the feedstock to and the severity of the VI improvement step, lubricating oil product fractions of either high (75–100) or super high (100–140) VI may be produced.

This invention relates to the production of lubricating oils by a wholly catalytic route without the use of solvents.

As conventionally practised the production of lubricating oils requires the steps of solvent extraction with e.g. furfural to remove aromatics and improve the viscosity index, solvent dewaxing with e.g. an alkyl ketone to remove n-paraffins and improve the pour point and, possibly, a finishing treatment with acid, bauxite or clay to improve colour and colour stability. Hydrocatalytic treatment to replace the acid, bauxite or clay finishing treatment is now well established commercially but although it is acknowledged that solvent extraction of aromatics and solvent dewaxing are expensive and complicated operations, progress at replacing them with hydrocatalytic treatments has been slower. One possible reason for this is that the full economic advantages of hydrocatalytic treatment can only be achieved when satisfactory hydrocatalytic processes have been developed for both stages.

According to the present invention a wholly catalytic process for the production of lubricating oils from a petroleum feedstock boiling above 350° C. without the use of solvents for aromatic and wax removal comprises the steps of passing the oil over a catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the Periodic Table on a refractory oxide support together with hydrogen at a temperature of from 650 to 850° F. and a pressure of from 1000 to 3000 p.s.i.g. to give a material of reduced aromatic content and improved viscosity index and also over a catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the Periodic Table incorporated with a crystalline mordenite of reduced alkali-metal content together with hydrogen at a temperature of from 450 to 950° F. and a pressure of 100 to 3000 p.s.i.g. to give a material of reduced wax content and improved pour point and recovering a lubricating oil fraction of both improved viscosity index and improved pour point.

The steps of viscosity index improvement and catalytic

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dewaxing may be carried out in either order. Preferably the viscosity index improvement step precedes the catalytic dewaxing step. The abbreviation "VI" will be used hereinafter to refer to viscosity index.

The feedstock to the process is preferably a vacuum distillate fraction boiling within the range 350–600° C. Since lubricating oils are marketed in several grades with relatively narrow boiling ranges, distillation to give relatively narrow boiling range cuts is required at some stage. In the present invention, a wide boiling range cut may be used as feedstock and distillation into narrower cuts given after the first or second hydrocatalytic treatment or distillation may take place before the hydrocatalytic treatment and individual cuts may be hydrotreated. The former route has the advantage of avoiding blocked operation but the latter route has the advantage that optimum hydrotreating conditions may be chosen for each cut.

Within the context of hydrocatalytic treatment for VI improvement, two general processes are recognised. These are (i) a relatively mild treatment to give a moderate VI improvement e.g. 75 to 100 VI after dewaxing with minimum breakdown and decrease in viscosity. Increase in VI of the product to bring it up to multi grade oil requirements, if desired, can be achieved by the use of known polymeric VI improvers. (ii) a more severe treatment with extensive breakdown and decrease in viscosity but giving a larger VI improvement e.g. greater than 100 VI after dewaxing. A larger amount of lower boiling gas oil, kerosene and gasoline by-products are obtained with the more severe treatment.

Preferably the milder type (i) process is used with a distillate feedstock boiling in the range 350–600° C. having a VI of 30 to 90 and with a breakdown to products boiling below 350° C. of less than 30 percent wt. under the following ranges of conditions:

Temperature, ° F.	650–850
Pressure, p.s.i.g.	1000–3000
Space velocity, v./v./hr.	0.1–5
Hydrogen treating rate, s.c.f./b.	2000–10,000

If the VI improvement step precedes the catalytic dewaxing step the VI of the product boiling above 350° C. should be in the range 90–120 to allow for some reduction in VI in the subsequent catalytic dewaxing step. If it follows the catalytic dewaxing step this dewaxing step will already have lowered the VI of the original feedstock, and the VI of the product boiling above 350° C. from the VI improvement step should be from 75 to 100.

The refractory inorganic oxide support may be one or more oxides of elements of Groups II, III or IV of the Periodic Table, and may contain halogen. Preferably the amounts of the components of the support are chosen from the following ranges:

	Percent wt.
Al ₂ O ₃	50–100
SiO ₂	0–50
B ₂ O ₃	0–25
F or Cl	0–10

A particularly preferred support has from 50–90 percent wt. of Al₂O₃ and 10–50 percent wt. of SiO₂.

The hydrogenating component is preferably one or more of the oxides or sulphides of the Group VIa metals (i.e. chromium, molybdenum or tungsten) and the iron group metals (i.e. iron, cobalt or nickel). If platinum group metals are used they are desirably in metallic form. The amounts of the hydrogenating components may be

	Percent wt. (expressed as metal)
Group VIa metals	2–25
Iron group metals	1–15
Platinum group metals	0.1–5

Preferred hydrogenating components are from 2-25 percent wt. of molybdenum (expressed as metal but present as oxide or sulphide) and 1-15 percent wt. of nickel and/or cobalt again expressed as metal but present as oxide or sulphide.

With the more severe type of VI improvement process the preferred feedstocks boil with the range 450-600° C. and have a VI of 30-90. The following ranges of conditions may be used to give a conversion to products boiling below 350° C. of 30-80 percent wt.

Temperature, ° F. -----	725-850
Pressure, p.s.i.g. -----	1000-3000
Space velocity, v./v./hr. -----	0.1-5
Hydrogen treating rate, s.c.f./b. -----	2000-10,000

If the VI improvement step precedes the catalytic dewaxing step the VI of the product boiling above 350° C. should be in the range 120-160 to allow for some reduction on VI in the subsequent catalytic dewaxing step. If it follows the catalytic dewaxing step, this dewaxing step will already have lowered the VI of the original feedstock and the VI of the product boiling above 350° C. from the VI improvement step should be from 100 to 140.

The catalyst is preferably the same as for the milder type (i) process both in terms of the hydrogenating component and the support. The support may, however, be more acidic than the preferred support of the milder process and may consist for example of 60-90 percent wt. SiO₂ and 10-40 percent wt. Al₂O₃ or 85-90 percent wt. Al₂O₃ and 5-15 percent wt. halogen.

It will be appreciated that the acidity of the catalyst support, the temperature, pressure and space velocity all affect the severity of the operation and hence the degree of VI increase and viscosity decrease obtained and that any one or more of these may be varied as required. Preferably the space velocity is lower for the more severe type (ii) process than for the milder type (i) process.

The term VI as used in this specification means viscosity index as measured by ASTM D567. Currently two methods are in use ASTM Test Method D567 and the VIE extended method ASTM D2270. Appreciable differences in numbers are possible between the two methods when testing the same oil.

In addition to VI improvement, sulphur and nitrogen in the feedstock will be converted, if present, to H₂S and NH₃. The latter is desirably removed (by scrubbing with water) from any gas recycled and both H₂S and NH₃ are desirably removed from any gas passing to the hydrocatalytic dewaxing stage.

The catalytic dewaxing step operates selectively to break down the paraffinic waxes to lower boiling materials which are readily removable from the remainder of the lubricating oil feedstock, which remains substantially unaffected. It may follow the general procedure described in U.K. patent specifications Nos. 1,088,933; 1,134,014 and 1,134,015. The term "paraffinic wax" refers to those waxy hydrocarbons which have a long straight chain as part of their structure, e.g. particularly n-paraffins themselves but also very slightly branched chain hydrocarbons, and cyclic hydrocarbons having an unbranched alkyl side-chain. The selective breakdown of the paraffin wax hydrocarbons is due to the nature of the mordenite based catalyst which promotes the selective attack of these hydrocarbons but not of the other hydrocarbons present in the feedstock.

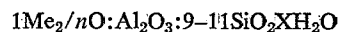
Suitable process conditions for the catalytic dewaxing include besides a temperature within the range 450-950° F. and a pressure within the range 100-3000 p.s.i.g. as indicated above, a space velocity between 0.1-20.0 v./v./hr., and a gas rate of 500-30,000 s.c.f. of hydrogen.

Preferred catalytic dewaxing conditions are:

Temperature, ° F. -----	500-800
Space velocity, v./v./hr. -----	0.5-10.0
Pressure, p.s.i.g. -----	500-3000
Hydrogen treating, s.c.f./b. -----	5000-15,000

The term "crystalline mordenite of reduced alkali metal content" means, preferably, a mordenite with an alkali metal content of less than 3 percent wt. The deficiency of alkali metal cations can be made up with other metal cations for example Group II metal cations, particularly magnesium or rare earth metal cations. Preferably however the mordenite is a "decationised mordenite" which means a mordenite having a deficiency of metal cations. An alternative term in the art is hydrogen mordenite, since it is assumed that when metal cations are removed they are replaced by hydrogen ions. However, since it is not possible to detect the presence of hydrogen ions in zeolites, the precise structure remains in doubt. A cation deficiency can, on the other hand, be readily measured by analysis of the metallic elements present in the zeolite.

Natural or freshly prepared synthetic mordenite has the formula:



where Me is a metal cation, *n* is the valency of the cation and X is variable between nil and 7 depending on the thermal history of the sample. Me is commonly sodium and in one common form of decationisation sodium mordenite is base exchanged with ammonium cations. The ammonium form is then heated to drive off ammonia, leaving behind the hydrogen form or decationised mordenite. According to the second method the mordenite may be treated with a mineral acid, for example hydrochloric or sulphuric acid, in order directly to decationise the mordenite. A combination of acid treatment and ammonium treatment can also be used.

Preferably the decationised mordenite used in the present invention has a higher than normal silica:alumina ratio of at least 14:1. In specific examples ratios of as high as 90:1 have been obtained and a practical upper limit may thus be 100:1. Particularly preferred silica:alumina ratios are in the range 14:1 to 50:1.

It has been found that certain decationisation treatments remove aluminium as well as the expected metal cations and desirably therefore the mordenite used in the present invention having a higher than normal silica:alumina ratio is obtained by treatment of a metal cation-containing mordenite, particularly sodium mordenite, with a strong acid, for example sulphuric or hydrochloric acid, of from 5-50 percent wt. strength and preferably from 10 to 20 percent wt. strength. A single treatment or two or more successive treatments may be given with acids of the strengths stated above.

A convenient method of treatment is to treat the mordenite with acid under reflux for a period of 2-12 hours.

In the decationised mordenite the residual metal cation content, for example the sodium cation content, should be less than 2 percent wt. of the mordenite and preferably less than 1.5 percent wt. of the mordenite.

It should be emphasised that mordenites with higher than normal silica:alumina ratios retain the crystal structure of mordenite and are not significantly altered in terms of physical strength, stability or crystallinity.

The hydrogenating component is preferably a platinum group metal, particularly platinum or palladium, and it is preferably added by ion-exchange. Preferably the mordenite is aged in water before adding the platinum group metal as described in the specification of U.K. application No. 4421/68, which application corresponds to U.S. patent application Ser. No. 790,829, filed Jan. 13, 1969 and now Pat. 3,553,103. The amount of the platinum group metal is preferably within the range 0.01 to 10 percent wt., particularly 0.1 to 5 percent wt. However, iron group metals, particularly nickel, also give useful results and they may be used in amounts similar to the platinum group metals. Mixtures of certain Group VI and VIII metals and compounds may also be used, e.g. cobalt and molybdenum.

The catalyst is preferably calcined at for example 250-600° C. before use to remove any water and to eliminate any ligands attached to the hydrogenation component.

The catalytic dewaxing step is capable of treating lubricating oil fractions with pour points of from 60 to 120° F. to give products of from +40 to -60° F. Some reduction of VI is to be expected since n-paraffins are materials of high VI.

The lubricating oil product from the dewaxing stage is stabilised to remove the breakdown products, which consist largely of C₃ and C₄ paraffins, which are valuable for use in LPG and as petrochemical feedstocks. There may be a small amount of C₅-C₇ hydrocarbons produced, but it is a particular feature of the catalyst of the present invention that it does not give large amounts of C₅+ hydrocarbons in the gasoline and middle distillate boiling ranges and is not a hydrocracking catalyst as generally understood.

As stated above, lubricating oils are usually given a finishing treatment to improve colour and colour stability which is now usually a mild hydrocatalytic treatment known as hydrofinishing. It has been found that the catalytic dewaxing process used in the present invention itself affects improvement in colour and colour stability so that it may be possible to dispense with a finishing step altogether.

The invention is illustrated by the following examples.

EXAMPLE 1

A Kuwait wax distillate having a 2% TBP point of 350° C. and an end point of 560° C. and having the inspection data shown in Table 1 below was hydrogenated under the conditions shown in Table 1.

TABLE 2

Conditions for catalytic dewaxing:												
Pressure, p.s.i.g.	1,000											
Temperature, ° F.	700											
Space velocity, v./v./h.	2.0											
Gas rate, s.c.f./bbl.	10,000											
Inspection data on dewaxed liquid product:												
Pour point, ° F.	-40											
Average yield on feed, percent wt.	71.0											
Yield of residue >371° C. on feed, percent wt.	52.0											
	50.8											
	Yield on residue, percent wt.	Yield on feed, percent wt.	KV, 100° F., cst.	KV, 210° F., cst.	VI	Pour point, ° F.	Yield on residue, percent wt.	Yield on feed, percent wt.	KV, 100° F., cst.	KV, 210° F., cst.	VI	Pour point, ° F.
Inspection data on fractions ex residue >371° C.:												
Fraction 1	17.1	8.9	30.39	4.39	22	-55	17.1	8.7	35.51	4.89	44	-50
Fraction 2	16.9	8.8	36.97	4.97	42	-45	16.8	8.5	40.39	5.36	59	-40
Fraction 3	17.8	9.3	44.90	5.72	63	-40	16.9	8.6	46.91	5.96	69	-15
Fraction 4	15.9	8.3	57.83	6.84	74	-25	16.7	8.5	57.49	6.95	80	0
Fraction 5	16.3	8.4	71.37	8.19	89	0	16.7	8.5	69.73	8.07	89	15
Fraction 6 (residue)	16.0	8.3	118.4	11.85	96	25	15.8	8.0	108.8	11.35	99	30

TABLE 1

Inspection data on feed:	
Kinematic viscosity at—	
100° F., cst.	75.00
210° F., cst.	7.92
Viscosity index	73.00
Pour point, ° F.	95
Sulphur content, percent wt.	2.76
Nitrogen content, percent wt.	0.10
Wax content, percent wt.	10.6
Hydrogenation conditions employed:	
Catalyst	(1)
Pressure, p.s.i.g.	1500
Temperature, ° F.	770
Space velocity, v./v./h.	1.5
Recycle gas rate, s.c.f./bbl.	7500
Recycle gas scrubbing	(2)
Hydrogen consumption, s.c.f./bbl.	750
¹ CoMo/silica alumina. Catalyst composition was:	
Cobalt	1.75 percent wt.
Molybdenum	12.7 percent wt.
Silicon	9.0 percent wt.
Alumina	Balance.
Surface area	331 m. ² /g.
Form	½" extrudates.
² Ammonia removal.	
The product from this stage after topping to remove	

material beoling below 240° C. had the following inspection data:

Yield of product boiling above 371° C., percent wt.	74.1
Kinematic viscosity at 100° F., cs.	22.3
Kinematic viscosity at 210° F., cs.	4.29
Viscosity index	111
Pour point, ° F.	90
Sulphur content, percent wt.	0.06
Wax content, percent wt.	13.8
Wax melting point, ° F.	124

The topped product was then passed to a catalytic dewaxing stage using a platinum-decaionised mordenite catalyst having the following composition (calculated on material stable at 550° C.)

Platinum	percent wt.	0.62
Aluminium	do.	5.08
Silicon	do.	40.3
Sodium	do.	0.93
SiO ₂ :Al ₂ O ₃ mol ratio		15.3
Surface area	m. ² /g.	425
Pore volume	ml./g.	0.22

The space velocity and gas rate were varied during the run to assess the effect of these variables. Temperature and pressure were kept constant. After the catalytic dewaxing the product was distilled to remove material boiling below 371° C. and the material boiling above 371° C. was vacuum distilled to give 6 fractions. The process conditions used, the yields obtained and inspection data on the 6 fractions are given in Table 2 below.

Inspection data on fractions ex residue >371° C.:											
<hr/>											
1,000 700											
<hr/>											
4.0											
5,000											
<hr/>											
-25											
75.0											
<hr/>											
50.8											
<hr/>											
V, 210° F., cst.	VI	Pour point, ° F.	Yield on residue, percent wt.	Yield on feed, percent wt.	KV, 100° F., cst.	KV, 210° F., cst.	VI	Pour point, ° F.			
4.39	22	-55	17.1	8.7	35.51	4.89	44	-50			
4.97	42	-45	16.8	8.5	40.39	5.36	59	-40			
5.72	63	-40	16.9	8.6	46.91	5.96	69	-15			
6.84	74	-25	16.7	8.5	57.49	6.95	80	0			
8.19	89	0	16.7	8.5	69.73	8.07	89	15			
11.85	96	25	15.8	8.0	108.8	11.35	99	30			

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Fractions 5 and 6 of Table 2 are products of increased VI and improved pour points according to the invention. Fractions 1-4 which have improved pour points but not increased VI are particularly suitable for use as very low pour point industrial oils (e.g. transformer oils).

EXAMPLE 2

A Kuwait wax distillate fraction having the inspection data shown in Table 3 below was hydrogenated under the conditions shown in Table 3.

TABLE 3

Inspection data on feed:	
Kinematic viscosity at—	
100° F., cs.	40.17
210° F., cs.	5.63
Viscosity index	82
Pour point, ° F.	85
Sulphur content, percent wt.	2.42
Nitrogen content, percent wt.	0.08
Wax content, percent wt.	12.6

Hydrogenation conditions employed:

Catalyst	(1)
Pressure, p.s.i.g.	1500
Temperature, ° F.	760
Space velocity, v./v./h.	1.5
Recycle gas rate, s.c.f./bbl.	7,500
Recycle gas scrubbing	(2)
Hydrogen consumption, s.c.f./bbl.	940
Liquid recovery, percent wt.	96.5

¹ CoMo/SiAl (as in Example 1).² Ammonia removal.

The total liquid product from this hydrogenation step was then stripped of H₂S and passed to a catalytic dewaxing stage using platinum-decationized mordenite (as in Example 1) as the dewaxing catalyst.

After the catalytic dewaxing step the product was distilled to remove material boiling below 371° C., and the material boiling above 371° C. was vacuum distilled to give 5 fractions.

The dewaxing conditions used, the yields obtained and the inspection data on the 5 fractions are given in Table 4 below.

TABLE 4

Conditions for catalytic dewaxing:		1,500									
Pressure, p.s.i.g.-----											
Temperature, ° F.-----	700	700									
Space velocity, v./v./h.-----	4.0	6.0									
Gas rate, s.c.f./bbl.-----	10,000	10,000									
Liquid yield on dewaxing.-----	73.3	75									
Inspection data on dewaxed liquid product 371° C.:											
Pour point, ° F.-----	-10	15									
Yield of residue 371° C. on feed, percent weight.-----	47.4	51.0									
						</					

Again it will be seen that fractions of high VI and low pour point are obtained. From the fractions on the right hand side of the table, 32.5 percent wt. (based on the original wax distillate feedstock) of a lubricating oil of 5.4 cs. viscosity, 100 VI and 15° F. pour point can be obtained, which (apart from a marginally high pour point) is within the specification for 65/1000 grade lubricating oil.

We claim:

1. A wholly catalytic process for the production from a wax-containing petroleum feedstock boiling in the range 350–600° C. and having a viscosity index of from 30 to 90, of an improved lubricating oil boiling above 350° C. and having a reduced pour point and an improved viscosity index of from 75 to 140, in the absence of any solvent refining for the removal of aromatics and wax from the feedstock, said process comprising the steps of passing the feedstock, together with the hydrogen, over a viscosity index improvement catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the Periodic Table, on a refractory oxide support, at a temperature of from 650 to 850° F., a pressure of from 1000 to 3000 p.s.i.g., a space velocity of 0.1–5 v./v./hr. and a hydrogen treating rate of 2000–10,000 s.c.f./b. at which there is produced a material of reduced aromatic content boiling above 350° C. and having an improved viscosity index of from 90–160, thereafter passing said material of improved viscosity index, together with hydrogen, over a dewaxing catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the

Periodic Table incorporated with a crystalline decationised mordenite having a sodium content of less than 2 percent wt., said mordenite having a SiO₂:Al₂O ratio of from 14:1 to 50:1, at a temperature of from 450 to 950° F., a pressure of 100 to 3000 p.s.i.g., a space velocity of 0.1–20.0 v./v./hr. and a hydrogen treating rate of 500–30,000 s.c.f./b. at which there is produced a material of reduced wax content boiling above 350° C. and having a reduced pour point and an improved viscosity index of from 75 to 140; and recovering a fraction having an improved viscosity index of at least 7 points higher than that of the original feedstock and a pour point of at least below 15° F.

2. A process as claimed in claim 1 wherein the feedstock is a vacuum distillate fraction boiling within the range 350–600° C. having a VI of 30 to 90 and the viscosity index improvement step is carried out at 700–850° F., 1000–3000 p.s.i.g., 0.1–5 v./v./hr. and 2000–10,000 s.c.f./b. to a conversion of less than 30 percent wt. of products boiling below 350° C.

3. A process as claimed in claim 1 wherein the feedstock is a vacuum distillate fraction boiling within the

range 450–600° C. having a VI of 30 to 90 and the viscosity index improvement step is carried out at 725–850° F., 1000–3000 p.s.i.g., 0.1–5 v./v./hr. and 2000–10,000 s.c.f./b. to a conversion of 30 to 80 percent wt. of products boiling below 350° C.

4. A process as claimed in claim 1 wherein the viscosity index improvement step catalyst comprises from 2–25 percent wt. of molybdenum and 1–15 percent wt. of nickel and/or cobalt on an alumina:silica support having from 50–90 percent wt. of alumina and 10–50 percent wt. of silica.

5. A process as claimed in claim 1 wherein the catalytic dewaxing step is carried out at 500–800° F., 500–3000 p.s.i.g., 0.5–10 v./v./hr. and 5000–15000 s.c.f./b. using a catalyst comprising from 0.01–10 percent wt. of a platinum group metal incorporated with the crystalline decationised mordenite having a sodium cation content of less than 2 percent wt.

6. A wholly catalytic process for the production of an improved lubricating oil boiling above 350° C. and having a reduced pour point and an improved viscosity index of from 75 to 140, from a wax-containing petroleum feedstock boiling in the range 350–600° C. and having a viscosity index of from 30 to 90, in the absence of any solvent refining of the feedstock for the removal of aromatics and wax from the feedstock, said process comprising the steps of passing said feedstock, together with the hydrogen, over a dewaxing catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the Periodic Table incorporated with a crystalline decationised mordenite having a sodium con-

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tent of less than 2 percent wt., said mordenite having a $\text{SiO}_2:\text{Al}_2\text{O}_3$ ratio of from 14:1 to 50:1, at a temperature of from 450 to 950° F., a pressure of 100 to 3000 p.s.i.g., a space velocity of 0.1–20.0 v./v./hr. and a hydrogen treating rate of 500–30,000 s.c.f./b., at which there is produced a material of reduced wax content boiling above 350° C. and having a reduced pour point, thereafter passing the feedstock having a reduced pour point, together with hydrogen, over a viscosity index improvement catalyst comprising one or more hydrogenating components selected from Groups VIa and VIII of the Periodic Table, on a refractory oxide support, at a temperature of from 650 to 850° F., a pressure of from 1000 to 3000 p.s.i.g., a space velocity of 0.1–5 v./v./hr. and a hydrogen treating rate of 2000–10,000 s.c.f./b., at which there is provided a material of reduced aromatic

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content boiling above 350° C. and having an improved viscosity index of from 75–140, and recovering a fraction having an improved viscosity index of at least 7 points higher than that of the original feedstock and a pour point of at least below 15° F.

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HERBERT LEVINE, Primary Examiner

U.S. Cl. X.R.

208—18, 111, DIG. 2

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,663,423 Dated May 16, 1972

Inventor(s) Robert Neil Bennett, Richard Nigel Parker and
Bernard Whiting Burbidge

It is certified that error appears in the above-identified patent
and that said Letters Patent are hereby corrected as shown below:

Col. 3, Line 29,
for "90" read -- 95 --

Col. 4, Line 18,
for "1Me₂/n" read -- 1Me₂/_n --

Col. 7, Table 4,
right-hand column
headed "Pour Point °F",
for "-70" read -- -10 --

Col. 8, Line 45,
for "30 o 90" read -- 30 to 90 --

Signed and sealed this 5th day of December 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents