



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : C10G 45/58, G01N 33/28, C10G 45/72	· A1	(11) International Publication Number: WO 00/08116 (43) International Publication Date: 17 February 2000 (17.02.00)
(21) International Application Number: PCT/US99/17265 (22) International Filing Date: 30 July 1999 (30.07.99) (30) Priority Data: 09/128,727 4 August 1998 (04.08.98) US (71) Applicant: EXXON RESEARCH AND ENGINEERING COMPANY [US/US]; 180 Park Avenue, P.O. Box 390, Florham Park, NJ 07932-0390 (US). (72) Inventors: ALDRICH, Haven, Scott; 10 Dartmouth Road, An- nandale, NJ 08801 (US). SZOBOTA, John, Stephen; 16 Alexandria Road, Morristown, NJ 07960 (US). WITTEN- BRINK, Robert, Jay; 6018 Riverchase Trail, Kingwood, TX 77345 (US). (74) Agents: BAKUN, Estelle, C. et al.; Exxon Research and Engineering Company, P.O. Box 390, Florham Park, NJ 07932-0390 (US).		(81) Designated States: CA, JP, SG, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: A PROCESS FOR PRODUCING A LUBRICANT BASE OIL HAVING IMPROVED OXIDATIVE STABILITY		
(57) Abstract <p>The instant invention is directed to a process for producing an improved lubricating base stock having a preselected desired oxidation stability comprising the steps of: (a) hydroisomerizing a hydrocarbon wax to obtain a lubricating base stock, (b) irradiating, within the frequency of about 4600 to about 3500 cm⁻¹ or about 1300 to about 600 cm⁻¹, said lubricating base stock produced from hydroisomerization of said hydrocarbon wax, (c) measuring the absorption spectrum, within said frequency range of step (b), of said lubricating base stock using FT-IR, (d) converting said absorption spectrum into a number representative of the viscosity index of said lubricating base stock of step (a), (e) comparing the viscosity index produced in step (c) to a preselected viscosity index correlating to a preselected desired oxidative stability for said lubricating base stock of step (a), and then, (f) modifying said hydroisomerization of said hydrocarbon wax to increase the production of lubricating base stock having said preselected desired oxidative stability of step (e).</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece			TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon	KR	Republic of Korea	PL	Poland		
CN	China	KZ	Kazakhstan	PT	Portugal		
CU	Cuba	LC	Saint Lucia	RO	Romania		
CZ	Czech Republic	LI	Liechtenstein	RU	Russian Federation		
DE	Germany	LK	Sri Lanka	SD	Sudan		
DK	Denmark	LR	Liberia	SE	Sweden		
EE	Estonia			SG	Singapore		

**A PROCESS FOR PRODUCING A LUBRICANT
BASE OIL HAVING IMPROVED OXIDATIVE STABILITY**

FIELD OF THE INVENTION

The instant invention is directed to a process for the production of high quality lubricant base oils having superior oxidation stability using molecular structure control and FT-IR chemometric controls.

BACKGROUND OF THE INVENTION

Studies to date have shown that lubricants prepared via the hydroisomerization of Fischer-Tropsch wax, are equivalent to synthetic lubricants, such as polyalphaolefins (PAO, oligomers of α -decene) in all areas of performance except low temperature performance and base oil oxidation stability. Therefore, a process is needed which is capable of increasing the oxidation stability of hydroisomerized Fischer-Tropsch waxes.

At present, the API "SH" rating is currently employed for passenger car motor oils for use in gasoline engines which represents a significant increase in the service requirements of lubricants from past ratings (e.g., API SF or API SG). Thus, there is a continuing need for lubricants with superior performance characteristics.

One of the performance characteristics which is of greatest significance is the viscosity index (VI). This represents the extent to which the viscosity of a lubricant varies with temperature. Lubricants of high VI change relatively little in viscosity as temperature increases, at least as compared to lubricants of lower VI. Since retention of viscosity at higher temperatures is a

desirable characteristic, high viscosity index is desirable. Satisfactory viscosity properties may be conferred either by suitable choice of the lubricant base stock or by the use of VI improvers which are generally high molecular weight polymers.

The extent to which VI properties can be varied by the use of these improvers is, however, limited because not only are large amounts of improver expensive but the improvers are subject to degradation in use so that service life of lubricants containing large amounts of improver may be limited. This implies that improvements in the VI of the base stock are desirable. However, such improvers will have no effect on the oxidation stability of a base stock.

Spectroscopic methods have been used in the art. U.S. Patent 5,475,612 is directed to determining the properties of a liquid hydrocarbon blend from the near IR spectrum of the components of the blend. The method involves determining the absorbance, at a minimum number of frequencies, for the components of arbitrary mixtures. A spectral mixture index is then determined for each component and property. The desired property sought is then calculated by a linear expression.

U.S. Patent 5,419,185 is directed to a method and apparatus for optimizing the extraction of aromatics from waxy distillates and the dewaxing of waxy raffinates in the manufacture of lubricating oils. The method involves irradiating a waxy raffinate, measuring the absorption spectrum and converting the spectrum into a number representative of the VI of the dewaxed oil produced from the waxy raffinate after dewaxing. The aromatics extraction of a waxy distillate which produces the waxy raffinate is then reduced or increased in severity in order to approach a desired VI.

Other articles include "An analysis of petroleum fuels by midband (4000-400/cm) infrared spectroscopy", Foder G. E. et al., SAE International Congress(Detroit 2/28-3/3/94) SAE Meeting Paper N 941019(1994)14P, ISSN 0148-7191 and "Multivariate Calibration in Fourier Transform Infrared Spectrometry for Prediction of Kerosene Properties", Garrigues S. et al., Anal. Chim. Acta., 317(1-3)95-105(1995) Chemical Abstracts ABSTR No. 92150 V124 N. 8 ISSN 0009-2258.

Several articles have been written on thermal diffusion of particular materials such as residual lube stocks (The Composition and Properties of Oil Fractions in Heavy Crudes from the Persian Deposits of Tartarstan, Petrova et al., Russian Academy of Sciences, Kazan Branch, Institute of Organic & Physical Chemistry, Khimiya i Tekhnologiya Topliv I Masel N.5 33-35 (1995) ISSN 0023-1169); MS-8 Oils (Oxidation Tendency of Fractions from the thermal diffusion separation of white oil, Klimov, A.K. et al., Neftepererab. Neftekhim (Moscow) (1979), (1), 23-5 CODEN:NNNSAF; ISSN 0028-1190: Deparaffinated heavy oil, refined neutral II and hydrofined neutral III (Properties of Lubricating oils produced by thermal diffusion, Christakudis et al., Schmierstoffe Schmierungstech. (1969), No. 35, 32-41, CODEN:SSWTBI; and petroleum lube oils (Composition and oxidation of petroleum fractions, Cranton, G.E., Thermochem Acta (1976), 14(1-2), 201-8 CODEN:THACAS.

SUMMARY OF THE INVENTION

The instant invention is directed to a process for producing a lubricating base stock having a preselected desired oxidation stability comprising the steps of:

- 4 -

- (a) hydroisomerizing a hydrocarbon wax to obtain a lubricating base stock,
- (b) irradiating, within the frequency of about 4600 to about 3500 cm^{-1} or about 1300 to about 600 cm^{-1} , said lubricating base stock produced from hydroisomerization of said hydrocarbon wax,
- (c) measuring the absorption spectrum, within said frequency range of step (b), of said lubricating base stock using FT-IR,
- (d) converting said absorption spectrum into a number representative of the viscosity index of said lubricating base stock of step (a),
- (e) comparing the viscosity index produced in step (c) to a preselected viscosity index correlating to a preselected desired oxidative stability for said lubricating base stock of step (a), and then,
- (f) modifying said hydroisomerization of said hydrocarbon wax to increase the production of lubricating base stock having said preselected desired oxidative stability of step (e).

DETAILED DESCRIPTION OF THE INVENTION

Applicants have discovered that there exists a particular branched hydrocarbon mixture having a degree of branchiness which confers highly improved oxidative stability to a hydroconverted hydrocarbonaceous feed stock. A highly improved product can be obtained from a fractionated hydroisomerized hydrocarbon feed, for example, a hydroisomerized Fischer-Tropsch wax.

The instant invention describes a process for improving the oxidation stability of lubricant base oils produced from hydroisomerized hydrocarbon waxes. The base oils are preferably derived from a synthetic wax produced using a Fischer-Tropsch (FT) process from a methane derived syngas.

Applicants have observed that a particular fraction of the 700°F+ fraction of a hydroisomerized FT wax having a defined degree of branchiness is more oxidatively stable than components of higher and lower degrees of branchiness. Combining this finding with FT-IR and chemometrics, then allows the skilled artisan to control the isomerization to increase the yield of component having the most beneficial oxidative stability.

All that is necessary to carry out the process of the instant invention is that a target or desired viscosity index be known. This is the preselected VI of the instant invention. The target or desired VI can be determined by any means known to the skilled artisan. The target or desired VI will preferably be correlated with a target or desired oxidative stability by methods known to the skilled artisan. This target or desired oxidative stability is the preselected oxidative stability of the instant invention. Once a target VI correlating to a target oxidative stability is known, the process is conducted.

Applicants separated the 700°F+ fraction of a hydroisomerized FT wax using thermal diffusion separation technique which separate hydrocarbons as a function of structure or molecular shape. Consequently, a mixture of hydrocarbons that range from normal paraffins to highly branched paraffins are separated such that the normal paraffins are eluded first while the most highly branched are eluded last. Branchiness increases as one proceeds to higher ports. One skilled in the art would expect that the least highly branched paraffins, those

with the highest VI, would show the greatest oxidation stability. Hence, the least highly branched paraffins would be selected for use as base stocks. Applicants have found that this is not the case. Applicants believe that a particular level, or mixture of branchiness, can retard the level of oxidation by interfering with the ability of hydroperoxides to react with other reactive hydrogens through steric blocking. Therefore, the random branchiness which result in tertiary hydrogens more reactive in an oxidation environment is being counterbalanced. This is unexpected and previously unknown. Though thermal diffusion was used to separate the fractions of the hydroisomerized wax, any method of separation based on molecular shape can be utilized.

By measuring the VI and oxidative stability, using known techniques, of each of the fractions produced by the thermal diffusion, applicants were able to identify a fraction comprising at least 50% of a mixture of branched paraffins, wherein said paraffins are paraffins having a carbon chain length of about C₂₀ to about C₄₀, having a molecular weight ranging from about 280 to about 562, and a boiling range of about 650°F to about 1050°F, and wherein said branched paraffins contain up to 4 methyl branches, and wherein the free carbon index of said branched paraffins is at least about 3. This particular fraction has a higher oxidative stability than the other fractions separated. The fraction will also preferably have a number of pendant carbon of about 4 or less. The number of pendant carbon is defined as the number of alkyl groups on the $\epsilon^{(+)}$ carbons. Thus pendent carbons will be present in the middle of the carbon chain at least $\epsilon^{(+)}$ carbons from each end of the chain. Knowing the VI of this material, applicants are then able to utilize FT-IR and chemometrics to measure the VI of the stream produced from the hydroisomerization of a Fischer-Tropsch wax, compare this measured VI to the target or desired VI, corresponding to the above fraction, and modify the hydroisomerization process to increase the concentra-

tion of the branched paraffin mixture corresponding to the target VI and hence the desired oxidation stability. Thus, modification of the molecular structure of wax isomerase produced from the hydrocarbon wax, by modifying the hydroisomerization, allows the skilled artisan to produce a product of target VI and oxidative stability. Though applicants were comparing the VI of the hydroisomerized wax to the fraction having the above characteristics, the instant method can be utilized to increase the concentration of product having any given VI. All that is necessary is that a desired or target VI be available for comparison. The desired or target VI may correspond to any oxidative stability being sought. Preferably, the VI will correspond to an improved oxidative stability.

The base stock applicants sought to produce has a "Free Carbon Index" (or FCI) of at least about 3. The term "Free Carbon Index" is a measure of the number of carbons in an iso-paraffin that are located at least 4 carbons from a terminal carbon and more than 3 carbons away from a side chain. The FCI of an isoparaffin can be determined by measuring the percent of methylene groups in an isoparaffin sample using ^{13}C NMR (400 megahertz); multiplying the resultant percentages by the calculated average carbon number of the sample determined by ASTM Test method 2502 and dividing by 100.

The FCI is further explained as follows. The base stock is analyzed by ^{13}C NMR using a 400 MHz spectrometer. All normal paraffins with carbon numbers greater than C_9 have only five non-equivalent NMR absorptions corresponding to the terminal methyl carbons (α) methylenes from the second, third and fourth positions from the molecular ends (β , γ , and δ respectively), and the other carbon atoms along the backbone which have a common chemical shift (ϵ). The intensities of the α , β , γ and δ are equal and the intensity of the ϵ depends on the length of the molecule. Similarly the side

branches on the backbone of an iso-paraffin have unique chemical shifts and the presence of a side chain causes a unique shift at the tertiary carbon (branch point) on the backbone to which it is anchored. Further, it also perturbs the chemical sites within three carbons from this branch point imparting unique chemical shifts (α' , β' and γ').

The Free Carbon Index (FCI) is then the percent of ϵ methylenes measured from the overall carbon species in the ^{13}C NMR spectra of the base stock, divided by the average carbon number of the base stock as calculated from ASTM method 2505, divided by 100.

If desirable, the skilled artisan could determine which fractions have equivalent VI and oxidative stability of a given PAO oil, run the process and prepare a lubricating oil from a Fischer-Tropsch wax which could be substituted for the given PAO. The prepared oil would then behave equivalently in all areas of performance including base oil oxidative stability.

Though the above discussion and the discussion to follow is in the context of Fischer-Tropsch waxes, those skilled in the art can readily see that the instant process can be compared to any wax hydroisomerization processes. All that is necessary is that a desired VI be known or determined.

The hydroisomerized waxes utilizable in the instant invention may originate from any number of sources including petroleum raffinates. Synthetic waxes from Fischer-Tropsch processes may be used, as may be waxes recovered from the solvent or autorefrigerative dewaxing of conventional hydrocarbon oils, or mixtures of these waxes. Waxes from dewaxing conventional hydrocarbon oils, commonly called slack waxes may also be used.

Though the waxes can be hydroisomerized by conventional prior art methods, typically the hydroisomerization is conducted over a catalyst containing a hydrogenating metal component-typically one from Group IV, or Group VIII of the Periodic Table, or mixtures thereof. The reaction is conducted under conditions of temperature between about 500 to 750°F (preferably 570 to 680°F) and pressures of from 500 to 3000 psi H₂ (preferably 500-1500 psi H₂), at hydrogen gas rates from 1000 to 10,000 SCF/bbl, and at space velocities in the range of from 0.1 to 10 v/v/hr, preferably from 0.5 to 2 v/v/hr.

Following the hydroisomerization, the isomerate may undergo hydrogenation to stabilize the oil and remove residual aromatics. The resulting product may then be fractionated into lubricants and fuels. Typically, the initial boiling point of the lubricants fraction will boil in the range of about 650°F to 700°F or higher. It is the lubricant fraction that is utilized in the instant invention and which is irradiated. For Fischer-Tropsch waxes, the 700°F+ fraction will typically be used.

The chemometric techniques utilized herein are known to the skilled artisan and are merely mathematical manipulations for converting an absorption spectrum into a number representative of VI. For example Multivariate Partial Least Squares (PLS), Multivariate Principal Component Regression (PCR), first derivative without baseline correction, mean centering, etc., can be used. PLS and PCR are preferred.

Though thermal diffusion was utilized to separate the hydroisomerized wax into fractions, any technique known to the skilled artisan which separates based on molecular structure can be utilized.

The method for monitoring the thermal diffusion fractions for oxidation stability can be any method known to those skilled in the art. For example, measurement of the oxidation induction time using high pressure differential scanning calorimetry can be used. Likewise, NMR techniques can be used to determine the molecular structure of the fractions.

Once it is determined which cuts of the thermally diffused feed stream have the most beneficial oxidation stability or any desired oxidation stability, the corresponding VI's are then determined. It is then possible to utilize FT-IR techniques to accurately measure the VI of these particular cuts and then to predict corresponding oxidation stability such cuts will have. Utilizing this information, it then becomes possible to monitor, on-line or at line, the hydro-isomerization process producing the Fischer-Tropsch feed stream, alter the process conditions to obtain the FT-IR spectra of the most beneficial cuts, and thereby selectively produce lubricants with enhanced oxidation stability. It is within the skill in the art to modify the process conditions to obtain the FT-IR spectra corresponding to the product desired.

The wavelength range for irradiation of the isomerate can be performed in one of two regions. The first region is about 4600 to about 3500, preferably about 4600 to about 3950, and the second region is about 1300 to about 600, preferably about 850 to about 650 cm^{-1} . Preferably a combination of the two wavelength regions will be utilized.

The following examples are merely for illustration and are not meant to be limiting in any way.

Example 1

A sample of Fischer-Tropsch wax was subjected to hydroisomerization under hydroconversion conditions which were sufficient enough to cause $\cong 50\%$ conversion of the 700°F+ wax into high quality liquid transportation fuels boiling below 700°F. The resulting 700°F+ hydroisomerate was then fractionated into a 700-950°F fraction. This fraction was then separated into 10 cuts using thermal diffusion (P1-P10). The feedstock and the cuts were evaluated to measure their oxidation stabilities using High Pressure Differential Scanning Calorimetry (HPDSC). Each sample was blended with a constant amount of dioctyldiphenyl amine antioxidant. The concentration of antioxidant was 0.5 wt% on the base oil in each case. The samples were evaluated in open aluminum pans under 200 psi of O₂ at constant temperature and the stability was measured by the oxidation induction time (OIT) in minutes. The longer the OIT for a cut at a fixed temperature, the more stable is that lubricant thermal diffusion cut. Each thermal diffusion cut was evaluated at 170, 180, and 190°C. The relative stability is determined by comparing OITs at a fixed temperature. The stability of the cuts was not equal and showed an increase between ports 2 and 6 followed by a steady decrease after that.

The results are shown in Table I.

TABLE I

Port Number	HPDSC Isothermal Temperature, °C	Oxidation Induction Time (Minutes)
1	170	21.0
1	180	14.4
1	190	5.8
2	170	32.4
2	180	14.8
2	190	8.5
3	170	25.2
3	180	15.1
3	190	17.6
4	170	37.4
4	180	20.7
4	190	9.9
5	170	34.6
5	180	19.0
5	190	9.5
6	170	32.8
6	180	16.0
6	190	7.6
7	170	25.1
7	180	13.3
7	190	6.4
8	170	16.4
8	180	10.1
8	190	5.8
9	170	15.6
9	180	10.0
9	190	5.9
10	170	15.3
10	180	10.5
10	190	6.9

FT-IR spectra recorded for 6 of the thermal diffusion cuts encompassing the range of viscosity and viscosity index values (P1, P3, P5, P7, P9, and P10, P = Port) can be used to predict the viscosity and viscosity indices. Spectra were recorded using a 1.0 mm path length standard liquid IR cell fitted with KBr windows. Spectra were obtained at 2 cm^{-1} resolution utilizing 100 scans for both sample and background. A Multivariate Partial-Least Squares Chemometric methodology was used to generate a calibration capable of predicting viscosity and viscosity index to a high degree of accuracy. The spectral regions found to provide the best fit for this analysis were from 4600 to 3950 cm^{-1} and 850 to 650 cm^{-1} . These regions exhibit features due to both methylene and methyl groups, consistent with degree of branchiness being an important factor.

Tables II through IV show the viscosity at 40 and 100°C , viscosity index, number of chemometric factors used and correlation coefficients and root mean square deviation (RMSD) for two different predictions of viscosity and VI-self prediction, i.e., sample predicted was part of calibration set, and Leave-one-out Prediction, i.e., sample predicted was not included in calibration set (this is considered to give a better measure of model prediction capability for real unknown samples).

- 14 -

TABLE II

FT-IR PREDICTIONS OF VISCOSITY AT 40°C
3 FACTORS USED

Sample Port Number	Actual Viscosity	Self Prediction Viscosity	Leave-one-out Prediction Viscosity
1	23.91	23.969	24.024
3	27.023	27.047	26.988
5	28.75	28.809	28.830
7	30.44	30.508	30.753
9	32.62	32.743	32.882
10	33.94	33.812	32.320
RMSD		0.11305	0.32736
Correlation Coefficient		0.99886	0.99044

TABLE III

FT-IR CHEMOMETRIC PREDICTIONS OF VISCOSITY AT 100 °C
3 FACTORS USED

Sample Port Number	Actual Viscosity	Self Prediction Viscosity	Leave-one-out Prediction Viscosity
1	5.20	5.203	5.153
3	5.48	5.470	5.476
5	5.63	5.631	5.598
7	5.79	5.803	5.836
9	6.04	6.030	6.019
10	6.12	6.123	6.107
RMSD		0.0081523	0.0316
Correlation Coefficient		0.99933	0.99005

TABLE IV

FT-IR CHEMOMETRIC PREDICTIONS OF VISCOSITY INDEX
1 FACTOR USED

Sample Port Number	Actual Viscosity	Self Prediction Viscosity	Leave-one-out Prediction Viscosity
1	156	155.19	153.16
3	143	144.36	144.76
5	140	140.25	140.30
7	135	135.11	135.14
9	133	130.74	129.70
10	128	129.32	130.10
RMSD		1.2519	2.1041
Correlation Coefficient		0.98036	0.94452

Because the viscosities and VI of the thermal diffusion cuts can be predicted based on FT-IR data, the oxidation stabilities can also be predicted as both are a function of the branchiness of the 700 F+ LUBE-X fraction. In manufacturing operations, in which the temperature and space velocity of feed over catalyst are controllable, FT-IR can be used as an on line or at-line analytic to maximize the content of the feed stream that has the spectral properties corresponding to the branchiness that is responsible for the superior oxidation stability.

The following tables show the viscosity at 40°C, 100°C, viscosity index, number of chemometric factors used and correlation coefficients and RMSD for two different predictions of viscosity and VI-self prediction (i.e., the sample predicted was part of the calibration set) and leave-one-out prediction (i.e., the sample predicted was not included in the calibration set (considered to give a better measure of model prediction capability for real unknown examples).

Multivariate chemometric methodologies [partial least squares and principal component regression] were utilized to generate a calibration capable of predicting viscosity and viscosity index to a high degree of accuracy. Various region of the spectrum which exhibit features due to both methylene and methyl groups were utilized. Pre-processing included mean centering and baseline correction. Tabulated below are self-prediction (i.e., sample predicted was part of the calibration set) and correlation coefficient (r^2) obtained.

Spectral Region 4600-3510 cm^{-1}	Multivariate methodology	PLS
--	---------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r^2)
Viscosity @ 40°C	1	0.953
Viscosity @ 100°C	1	0.937
Viscosity Index	1	0.970

Spectral Region 4600-3900 cm^{-1}	Multivariate methodology	PLS
--	---------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r^2)
Viscosity @ 40°C	1	0.953
Viscosity @ 100°C	1	0.934
Viscosity Index	2	0.963

Spectral Region 1300-600 cm ⁻¹	Multivariate methodology	PLS
---	------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	4	0.953
Viscosity @ 100°C	3	0.993
Viscosity Index	1	0.947

Spectral Region 850-600 cm ⁻¹	Multivariate methodology	PLS
--	------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	3	0.965
Viscosity @ 100°C	3	0.988
Viscosity Index	1	0.949

Spectral Region 1200-850 cm ⁻¹	Multivariate methodology	PLS
---	------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	4	0.986
Viscosity @ 100°C	2	0.984
Viscosity Index	1	0.940

Spectral Region 1000-850 cm ⁻¹	Multivariate methodology	PLS
---	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	3	0.960
Viscosity @ 100°C	3	0.967
Viscosity Index	2	0.946

Spectral Region 1360-650 cm ⁻¹	Multivariate methodology	PLS
---	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	2	0.977
Viscosity @ 100°C	3	0.978
Viscosity Index	1	0.925

Spectral Region 4600-3510 cm ⁻¹	Multivariate methodology	PCR
--	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	3	0.923
Viscosity @ 100°C	3	0.916
Viscosity Index	3	0.962

Spectral Region 4600-3900 cm ⁻¹	Multivariate methodology	PCR
--	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	2	0.935
Viscosity @ 100°C	2	0.916
Viscosity Index	2	0.962

Spectral Region 1300-600 cm ⁻¹	Multivariate methodology	PCR
---	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	1	0.945
Viscosity @ 100°C	1	0.921
Viscosity Index	1	0.947

Spectral Region 850-650 cm ⁻¹	Multivariate methodology	PCR
--	---	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	1	0.942
Viscosity @ 100°C	1	0.917
Viscosity Index	1	0.949

- 20 -

Spectral Region 1200-850 cm ⁻¹	Multivariate methodology	PCR
---	-------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	1	0.945
Viscosity @ 100°C	1	0.923
Viscosity Index	1	0.940

Spectral Region 1000-850 cm ⁻¹	Multivariate methodology	PCR
---	-------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	2	0.952
Viscosity @ 100°C	2	0.954
Viscosity Index	2	0.943

Spectral Region 1360-650 cm ⁻¹	Multivariate methodology	PCR
---	-------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r²)
Viscosity @ 40°C	1	0.972
Viscosity @ 100°C	1	0.957
Viscosity Index	1	0.925

Utilization of other subregions within the 4600-3510 cm⁻¹ give rise to correlation coefficients of similar magnitude. Baseline correction gave better

correlation coefficients than utilization of mean centering and 1st derivative (without baseline correction) pre-processing.

Utilization of combination of two regions (one from 4600-3510 cm^{-1} and the other from the 1360-650 cm^{-1} region gave rise to significantly improved correlation as shown below.

Spectral Region 4600-3950 cm^{-1} and 850-650 cm^{-1}	Multivariate methodology	PLS
--	-------------------------------------	------------

Predicted Property	# of Factors	Correlation Coefficient (r^2)
Viscosity @ 40°C	3	0.999
Viscosity @ 100°C	3	0.999
Viscosity Index	1	0.980

CLAIMS:

1. A process for producing a lubricating base stock having a desired preselected oxidation stability comprising the steps of:

(a) hydroisomerizing a hydrocarbon wax to obtain a lubricating base stock,

(b) irradiating, within the frequency of about 4600 to about 3500 cm^{-1} or about 1300 to about 600 cm^{-1} , said lubricating base stock produced from hydroisomerization of said hydrocarbon wax,

(c) measuring the absorption spectrum, within said frequency range of step (b), of said lubricating base stock using FT-IR,

(d) converting said absorption spectrum into a number representative of the viscosity index of said lubricating base stock of step (a),

(e) comparing the viscosity index produced in step (c) to a preselected viscosity index correlating to a preselected desired oxidative stability for said lubricating base stock of step (a), and then,

(f) modifying said hydroisomerization of said hydrocarbon wax to increase the production of lubricating base stock having said preselected desired oxidative stability of step (e).

2. The process of claim 1 wherein said hydrocarbon wax is a Fischer-Tropsch wax.

3. The process of claim 1 wherein when said wax is a Fischer-Tropsch wax, the lubricating base stock having the preselected viscosity index correlating to the preselected desired oxidative stability of step (e) is a mixture of branched paraffins, wherein said paraffins are paraffins having a carbon chain length of about C₂₀ to about C₄₀, having a molecular weight ranging from about 280 to about 562, and a boiling range of about 650°F to about 1050°F, and wherein said branched paraffins contain up to 4 methyl branches, and wherein the free carbon index of said branched paraffins is at least about 3.

4. The process of claim 1 wherein said hydrocarbon wax is selected from the group consisting of petroleum raffinates, slack waxes, Fischer-Tropsch waxes and mixtures thereof.

5. The process of claim 1 wherein said preselected viscosity index correlating to a preselected desired oxidative stability is determined by separating, based on molecular structure, said hydroisomerized hydrocarbon wax of step (a) into a plurality of fractions and determining the viscosity index and corresponding oxidative stability of each of said separated fractions to identify the fractions having a desired viscosity index and oxidative stability.

6. The process of claim 5 wherein said separation of said hydroisomerized wax is conducted using thermal diffusion.

7. The process of claim 1 wherein a combination of said wavelengths of step (a) is utilized.

8. The process of claim 3 wherein said mixture of branched paraffins comprises paraffins having an average number of pendant carbons of 4 or less.

9. The process of claim 5 wherein said preselected desired oxidative stability is the highest oxidative stability.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/17265

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10G45/58 G01N33/28 C10G45/72

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 G01N C10G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, A	US 5 895 506 A (BERLOWITZ PAUL JOSEPH ET AL) 20 April 1999 (1999-04-20) -----	
A	US 5 475 612 A (ESPINOSA ALAIN ET AL) 12 December 1995 (1995-12-12) cited in the application -----	

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

23 November 1999

Date of mailing of the international search report

02/12/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

De Herdt, O

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/17265

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5895506 A	20-04-1999	WO 9948846 A	30-09-1999
US 5475612 A	12-12-1995	FR 2619624 A	24-02-1989
		FR 2632408 A	08-12-1989
		FR 2632409 A	08-12-1989
		AT 92629 T	15-08-1993
		AU 603920 B	29-11-1990
		AU 2095488 A	23-02-1989
		CA 1325732 A	04-01-1994
		DE 3882847 A	09-09-1993
		DE 3882847 T	18-11-1993
		EP 0305090 A	01-03-1989
		JP 1113636 A	02-05-1989
		JP 2880171 B	05-04-1999
		NO 300027 B	17-03-1997