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(54) **LIGHT OIL COMPOSITION**

(57) The invention provides a light oil composition that does not cause deterioration in a Nylon-based material. Specifically, the invention provides a light oil composition containing paraffin(s) at a concentration of 97%

by mass or more, wherein the content of isoparaffin(s) having 14 or fewer carbon atoms in the paraffin(s) is 10% by mass or less.

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Description

Technical Field

5 **[0001]** The present invention relates to a light oil composition, and in particular to a GTL (Gas to Liquid) light oil composition.

Background Art

10 **[0002]** There has been a lot of research relating to particulate matter (PM) undertaken since reports in the 1970s of the carcinogenic nature of particulate matter (PM) discharged from diesel vehicles. Measures to reduce PM include: for vehicles, developments such as increasing fuel injection to a higher pressure, and post-processing systems for exhaust gas; and for light oils there has been progress in reducing the sulfur content thereof. City light oil (Class-1), like that use in Sweden since 1993, is a representative example of a low sulfur light oil (see for example Non-Patent Document 1).

15 **[0003]** The use of GTL light oil has been receiving a lot of attention from the perspective of reducing the amount of PM discharge. GTL light oil is a synthetic oil fraction with a boiling point within the range of that of light oil obtained by: conversion of natural gas and heavy oil into a water gas; synthesis thereof using a Fischer-Tropsch reaction (FT synthesis); fractionating off the highly volatile components of this synthetic oil; and carrying out hydrocracking and isomerization as required.

20 **[0004]** Such a GTL light oil was produced in Germany during the Second World War, however production was halted thereafter. Interest in GTL light oils was rekindled in the late 1980s as the problems of environmental pollution became of global importance. Production of GTL light oil was restarted in 1992 by a South African company, Moss gas. Since 1998 when Melinda da Sirman of SwRI reported at an SAE international conference that GTL light oils have the lowest amounts of PM discharge (see for example Non-Patent Documents 2 and 3) the US Department of Energy, oil majors
25 etc. have shown interest therein, so that GTL plants are now to be built in every region of the world.

[0005] Global production of GTL light oils is currently at 100,000 barrels a day, and when all of the plants currently under construction are in operation in 2010 this amount is said to become 600,000 barrels a day (this is equivalent to the total consumption of light oils in Japan) (see for example Non-Patent Document 4).

30 Non-patent document 1: Sweden Class-1: R.F. Tucker, R.J. Stradling, P.E. Wolveridge, K.J. Rivers and A. Ubbens, The Lubricity of Deeply Hydrogenated Diesel Fuels - The Swedish Experience, SAE942016

Non-patent document 2: Noboru Kawada, Outlook for the Next Generation of Synthetic Fuel Oils (First) "Quality Trends in Petroleum Products, and Issues with Existing Refining Techniques", Petrotech, 23, (12)pp1061 to 1066

35 Non-patent document 3: Kaoru Fujimoto, Seiichi Kiryu, Outlook for the Next Generation of Synthetic Fuel Oils (Third) "Trends in Technical Developments for FT Synthesis", Petrotech, 24, (2)pp113 to 118

Non-patent document 4: Yukihiro Tsukasaki, Recent Trends in Vehicle GTL Fuels, Vehicle Technology, 55, (5), pp. 67 - 72, (2001)

Disclosure of Invention

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Problem to be Solved by the Invention

[0006] While it is thought that from now on GTL light oils will steadily become more widely used, the effect of GTL light oils on fuel system materials is hardly known at all. We have investigated the effect of GTL light oils on resins and rubbers used in the fuel systems of diesel vehicles. As a result of immersing resins and rubbers in GTL light oils and carrying out tensile tests thereon, we have determined that there is a large decrease in the extension of Nylon-66 that has been immersed in a standard GTL light oil. Since up to now Nylon has been widely known as a hydrocarbon-resistant material, the phenomenon of the large reduction in the extension of Nylon that has been immersed in a GTL light oil is a completely unexpected result.

50 **[0007]** There has not been an interval of time since GTL light oils have been used commercially, and so there are no reports of investigations into the effects of GTL light oils on fuel systems. Consequently the effect of GTL light oils on the deterioration of Nylon is completely unpredictable, and there is an urgent requirement to develop a GTL light oil that does not cause deterioration in Nylon, in light of the expected large increase in demand for GTL light oils.

55 **[0008]** Therefore, the objective of the present invention is to address the above issue. Namely, objective of the present invention is to provide a light oil composition that does not cause deterioration in Nylon-based materials.

Method of Solving the Problem

[0009] As a result of diligent research into solving the above problem, the inventors have arrived at the present invention and discovered that the above problem can be solved. Namely, the present invention is a light oil composition containing paraffin(s) at a concentration of 97% by mass or greater, with isoparaffin(s) having 14 or fewer carbon atoms at a concentration of 10% by mass or less of the above paraffin(s).

Effect of the Invention

[0010] According to the present invention, a light oil composition that does not cause deterioration in Nylon-based materials can be provided.

Brief Description of Drawings

[0011]

FIG. 1 is a diagram showing the dissolved oxygen amount before and after immersion in GTL light oils.

FIG 2 is a diagram showing the dissolved oxygen amount before and after immersion in paraffins.

FIG. 3 are diagrams showing total ion chromatograms using thermal desorption- GC/MS, (A) is Nylon that has been immersed in Test Sample - A, and (B) is Nylon that has been immersed in Test Sample - B.

FIG 4 are diagrams showing MALDI-MS mass spectra of Nylon, (A) is untreated Nylon, (B) is Nylon that has been immersed in Test Sample - B, (C) is Nylon that has been immersed in Test Sample - A, (D) is Nylon subjected to oxidizing treatment, and (E) is Nylon subjected to hydrolysis treatment.

FIG 5 is a diagram showing results of analysis of Nylons in the depth direction.

FIG. 6 are diagrams showing the results of IR imaging analysis of Nylons in the depth direction, (A) is Nylon that has been immersed in Test Sample - A, and (B) is Nylon that has been immersed in Test Sample - B.

FIG 7 are diagrams showing the results of analysis by gas chromatographic/ mass spectroscopic methods of model fuels before and after immersion, (A) is for types of paraffin, (B) is for oxidation products (alcohols).

FIG 8 is a diagram showing Nylon analyzed by thermal desorption -gas chromatographic/mass spectroscopic methods before and after immersion.

FIG 9 is a diagram showing the relationship between isoparaffin concentration and extension at breakage.

Best Mode for Carrying Out the Invention

[0012] The present invention is a light oil composition, and in particular, a GTL light oil or a light oil composition containing a GTL light oil, wherein paraffin(s) are contained at a concentration of 97% by mass or greater, and isoparaffin(s) having 14 or fewer carbon atoms are contained in the above paraffin(s) at a concentration of 10% by mass or less. If the concentration of isoparaffin(s) is less than 97% by mass, then it ceases to be called a clean fuel due to the PM discharge properties and the like. If bio light oil were to be permitted, then deterioration of the fuel itself and deterioration of other materials becomes a problem. If the concentration of the isoparaffin(s) having 14 or fewer carbon atoms exceeds 10% by mass, then Nylon materials used in fuel systems oxidize, and become low molecular weight molecules by being depolymerized or reduce the extension thereof, leading to deterioration in Nylon-based materials.

[0013] By the way, the presence of isoparaffins lowers the cetane number of GTL light oils (lowers the too high cetane number of normal paraffins), and also exhibits the effect of lowering the viscosity. Consequently, a highly branched isoparaffin having 15 or more carbon atoms is preferably added if the viscosity of the light oil of the present invention becomes too high, thereby adjusting the viscosity. There is no particular limitation to the amount contained of the highly branched isoparaffin having 15 or more carbon atoms as long as the resultant has the desired viscosity and cetane number.

[0014] The light oil of the present invention can, for example, be manufactured as described below. Namely, manufacture is by hydrocracking paraffin obtained by FT reaction (Fischer-Tropsch reaction) over a solid acid catalyst to obtain an isoparaffin. This isoparaffin is next analyzed by gas chromatography and the concentration of isoparaffin(s) having 14 or fewer carbon atoms is investigated. An addition amount is decided in consideration of the gas chromatography-analyzed concentration of isoparaffin(s) having 14 or fewer carbon atoms (such that the isoparaffin concentration is 10% by mass or less after mixing) and the above isoparaffin is then mixed with the paraffin obtained by FT reaction.

[0015] The light oil of the present invention such as above has the following advantages.

(1) By composing a GTL light oil as in the present invention, there is no need to change the Nylon-based materials used in current fuel systems, and application can be made to diesel vehicles.

(2) When the GTL light oil of the present invention is mixed with a current light oil, there is also no need to change the Nylon-based materials used in current fuel systems, and application can be made to diesel vehicles.

(3) By switching light oil to GTL light oil or to a GTL light oil composition, the emissions of diesel vehicles can be made clean.

Examples

[0016] More specific explanation will be given of the present invention below by way of Test Samples and Examples, however the present invention is not limited thereto.

Test Example 1

[0017] Two types of GTL light oil having different concentrations of low molecular weight (low number of carbon atoms) paraffin were prepared (Simulation Test Sample - A and Simulation Test Sample - B). Test Sample - A was prepared to include a greater amount of low molecular weight paraffin in comparison to Test Sample - B, and this composition was confirmed using a GC method.

[0018] Next the dissolved oxygen amount in Test Sample - A and Test Sample - B was derived using a gas chromatography method (mounted to a thermal conductivity detector) with a 3 meter long column packed with molecular sieve 13X. The results are shown in FIG. 1. It is clear from FIG. 1 that the dissolved oxygen amount of Test Sample - A is greater in comparison to that of Test Sample - B. This is thought to be because the Test Sample - A contains a greater amount of low molecular weight isoparaffin than Test Sample - B.

[0019] The dissolved oxygen amount was measured for normal paraffins and isoparaffins (2-methyl paraffin) having 8 to 12 carbon atoms. The results are shown in FIG. 2. The dissolved oxygen concentration at room temperature is shown on the vertical axis of FIG. 2 (in a similar manner to FIG. 1). It can be seen from FIG. 2 that (1) isoparaffin contains a greater amount of oxygen than normal paraffin, and (2) for the same type of paraffin, the smaller the number of carbon atoms the more oxygen is contained.

[0020] Nylon-66 (referred to below simply as Nylon) was immersed in Test Sample - A and in Test Sample - B, respectively. After immersion for 500 hours the surface layer was removed from each Nylon and heated to 250 °C in Helium, and any gas generated was analyzed by gas chromatographic/ mass spectroscopic methods. The results thereof are shown in FIG. 3.

[0021] It can be seen from FIG. 3 that (1) Nylon that had been immersed in Test Sample - A contained more low molecular weight paraffin than Nylon that had been immersed in Test Sample - B. In addition, it can be seen that (2) the paraffin that had permeated into the Nylon had a higher proportion of isoparaffin than the original Test Sample.

[0022] Samples extracted from the surface of each of the Nylons above were analyzed using Matrix Assisted Laser Desorption Ionisation Mass Spectrometry (MALDI-MS). In addition similar analysis was carried out on untreated Nylon (FIG. 4A), Nylon subjected to oxidizing treatment (FIG. 4D), and Nylon subjected to hydrolysis treatment (FIG. 4E). The results are shown in FIG. 4. It can be seen from FIG. 4 that the mass spectrograph of Nylon that has been immersed in Test Sample - A substantially matches the mass spectrograph of Nylon that has been force-oxidized in the atmosphere. Namely it is seen that Nylon that has been immersed in Test Sample - A is oxidized. FIG. 4B is Nylon that has been immersed in Test Sample - B, and FIG. 4C is Nylon that has been immersed in Test Sample - A.

[0023] The surface of each of the above Nylons was machined at an angle, and infrared spectroscopy was carried out on this cut face at intervals of about 4 μm in the real depth of the Sample. These results are shown in FIG. 5. The relative intensity of the absorption for carbonyl groups generated by oxidation (1710 cm⁻¹) to the absorption for methylene of Nylon main chains (2930 cm⁻¹) is shown on the vertical axis.

[0024] It can be seen from FIG. 5 that the Nylon that has been immersed in Test Sample - A is oxidized to a depth of 400 μm from the surface, and that the Nylon that has been immersed in Test Sample - B is only oxidized to a depth of a few μm from the surface.

[0025] The cut faces of the Nylon that has been immersed in Test Sample - A and of the Nylon that has been immersed in Test Sample - B were analyzed with infrared spectroscopic imaging. The results are shown in FIG. 6. The relative intensity of the absorption for carbonyl groups generated by oxidation (1710 cm⁻¹) to the absorption for amide bonds of Nylon main chains (1650 cm⁻¹) is shown on the vertical axis. It can be seen from FIG. 6 that the Nylon that has been immersed in the GTL light oil Test Sample - A is oxidized to a depth of 400 μm from the surface. It can be seen from the results that there is a high probability that the Nylon that has been immersed in the GTL light oil of Test Sample - A has deteriorated as in the manner of the following (1) to (6).

[0026]

(1) the low molecular weight paraffins contained in Test Sample - A permeate into the Nylon.

(2) accompanying this action, water and oxygen dissolved in these paraffins also permeate into the Nylon.

(3) the permeated paraffins oxidize, generating radicals.

(4) radicals of the oxidized paraffins react to abstract hydrogen atoms from within molecules, generating a large number of radicals. For details of this matter reference can be made to "Sabrina Carroccio, Concetto Puglisi and Giorgio Montaudo, MALDI Investigation of the Photooxidation of Nylon-66, *Macromolecules* 2004, 37, (16), and 6037-6049".

(5) these radicals oxidize the carbon atoms adjacent to amide bonds of the Nylon molecules, breaking the molecular chains.

(6) hydrogen bonding is reduced between Nylon molecules of oxidized and reduced molecular weight, and extension is reduced.

[0027] It can be seen from the above that paraffins having 14 or fewer carbon atoms within the Samples swell the Nylon, the paraffins having 14 or fewer carbon atoms oxidize within the Nylon, and cause oxidation of the Nylon.

Test Example 2

[0028] A model fuel was prepared of the compositions shown in Table 1 below, from reagents of normal paraffin having 7 to 13 carbon atoms, isoparaffin having 7 to 12 carbon atoms (2-methyl paraffin) and an isomer of heptane (7 carbon atoms). Note that in Table 1: "n-C7 to n-C13" indicates normal heptane, normal octane, normal nonane, normal decane, normal undecane, normal dodecane, and normal tridecane; "2-Me-C6 to 2-Me-C11" indicates 2-methylhexane, 2-methylheptane, 2-methyloctane, 2-methylnonane, 2-methyldecane, and 2-methylundecane; and "n-C7 to n-C13 and 2-Me-C6 to 2-Me-C11" indicates a mixture of both of the above.

Moreover, "○" indicates that 2 ml of normal hexadecane has been added, as an internal standard.

[0029]

Table 1

Composition	Details	
	2ml of each	Normal Paraffin having 16 Carbon Atoms
Normal Paraffin	n-C7 to n-C13	○
Isoparaffin	2-Me-C6 to 2-Me-C11	○
Normal Paraffin and Isoparaffin	n-C7 to n-C13 and 2-Me-C6 to 2-Me-C11	○

[0030] Nylon was immersed in these model fuels, the model fuels were analyzed before and after immersion using gas chromatographic/ mass spectrographic methods, and the Nylons were analyzed before and after immersion using thermal desorption - gas chromatographic/mass spectrographic methods. The analysis results of using gas chromatographic/ mass spectrographic methods on the model fuels before and after immersion are shown in FIG 7.

[0031] The following can be seen from FIG. 7.

(1) isoparaffins are relatively easily oxidized in comparison to normal paraffin.

(2) the amount of alcohols generated from isoparaffins is about 100 times the amount of alcohol generated from normal paraffins.

(3) the amount of alcohol generated differs depending on the structure of the isomer. Namely, the ease of oxidation is different.

[0032] The analysis results of using thermal desorption - gas chromatographic/ mass spectrographic methods on the Nylons before and after immersion are shown in FIG. 8. It should be noted that there were no oxidation products detected in the Nylon that has been immersed in normal paraffin.

[0033] The following can be seen from FIG. 8.

(1) for isoparaffins and for normal paraffins, lower molecular weight paraffins permeate more readily into Nylon.

(2) in a comparison between a normal paraffin and isoparaffin of the same number of carbon atoms, it is the isoparaffin that permeates more readily.

(3) alcohols were detected from Nylon that has been immersed in isoparaffin, but alcohols were not detected from Nylon that has been immersed in normal paraffin.

[0034] It can be seen from the above that it is important to reduce paraffins having 14 or fewer carbon atoms in a GTL light oil in order to suppress swelling of the Nylon, and it is important to reduce isoparaffins having 14 or fewer carbon atoms in a GTL light oil in order to lower oxidation and suppress the reduction in extension of the Nylon.

5 Examples

[0035] Model fuels (light oils) A to E were produced of Component A and Component B as below, in blends as shown in Table 2. The paraffin concentration in the model fuels was 97% by mass or more in all cases.

[0036] Component A: mixture of equal amounts of normal paraffins having 8, 10, 12, and 14 carbon atoms.

Component B: mixture of isoparaffins having 8 and 9 carbon atoms (2-methylheptane: 3-methylheptane : 2-methyloctane, at 4:3:3 (volume ratio)).

[0037] Tensile tests were carried out after immersion treatment in each of the above model fuels of test pieces produced as described below.

15 (1) Test Piece Production:

[0038] Nylon-66 was formed according to JISK7162 (ISO3167) and manufactured into test pieces.

20 (2) Immersion Processing

[0039] 250ml of model fuel was placed in a 300ml volume pressure container (internal diameter: 45mm, internal height: 235mm), 3 test pieces were immersed therein and heated to 120 °C for 475 hours. Before immersion the test pieces were dried for 4 hours at 100 °C in a vacuum.

25 (3) Tensile Test:

[0040] Tensile testing was carried out on the test pieces after immersion using a tension testing device (made by Shimadzu: AG-10kNC) in accordance with ISO527 (the extension velocity was set at 50mm/min). The test pieces were stored in a desiccator from after immersion up until just before tensile testing.

30 (4) Results:

[0041] The results of tensile testing are shown in Table 2 below. Table 2 shows values of the average extensions measured at breakage of the three test pieces. A plot of the results of Table 2 is shown in FIG. 9. It can be seen from FIG 9 that the extension of the Nylon test pieces falls off rapidly when the concentration of isoparaffin exceeds 10% by mass.

[0042]

Table 2

Model Fuel	Isoparaffin Concentration in the Paraffin (%)	Extension on Breakage (mm)
A	0.0	38.4
B	3.0	35.3
C	7.0	34.2
D	10.0	35.4
E	13.0	22.1

[0043] The entire disclosure of Japanese Patent Application No. 2006-275311 is incorporated by reference herein. All publications, patent applications, and technical standards mentioned in this specification are herein incorporated by reference to the same extent as if each individual publication, patent application, or technical standard was specifically and individually indicated to be incorporated by reference.

Claims

1. A light oil composition comprising paraffin(s) at a concentration of 97% by mass or greater, wherein the paraffin(s) comprise isoparaffin(s) having 14 or fewer carbon atoms at a concentration of 10% by mass or less.
2. The light oil composition according to claim 1, further comprising highly branched isoparaffin(s) having 15 or more carbon atoms.

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FIG. 1

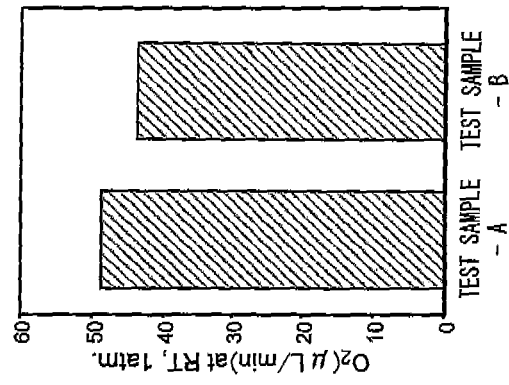


FIG. 2

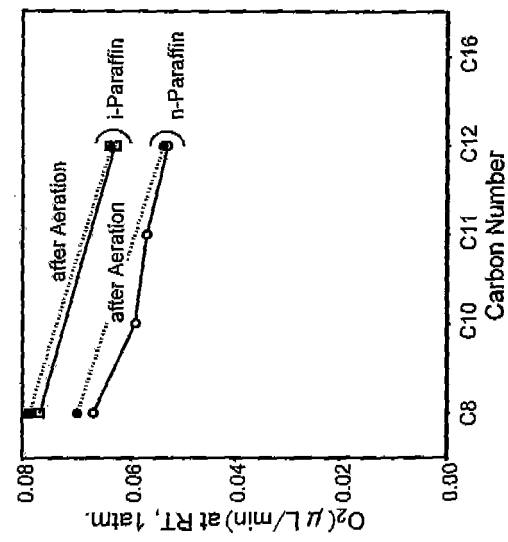


FIG. 3

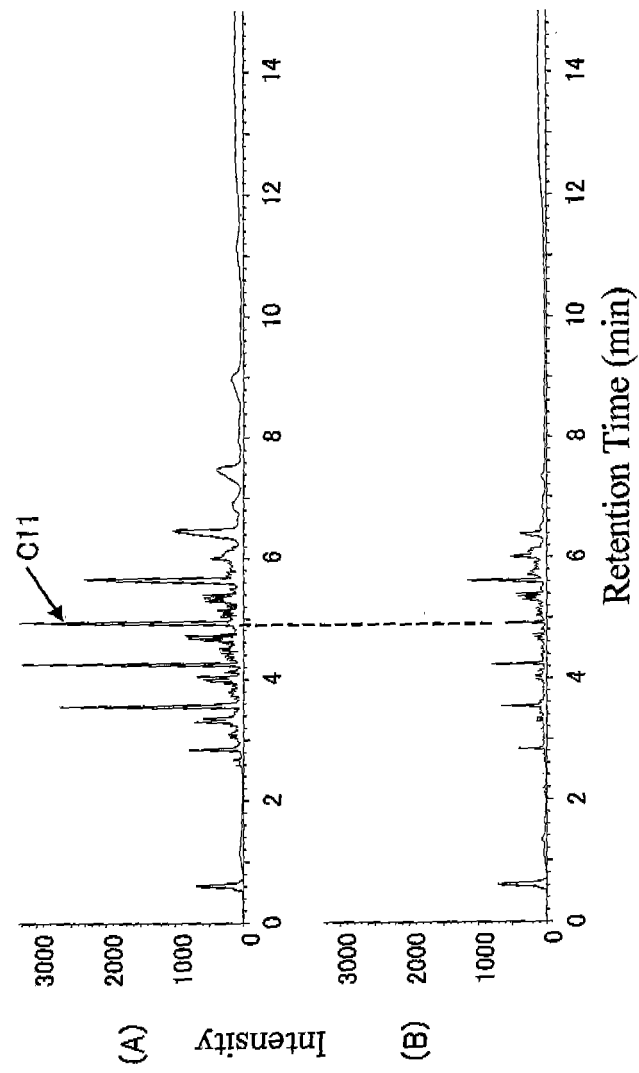


FIG. 4

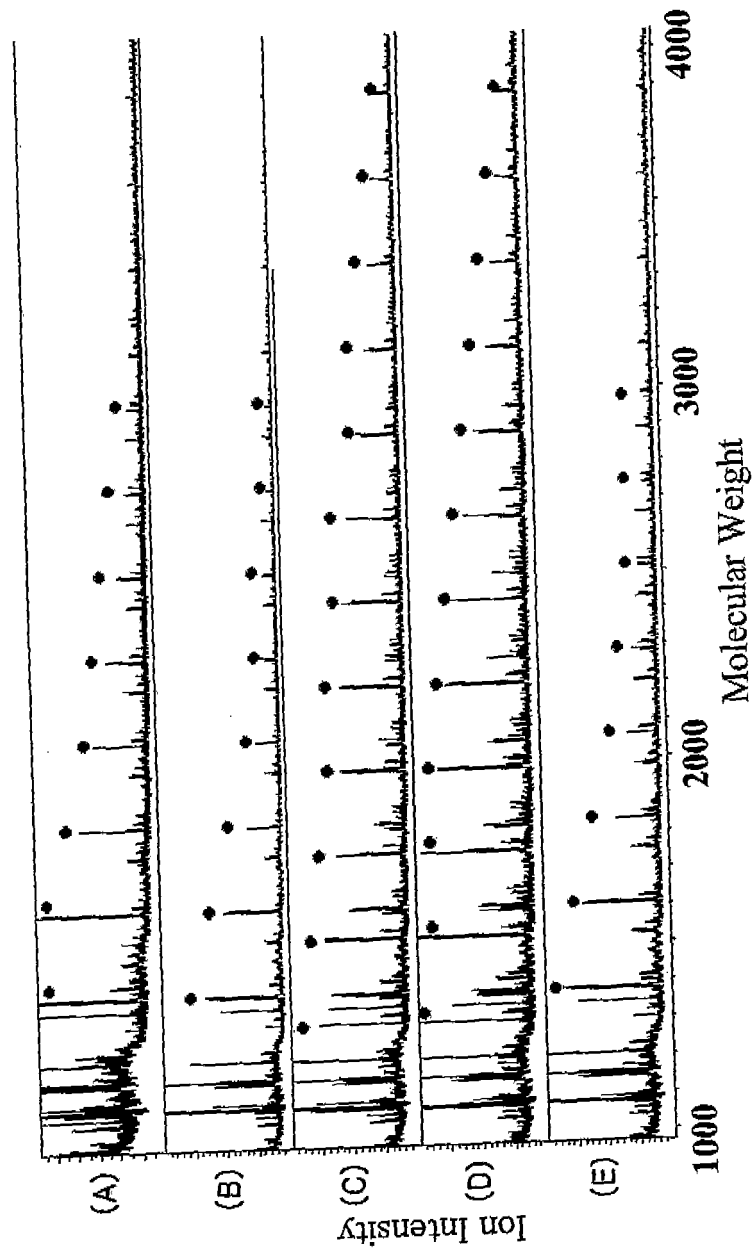


FIG. 5

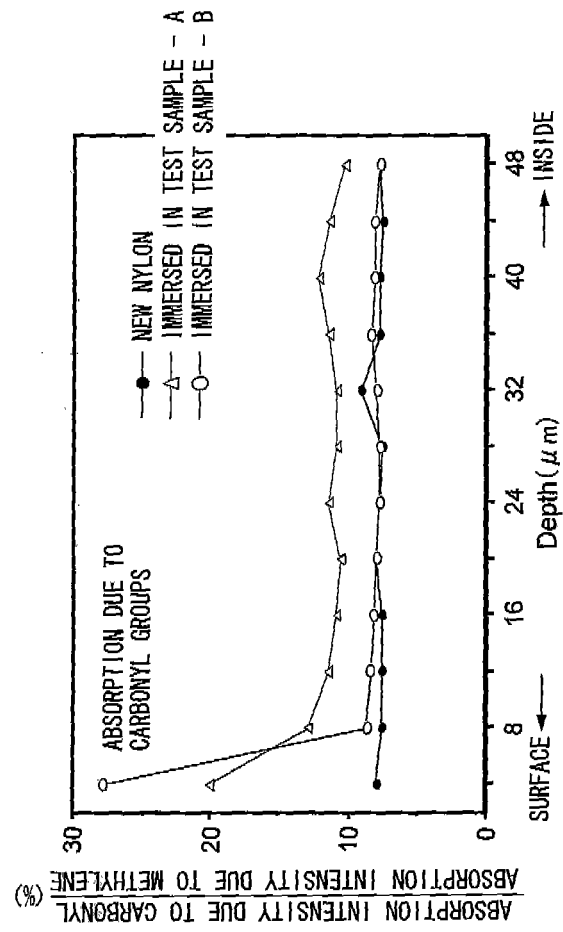


FIG. 6

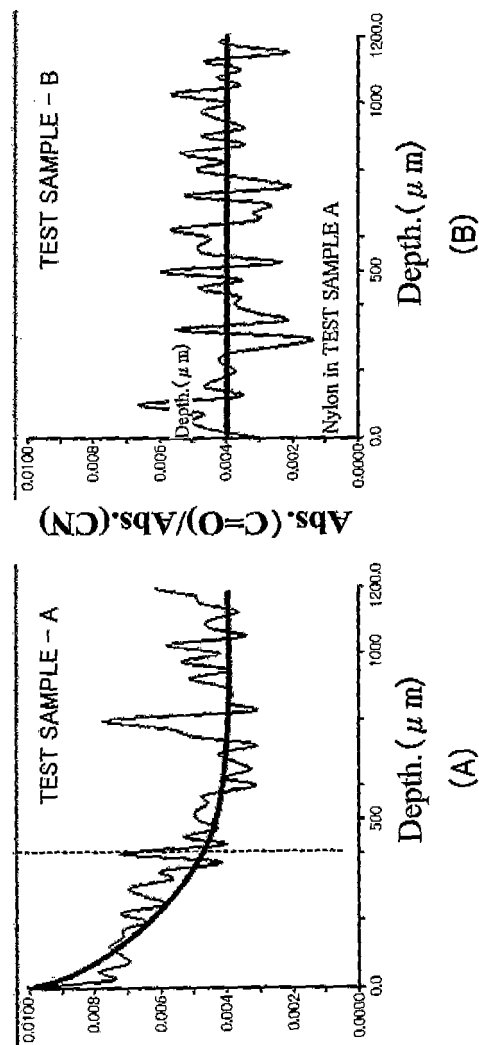


FIG. 7

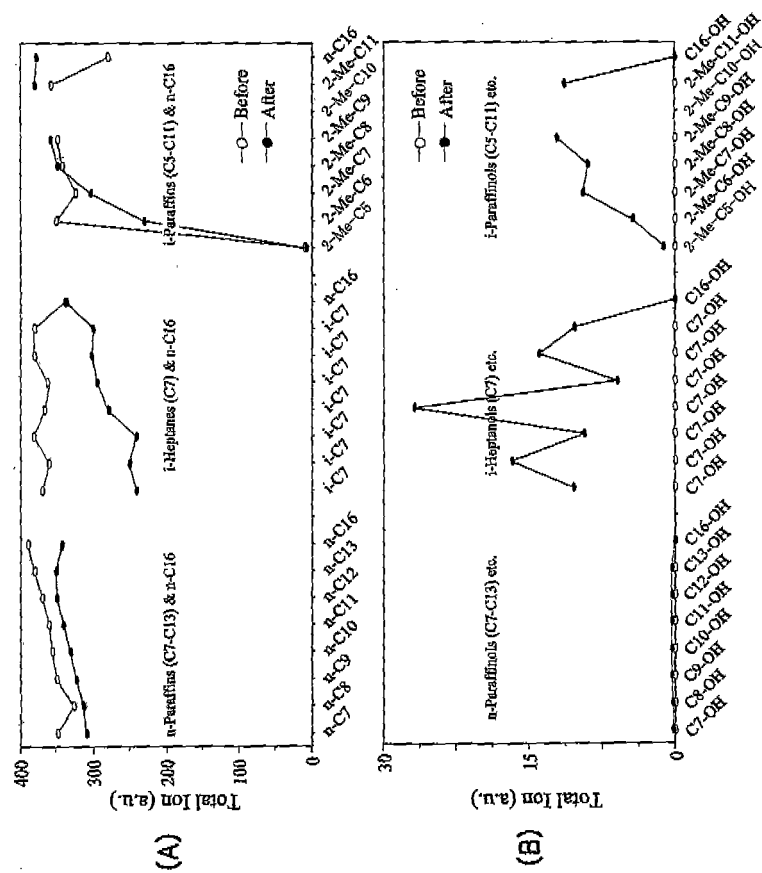


FIG. 8

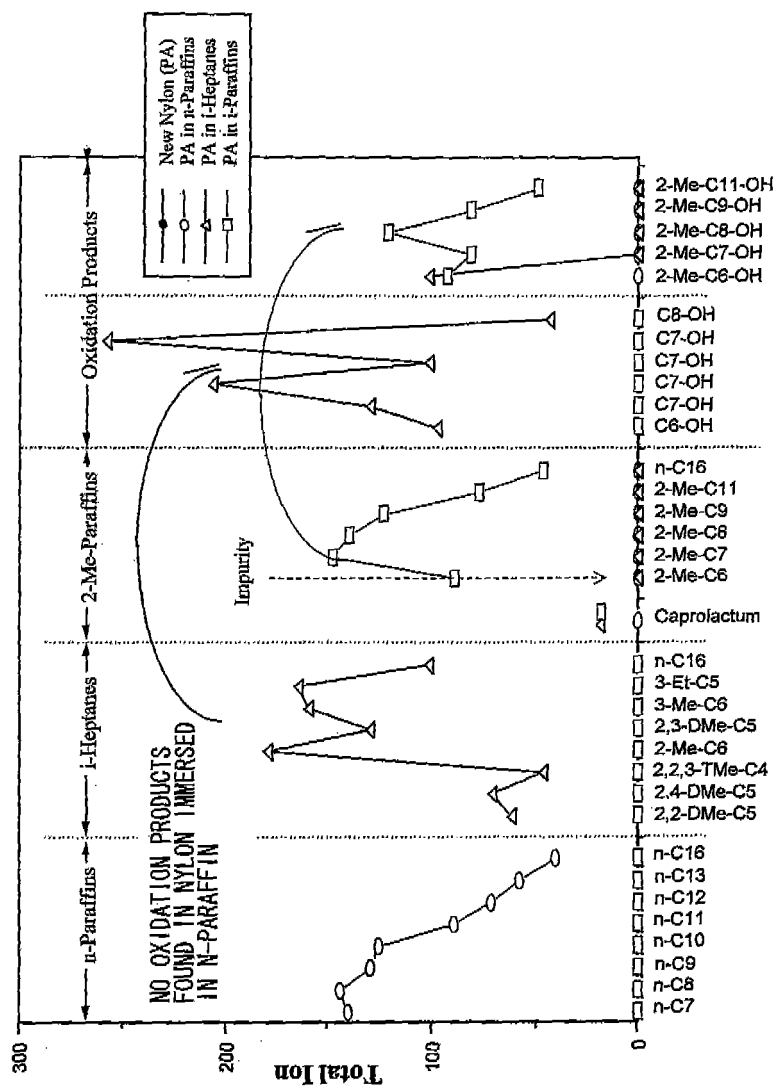
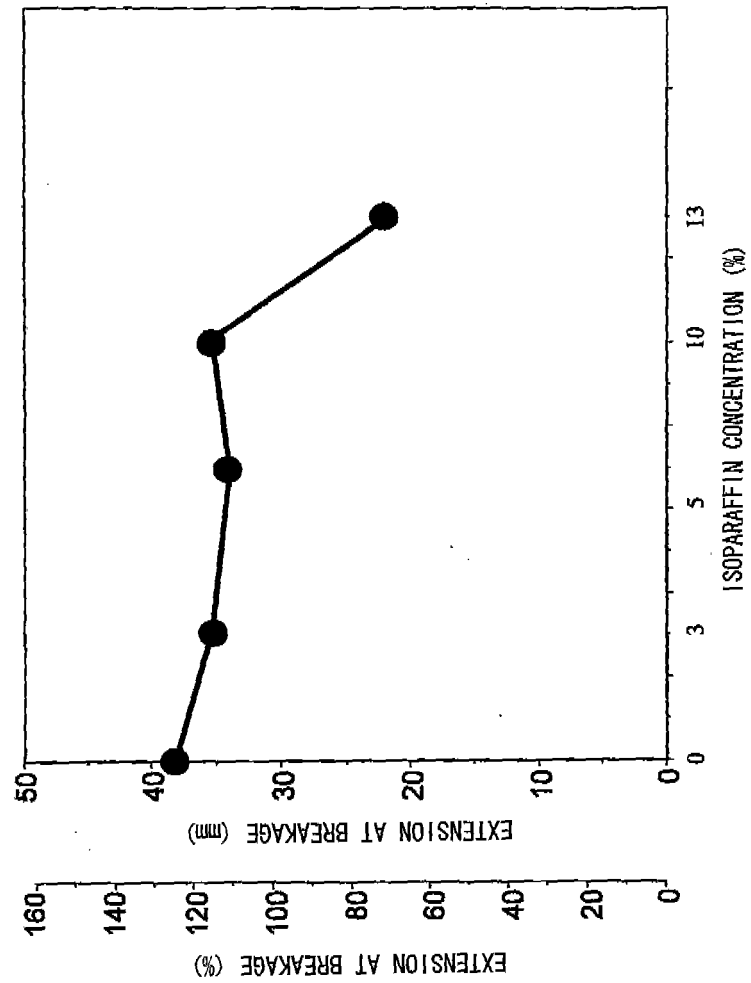


FIG. 9



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/069584

A. CLASSIFICATION OF SUBJECT MATTER

C10L1/08(2006.01) i

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)

C10L1/08

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

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2007
Kokai Jitsuyo Shinan Koho	1971-2007	Toroku Jitsuyo Shinan Koho	1994-2007

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

JST7580 (JDream2), JSTPlus (JDream2)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	Hideo OUCHI, "Diesel Jidoshayo Nenryo to Haishutsu Busshitsu", JARI Research Journal, 1985, 7(4), pages 127 to 131	1-2
X	JP 11-217575 A (Toyota Central Research and Development Laboratories, Inc.), 10 August, 1999 (10.08.99), Claim 2; Par. No. [0014] & EP 916716 A1	1-2

☒ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	"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
"A" document defining the general state of the art which is not considered to be of particular relevance	"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
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"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	

Date of the actual completion of the international search
18 October, 2007 (18.10.07)Date of mailing of the international search report
30 October, 2007 (30.10.07)Name and mailing address of the ISA/
Japanese Patent Office

Authorized officer

Facsimile No.

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2007/069584

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Form PCT/ISA/210 (continuation of second sheet) (April 2007)

REFERENCES CITED IN THE DESCRIPTION

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- **Noboru Kawada.** Outlook for the Next Generation of Synthetic Fuel Oils (First) "Quality Trends in Petroleum Products, and Issues with Existing Refining Techniques". *Petrotech*, vol. 23 (12), 1061-1066 [0005]
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