



(51) International Patent Classification:

C10M 169/04 (2006.01) C10N 40/08 (2006.01)
C10N 20/02 (2006.01)

(21) International Application Number:

PCT/EP2013/077539

(22) International Filing Date:

19 December 2013 (19.12.2013)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

2012-287655 28 December 2012 (28.12.2012) JP

(71) Applicant (for all designated States except US): **SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.** [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL).

(71) Applicant (for US only): **SHELL OIL COMPANY** [US/US]; PO Box 2463, One Shell Plaza, Houston, Texas 77252-2463 (US).

(72) Inventor: **NAGAKARI, Mitsuhiro**; 2-3-2 Daiba, Minato-ku, Tokyo, Tokyo 135-8074 (JP).

(74) Agent: **MATTHEZING, Robert Maarten**; PO Box 384, NL-2501 CJ The Hague (NL).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM,

AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

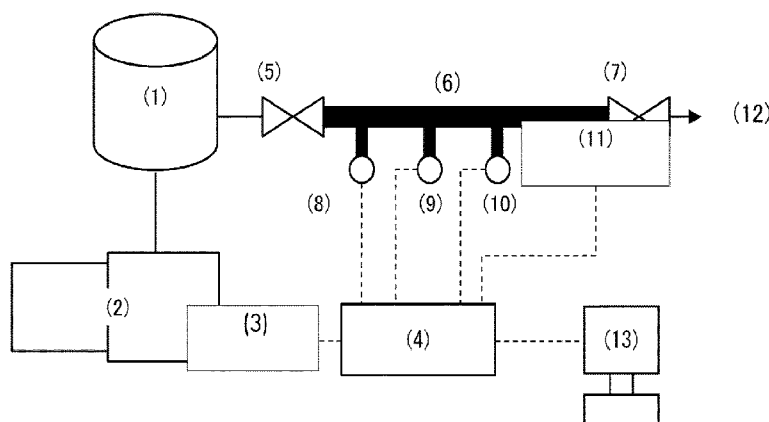
— as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))

Published:

— with international search report (Art. 21(3))

(54) Title: VACUUM PUMP OIL

Figure 1



(57) Abstract: A vacuum pump oil having excellent high temperature sealing properties, favourable low-temperature starting properties, a high flash point with both good thermal stability and degree of achieved vacuum is disclosed. The vacuum pump oil comprises a phenolic antioxidant, a thickening agent and a base oil manufactured using a gas- to-liquid process.

- 1 -

VACUUM PUMP OIL

Field of the Invention

This invention relates to vacuum pump oil and more specifically relates to vacuum pump oil which has excellent thermal oxidation stability and degree of achieved vacuum as well as low-temperature starting properties and sealing properties at high temperature.

Background of the Invention

Vacuum technology is widely employed in the fields of semiconductor fabrication, solar batteries, aircraft, automobiles, optoelectronics and the food industry.

In order to deploy this vacuum technology, a mechanical vacuum pump such as a reciprocating type vacuum pump and a rotary type vacuum pump, and a high vacuum pump such as an oil rotary vacuum pump and an oil diffusion vacuum pump have been conventionally used. Thus, for the purpose of lubricating such vacuum pump moving parts and for and a high vacuum and long life, synthetic oil-type and mineral oil-type vacuum pump oils have been used.

The widespread use of vacuum pumps has recently expanded and thermal stability and a high degree of vacuum have become requirements, and vacuum pump oils have been improved to deal with this. In particular, with high volatility of vacuum pump oil itself, the oil evaporates with the exhaust gas and the pressure rises, and it is known that the degree of achieved vacuum worsens (see Lubrication, Vol 33, No. 6 (1988) p454-457).

Moreover, sliding parts in the vacuum pump become hot due to friction and, if vacuum pump oil having inferior thermal oxidation stability is used, sludge

- 2 -

generated during operation becomes caught in sliding parts and this is a factor leading to worsening of the degree of achieved vacuum. Furthermore, there is the risk of equipment problems occurring when valves become stuck. Therefore, the productivity of the products of interest decreases and there is the problem that stable product quality is not achieved. Thus, it is desirable to improve the thermal oxidation stability of the vacuum pump oil and to decrease the amount of sludge formed.

In order to suppress the formation of sludge, the use of antioxidants like p-branched alkylphenyl- α -naphthylamine has been proposed but, as they are amine-type antioxidants, the decomposed components become basic and there is concern about their impact when mixed in a vacuum system (see JP 07-252489).

Vacuum pumps are also thought to aspirate acidic gases such as carbon monoxide and there is the concern that sludge will form due to a reaction between basic components and acidic gases so it is preferable not to use amine-type antioxidants.

Furthermore, in the area of application of vacuum technology, a short time is required to reach steady operation after starting the vacuum pump in order to increase productivity. However, conventionally known vacuum pump oils take a long time from start to reach a steady state, in particular, if used in winter and cold climates and improvement in low-temperature starting properties is required.

Many oils like petroleum and animal and plant oils are included in flammable fluids specified in Type 4 Hazardous Materials and the combustible liquid category specified in Designated Combustibles according to the Fire Services Act. These are stipulated in Hazardous

- 3 -

Materials according to the Fire Services Act because they are flammable, and, as the risk of fire is high because the flash point is low, legal regulations have been drawn up regarding the amount handled and stored depending on the flash point. An increase in flash point increases the safety and fire handling and the equipment required for storage and management costs can be reduced as handling such as storage management becomes easier.

As storage and management become easier and handling is economical due to this increase in safety and an increase in flash point, a high flash point for vacuum pump oils is preferable. The flash point measured with the Cleveland open flash point tester according to JIS K2265-4 may be 200°C or more, preferably 250°C or more and further more preferably 260°C.

The vacuum pump oil of the present invention, which was made in view of the above points, has both good thermal stability and an excellent degree of achieved vacuum, the flash point is high, the low-temperature starting properties are favourable and it has improved high-temperature sealing properties.

Summary of the Invention

Accordingly, the present invention provides a VG68 standard vacuum pump oil having a 100°C kinematic viscosity of 10.5mm²/s or more and a viscosity index of 150 or more, comprising from 0.01 to 5wt% phenolic antioxidants, from 3 to 15wt% of a poly- α -olefin thickening agent or an olefin copolymer having a number average molecular weight of 2,000 to 30,000, and a base oil manufactured using the gas-to-liquid process having a 100°C kinematic viscosity of from 5 to 20mm²/s and containing 3.0% or less hydrocarbons with a carbon number of 30 or less.

- 4 -

The vacuum pump oil in the invention shows high stability against heat and excellent results can be achieved in the degree of vacuum which is obtained. In addition, the vacuum pump oil in the invention has a high flash point so safety and fire handling are increased, handling such as storage and management also become easier and it is possible to reduce management cost and the equipment required for storage. Furthermore, low-temperature starting properties are good and allow early migration to a steady operation from starting, moreover, as it has excellent high temperature sealing properties it becomes possible to efficiently and economically operate a vacuum pump.

Detailed Description of the Invention

A base oil manufactured using the gas-to-liquid process (GTL) is used as a base oil for this vacuum pump oil. In this invention, base oil manufactured using the gas-to-liquid process (GTL) is manufactured using a condensation method according to the Fischer-Tropsch process, so carbon monoxide and hydrogen in the presence of an appropriate catalyst, high temperature (for example, 125-300°C preferably 175-250°C) and/or high pressure (for example, 5×10^5 to 10^7 N/m^2 preferably 1.2×10^5 to $5 \times 10^6 \text{ N/m}^2$) are converted into long-chain paraffinic hydrocarbons.

In addition, isoparaffinic base oil is also manufactured from wax according to the Fischer-Tropsch process.

In this way, Fischer-Tropsch-derived base oil has a tendency to have excellent low-temperature characteristics, for example, a low pour point, evaporation loss too is extremely low, and methods used in the manufacture of these base oils are also

- 5 -

advantageous because of the relatively simple process used to make them compared to similar oils prepared from mineral crude sources.

5 The American Petroleum Institute classifies this Fischer-Tropsch-derived base oil as a base oil belonging to the Group III category and does not contain any sulphur or nitrogen or they are at the undetectable level, if present. In addition, it contains no or virtually no aromatic components and, generally, this is
10 below 1wt%, preferably below 0.5wt% and further preferably below 0.1wt% (ASTM D-4629). Thus, the oxidative stability of the oil is improved.

Base oil manufactured using this gas-to-liquid process having a kinematic viscosity of from 5 to
15 20mm²/s, preferably from 6 to 15mm²/s and more preferably from 7 to 10mm²/s at 100°C and an amount of hydrocarbon with a carbon atom number of 30 or less of 3.0% or less, preferably 2.9% or less and more preferably 2.7% or less can be used.

20 The amount of hydrocarbon with a carbon number 30 or less in this invention is measured using a method for measuring the content by percentage (%) of the overall peak area value of a fraction having a chromatogram distillation point of 449°C or lower using the gas
25 chromatography distillation method stipulated in ASTM D2887-08.

In order to achieve a high degree of achieved vacuum in the vacuum pump, it is preferable to use a base oil having a low evaporation loss amount and, if it is a base
30 oil with the same kinematic viscosity, the molecular weight distribution of base oil is preferably narrow as described in Lubrication, Vol 33, No. 6 (1988) p454-457. As an evaluation method for the amount of base oil

- 6 -

evaporation loss, there is the NOACK volatility procedure stipulated in ASTM D5800.

Shell XHVI-8 (Royal Dutch Shell plc), a base oil manufactured using the gas-to-liquid process, only shows an evaporation loss in the NOACK volatility procedure of 2.0 mass% and can be suitably used in vacuum pump oil. The value for base oil evaporation loss for the vacuum pump oil is not particularly limited and is 2.8 mass% or lower, preferably 2.4 mass% or lower and more preferably 2.0 mass% or lower.

The API Group 3 base oil Yu-base 8 (SK Innovation Co. Ltd.) is known as a highly refined base oil. Yu-base 8 properties are a kinematic viscosity of 48.8mm²/s at 40°C, a kinematic viscosity of 7.8mm²/s at 100°C, a viscosity index of 129 and a density of 0.845g/cm³ at 15°C. It also has a high evaporation loss of 3.1 mass% according to the NOACK volatility procedure and is not considered suitable as a vacuum pump oil.

In addition, PAO8 is marketed as a polyalphaolefin synthetic base oil. The properties of PAO8 are a kinematic viscosity of 46.6mm²/s at 40°C, a kinematic viscosity of 7.8mm²/s at 100°C, a viscosity index of 136 and a density of 0.831g/cm³ at 15°C. It also has a high evaporation loss of 2.9 mass% according to the NOACK volatility procedure and, likewise, is not considered suitable as a vacuum pump oil.

The above-mentioned marketed PAO8 is produced by polymerisation of 1-decene and is composed of 5% of a trimer component having a carbon atom number of 30 (molecular weight 422), 65% of a tetramer component having a carbon atom number of 40 (molecular weight 562), 2% of a pentamer component having a carbon atom number of 50 (molecular weight 702) and 10% of a hexamer component

- 7 -

having a carbon atom number of 60 (molecular weight 842) as the main components and is substantially a low-molecular weight component mixture. These components exist in the low-molecular weight region because they have a non-continuous carbon atom number distribution being a mixture and, as evaporation loss becomes significant, they are thus not considered suitable as a vacuum pump oil.

Phenolic antioxidants are added to the above base oil.

Phenolic antioxidants include, for example, 2-t-butylphenol, 2-t-butyl-4-methylphenol, 2-t-butyl-5-methylphenol, 2,4-di-t-butylphenol, 2,4-dimethyl-6-t-butylphenol, 2-t-butyl-4-methoxyphenol, 3-t-butyl-4-methoxyphenol and 2,5-di-t-butylhydroquinone (Kawaguchi Chemical Industry: Antage DBH), 2-6-di-t-butylphenol, 2,6-di-t-butyl-4-alkylphenols such as 2,6-di-t-butyl-4-methylphenol and 2,6-di-t-butyl-4-ethylphenol, and 2,6-di-t-butyl-4-alkoxyphenols such as 2,6-t-butyl-4-methoxyphenol and 2,6-di-t-butyl-4-ethoxyphenol.

In addition, there are propionates such as 3,5-di-t-butyl-4-hydroxybenzylmercaptooctyl acetate, alkyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionates such as n-octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl) propionate (Yoshitomi Fine Chemicals: Yoshinox SS), n-dodecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl) propionate and 2'-ethylhexyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate, benzenepropanoic acid 3,5-bis(1,1-dimethyl-ethyl)-4-hydroxy-C7-C9 side-chain alkyl esters (BASF: Irganox L135), 2,6-di-t-butyl- α -dimethylamino-p-cresol and 2,2'-methylenebis(4-alkyl-6-t-butylphenol)s such as 2,2'-methylenebis(4-methyl-6-t-butylphenol) (Kawaguchi Chemical Industry: Antage W-400) and 2,2'-methylenebis(4-

- 8 -

ethyl-6-t-butylphenol) (Kawaguchi Chemical Industry: Antage W-500).

Furthermore, there are bisphenols such as 4,4'-butylidenebis (3-methyl-6-t-butylphenol) (Kawaguchi
5 Chemical Industry: Antage W-300), 4,4'-methylenebis(2,6-di-t-butylphenol) (Shell Japan: Ionox 220AH), 4,4'-bis(2,6-di-t-butylphenol), 2,2-(di-p-hydroxyphenyl)propane (Shell Japan: Bisphenol A), 2,2-bis(3,5-di-t-butyl-4-hydroxyphenyl)propane, 4,4'-cyclohexylidenebis(2,6-t-
10 butylphenol), hexamethylene glycol bis[3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate] (BASF: Irganox L109), triethylene glycol bis[3-(3-t-butyl-4-hydroxy-5-methylphenyl) propionate] (Yoshitomi Fine Chemicals: Tominox 917), 2,2'-thio-[diethyl-3-(3,5-di-t-butyl-4-
15 hydroxyphenyl) propionate (BASF: Irganox L115), 3,9-bis{1,1-dimethyl-2-[3-(3-t-butyl-4-hydroxy-5-methylphenyl) propionyloxy]ethyl}2,4,8,10-tetraoxaspiro[5,5] undecane (Sumitomo Chemicals: Sumilizer GA80), 4,4'-thiobis(3-methyl-6-t-butylphenol)
20 (Kawaguchi Chemical Industry: Antage RC) and 2,2'-thiobis(4,6-di-t-butyl-resorcinol).

Mention may also be made of polyphenols such as tetrakis[methylene-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate]methane (BASF: Irganox L101),
25 1,1,3-tris(2-methyl-4-hydroxy-5-t-butylphenyl)butane (Yoshitomi Fine Chemicals: Yoshinox 930), 1,3,5-trimethyl-2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)benzene (Shell Japan : Ionox 330) bis-[3,3'-bis-(4'-hydroxy-3'-t-butylphenyl)butyric
30 acid]glycol ester, 2-(3',5'-di-t-butyl-4-hydroxyphenyl)methyl-4-(2'',4''-di-t-butyl-3''-hydroxyphenyl)methyl-6-t-butylphenol and 2,6-bis(2'-hydroxy-3'-t-butyl-5'-methyl-benzyl)-4-methylphenol, and

- 9 -

phenol-aldehyde condensates such as condensates of p-t-butylphenol and formaldehyde and condensates of p-t-butylphenol and acetaldehyde.

5 The above-mentioned phenolic antioxidants may be used alone or more than one may be used in an appropriate combination.

10 These phenolic antioxidants are added in the range of from 0.01 to 5wt%, preferably from 0.05 to 3wt% and more preferably from 0.1 to 1wt% to the total amount of vacuum pump oil.

Furthermore, a thickening agent is added to the above base oil. As a thickening agent, mention may be made of an olefin copolymer, an ethylene/propylene copolymer and an ethylene/isobutylene copolymer and, 15 generally, an ethylene propylene copolymer is preferably used.

20 In addition, poly- α -olefin can be used as a thickening agent. This poly- α -olefin comprises a single type of α -olefin having a carbon atom number of 5 or more or 2 or more are combined and polymerised.

25 The ethylene/propylene copolymer and poly- α -olefin preferably have a number average molecular weight of from 2,000 to 30,000, more preferably from 3,000 to 20,000 and further more preferably from 5,000 to 8,000. When the above molecular weight is exceeded, shearing occurs in the sliding parts of the pump, the viscosity of the vacuum pump oil decreases and there is the possibility of sludge formation under high temperature conditions. However, if the molecular weight is too low, the 30 thickening effect weakens so it is necessary to add a large amount of thickening agent and this becomes a factor in the increased cost of lubricating oil products.

- 10 -

Such thickening agents are used in a range of from 3 to 15% and preferably from 4 to 14% in the total amount of vacuum pump oil.

5 In order to obtain a high degree of achieved vacuum using a vacuum pump, it is desirable to seal movable parts with an oil membrane formed from the vacuum pump oil. If the kinematic viscosity of vacuum pump oil is high and a sufficient oil membrane is maintained, the sealing effect of lubricating oil is thought to be high.
10 However, if the kinematic viscosity is excessively low and a sufficient oil membrane is not maintained, gaps arise in sliding parts and the degree of achieved vacuum worsens.

The moving parts of the vacuum pump are thought to
15 become hot exceeding 100°C due to friction and it becomes necessary to maintain a high kinematic viscosity for the vacuum pump oil under such high temperature conditions.

In VG68 standard vacuum pump oil, a 100°C kinematic viscosity is preferably 10.5mm²/s or more, is more
20 preferably 10.7mm²/s or more and is further more preferably 10.9mm²/s or more. Furthermore, the 120°C kinematic viscosity is preferably 7.0mm²/s or more, is more preferably 7.2mm²/s or more and is further more preferably 7.4mm²/s or more. Moreover, the flash point of
25 the vacuum pump oil of the invention is preferably 260°C or more, is more preferably 265°C and is further more preferably 268°C.

Each additive can be suitably added to this vacuum pump oil as necessary.

30 Next, the present invention will be described in more detail by way of Examples and Comparative Examples, although the present invention is not limited by these in any way.

- 11 -

The following preparations were made in order to produce the examples and comparative example.

Base Oil

Base Oil 1: Fischer-Tropsch base oil using the gas-to-liquid process - Shell XHVI-8 (Royal Dutch Shell)
[properties: 40°C kinematic viscosity 44.4mm²/s, 100°C kinematic viscosity 7.7mm²/s; 2.6% hydrocarbons with a carbon number of 30 or less obtained using the chromatogram distillation method specified in ASTM D2887-08; viscosity index 142; 15°C density 0.828g/cm³; evaporation loss in engine oil evaporation (NOACK method) 2.0 mass%]

Additives

Additive A1: ethylene-propylene copolymer (Mitsui Chemicals: Lucant HC-1100)

- (a) number average molecular weight: 6,000
- (b) 40°C kinematic viscosity: 18,900mm²/s
- (c) 100°C kinematic viscosity: 1,100mm²/s
- (d) viscosity index: 270
- (e) 15°C density: 0.850g/cm³

Additive A2: poly- α -olefin (Exxon Mobil Chemical: SpectraSyn Elite 150)

- (a) number average molecular weight: 7,000
- (b) 40°C kinematic viscosity: 1,731mm²/s
- (c) 100°C kinematic viscosity: 157mm²/s
- (d) viscosity index: 205
- (e) 15°C density: 0.850g/cm³

Additive A3: polyisobutylene (JX Nippon Oil & Energy: Nisseki Polybutene HV300)

- (a) number average molecular weight: 1,400
- (b) 40°C kinematic viscosity: 26,000mm²/s
- (c) 100°C kinematic viscosity: 590mm²/s
- (d) viscosity index: 155

- 12 -

(e) 15°C density: 0.898g/cm³

Additive A4: High viscosity mineral oil

(a) number average molecular weight: 740 (calculated using the formula of ASTM D3238 ring analysis)

5 (b) 40°C kinematic viscosity: 430mm²/s

(c) 100°C kinematic viscosity: 31.4mm²/s

(d) viscosity index: 105

(e) 15°C density: 0.885g/cm³

10 Additive B: phenolic antioxidant (BASF: Irganox L135)

Examples 1-2, Comparative Examples 1-2

Using the above materials, vacuum pump oils of examples 1-2 and comparative examples 1-2 were prepared according to the composition shown in Table 1. The amount of combined component is given in wt%.

Tests

The following tests were carried out to investigate the properties and performance of the vacuum pump oil in examples 1-2 and comparative examples 1-2.

20 40°C Kinematic Viscosity

According to JIS K2283.

100°C Kinematic Viscosity

According to JIS K2282.

25 Evaluation criteria: 10.5mm²/s or more..... ○
less than 10.5mm²/s... ×

120°C Kinematic Viscosity

According to JIS K2282.

Evaluation criteria: 7.0mm²/s or more..... ○
less than 7.0mm²/s... ×

30 Viscosity Index

According to JIS K2282.

Evaluation criteria: 150 or more..... ○
less than 150... ×

- 13 -

Pour Point

According to JIS K2269.

Evaluation criteria: less than -25°C ... ○
 -25°C or more..... ×

5 Thermal Stability Test

It is preferable for vacuum pump oil to have low sludge in order to prevent equipment problems due to vacuum pump oil sludge. The Cincinnati Milacron stability test is a known evaluation method for sludge formation
10 behaviour of lubricating oil under thermal oxidation conditions.

Therefore, a thermal stability test was conducted according to ASTM D2070.

In the test, 200ml test oil was initially put in a
15 container then it was left to stand in a thermostat bath at 120°C for 45 days in the presence of both copper catalyst and iron catalyst. Following completion of the test, a sample of oil was filtered through a membrane filter with $0.8\mu\text{m}$ pores and the amount of sludge formed
20 was measured.

Evaluation criteria: less than $3.0\text{mg}/200\text{mL}$... ○
 $3.0\text{mg}/200\text{mL}$ or more..... ×

Achieved Vacuum Pressure

Achieved vacuum pressure in an oil rotary vacuum
25 pump was measured in accordance with JIS B8316 using the system shown in Figure 1. The system consists of a test chamber (1), a rotary vacuum pump (2), a thermoelectric couple (3), a data logger (4), a first valve (5) a vacuum chamber (6), a second valve (7), a diaphragm vacuum gauge
30 (8), a CC gauge (9), a B-A gauge (10), a thermoelectric couple (11), an outlet to high vacuum exhaust (12) and a PC (13). The oil rotary vacuum pump used, the test conditions and test procedure are shown below.

- 14 -

(a) oil rotary vacuum pump: Alcatel M2010SD (motor rated output 450W)

(b) pumping speed (50Hz): 162L/min (design pumping speed), 142L/m (actual pumping speed)

5 (c) volume removed in 1 compression (total value): 0.054L

(d) test procedure: after venting the vacuum pump to the open air, the pump is started at an oil temperature from 35°C without gas ballast and the oil temperature is
10 adjusted to 50°C while cooling the pump using cooling fins. The oil temperature becomes 50°C about 50 minutes after starting the pump and is stable. The average inlet pressure value in the 5 minutes from 55-60 minutes after starting the pump is taken as the achieved vacuum
15 pressure. The test dome temperature was maintained at 25°C during the test.

Evaluation criteria: less than 0.1Pa... o
0.1Pa or more..... x

Vapour Pressure

20 Measured using the direct method (MST 0402-1).

Evaluation criteria: less than 0.1×10^{-3} Pa... o
 0.1×10^{-3} Pa or more..... x

Test Results

Test results are shown in Table 1. As shown in Table
25 1, the olefin copolymer (additive A1) used in example 1 and the poly- α -olefin (additive A2) used in example 2 showed a viscosity index of 152 and 154 and showed high values of 150 or more, the kinematic viscosity was
11.0mm²/s for both at 100°C and 7.4mm²/s at 120°C and
30 were high values and favourable. The pour points were -32.5°C and -27.5°C and both satisfied less than -25°C. In addition, in the Cincinnati Milacron thermal stability test, the amount of sludge was 0.8mg/200mL and

- 15 -

1.3mg/200mL and both were low values of less than 3.0mg/200mL. The achieved vacuum pressure was 0.091Pa and 0.087Pa, both were less than 0.1Pa and a high vacuum was achieved. Vapour pressure was 0.96×10^{-3} Pa and 0.50×10^{-3} Pa and both showed low values of less than 1.0×10^{-3} Pa. In addition, the flash point was high at 268°C and 276°C and shown high safety.

Comparative example 1 used polyisobutylene (Additive A3), had a low kinematic viscosity at 100°C, kinematic viscosity at 120°C and viscosity index compared to example 1 and 2 and did not satisfy the evaluation criteria values. Although the amount of sludge satisfied the evaluation criteria values in the pour point and the Cincinnati Milacron thermal stability tests, the achieved vacuum pressure showed a large value of 0.17Pa and the degree of achieved vacuum was not sufficient.

Comparative example 2 used high viscosity base oil (Additive A4) and did not satisfy the evaluation criteria values for 100°C kinematic viscosity and 120°C kinematic viscosity and viscosity index which were low compared to example 1 and example 2. The achieved vacuum pressure and vapour pressure evaluation criteria values were satisfied but the pour point was high, it did not satisfy the evaluation criteria value for sludge amount in the Cincinnati Milacron thermal stability test and was unsuitable.

In the same way as above, in order to achieve a high degree of achieved vacuum using a vacuum pump, it is desirable to seal movable parts with an oil membrane formed from the vacuum pump oil. For example, a 100°C kinematic viscosity of $10.5 \text{ mm}^2/\text{s}$ or more is preferred and example 1 and example 2 showed high values for 100°C kinematic viscosity of $11.0 \text{ mm}^2/\text{s}$ or more, an oil membrane

- 16 -

is sufficiently maintained and it is thought that the sealing effect with lubricating oil is high. However, the 100°C kinematic viscosity in comparative example 1 was 10.1mm²/s and in comparative example 2 was 10.2mm²/s and were low.

Furthermore, a 120°C kinematic viscosity of 7.0mm²/s or more is preferred, the 120°C kinematic viscosity in example 1 and example 2 are 7.4mm²/s and, as comparative example 1 showed a low value of 6.7mm²/s and comparative example 2 showed a low value of 6.8mm²/s, it is feared that a sufficient oil membrane cannot be maintained at high temperature.

Table 1

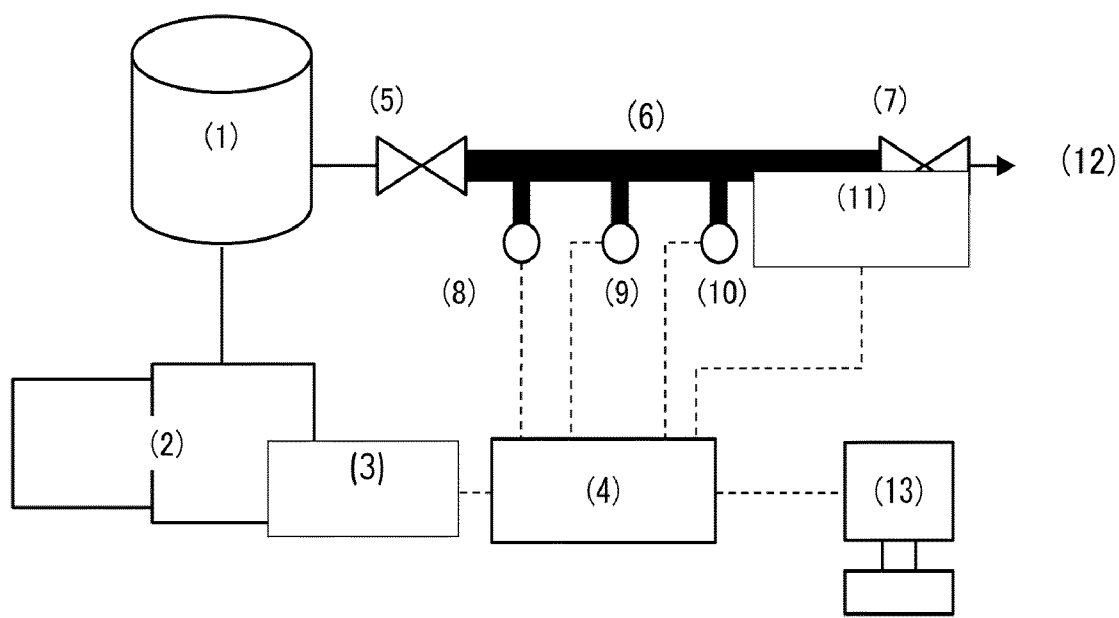
	Example 1	Example 2	Comparative Example 1	Comparative Example 2
Composition	Base oil 1: Shell XHVI-8	85.9	89.7	75.0
	Additive A1: Lucant HC-1100	4.8		
	Additive A2: SpectraSyn Elite 150	13.8		
	Additive A3: Nisseki Polybutene HV300		10.0	
	Additive A4: High viscosity mineral oil			24.7
	Additive B: Irganox L135	0.3	0.3	0.3
Test Results	40°C kinematic viscosity, mm ² /s	68.2	66.5	69.3
	100°C kinematic viscosity, mm ² /s	11.0 o	10.1 x	10.2 x
	120°C kinematic viscosity, mm ² /s	7.4 o	6.7 x	6.8 x
	viscosity index	152 o	138 x	133 x
	pour point, °C	-32.5 o	-27.5 o	-22.5 x
	flash point (COC), °C	268	—	—
	Cincinnati Milacron thermal stability test sludge amount, mg/200mL	0.8 o	0.6 o	7.1 x
	achieved vacuum pressure, Pa	0.091 o	0.17 x	0.090 o
	vapour pressure, 10 ⁻³ Pa	0.96 o	—	0.13 o

- 18 -

C L A I M S

1. A VG68 standard vacuum pump oil having a 100°C kinematic viscosity of 10.5mm²/s or more and a viscosity index or 150 or more, comprising from 0.01 to 5wt% phenolic antioxidant, from 3 to 15wt% of a thickening agent which is a poly- α -olefin or an olefin copolymer and has a number average molecular weight of from 2,000 to 30,000, and a base oil manufactured using a gas-to-liquid process having a 100°C kinematic viscosity of from 5 to 20mm²/s and containing 3.0% or less hydrocarbons with a carbon number of 30 or less.
2. A vacuum pump oil according to claim 1 which is a VG68 standard vacuum pump oil having a 120°C kinematic viscosity of 7.0mm²/s or more.
3. A vacuum pump oil according to claims 1 or 2 which is a VG68 standard vacuum pump oil having a flash point or 260°C or more.
4. A vacuum pump oil according to any preceding claim, wherein the thickening agent is an ethylene propylene copolymer.

Figure 1



INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/077539

A. CLASSIFICATION OF SUBJECT MATTER

INV. C10M169/04

ADD. C10N20/02 C10N40/08

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)

C10M C10N

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 practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2009/181871 A1 (SHAH RAVINDRA [US] ET AL) 16 July 2009 (2009-07-16) paragraphs [0069], [0088], [0089]; examples 12-14; tables 4,5 -----	1-4
X	WO 2006/116775 A1 (RENEWABLE LUBRICANTS INC [US]; GARMIER WILLIAM W [US]) 2 November 2006 (2006-11-02) paragraphs [0089] - [0091], [0108], [0109] -----	1-3
X	US 2007/142250 A1 (LOH WILLIAM [US] ET AL) 21 June 2007 (2007-06-21) paragraphs [0056] - [0065], [0118]; claims; examples 5,6 ----- -/-	1-4



Further documents are listed in the continuation of Box C.



See patent family 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 application or patent 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

4 April 2014

Date of mailing of the international search report

11/04/2014

Name and mailing address of the ISA/

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

Authorized officer

Kazemi, Pirjo

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2013/077539

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2006/199743 A1 (ROSENBAUM JOHN M [US] ET AL) 7 September 2006 (2006-09-07) table VII -----	1-3
Y	US 2003/236177 A1 (WU MARGARET MAY-SOM [US] ET AL) 25 December 2003 (2003-12-25) claims; examples -----	1-4
Y	WO 2004/074412 A2 (SHELL INT RESEARCH [NL]; BABA YOSHIHARU [JP]; MORIKI KEIICHI [JP]; NAG) 2 September 2004 (2004-09-02) page 21, line 30 - page 22, line 13; claims; examples; table 1 -----	1-4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2013/077539

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2009181871	A1	16-07-2009	NONE
WO 2006116775	A1	02-11-2006	AU 2006239188 A1 02-11-2006
		BR PI0610621 A2 13-07-2010	
		CA 2609756 A1 02-11-2006	
		CN 101203587 A 18-06-2008	
		EP 1877529 A1 16-01-2008	
		JP 5091118 B2 05-12-2012	
		JP 2008540701 A 20-11-2008	
		KR 20080005590 A 14-01-2008	
		WO 2006116775 A1 02-11-2006	
		ZA 200709215 A 29-10-2008	
US 2007142250	A1	21-06-2007	AU 2006331635 A1 05-07-2007
		BR PI0620160 A2 03-07-2012	
		CN 101365773 A 11-02-2009	
		EP 1973997 A2 01-10-2008	
		JP 5334591 B2 06-11-2013	
		JP 2009521572 A 04-06-2009	
		KR 20080081056 A 05-09-2008	
		US 2007142250 A1 21-06-2007	
		WO 2007075831 A2 05-07-2007	
		ZA 200806229 A 25-11-2009	
US 2006199743	A1	07-09-2006	AU 2006221003 A1 14-09-2006
		CN 101287814 A 15-10-2008	
		GB 2438989 A 12-12-2007	
		JP 2008531813 A 14-08-2008	
		NL 1031299 A1 06-09-2006	
		NL 1031299 C2 25-06-2007	
		US 2006199743 A1 07-09-2006	
		WO 2006096304 A2 14-09-2006	
		ZA 200707485 A 28-01-2009	
US 2003236177	A1	25-12-2003	AU 2003213706 A1 22-09-2003
		CA 2477331 A1 18-09-2003	
		EP 1490459 A1 29-12-2004	
		JP 2005519184 A 30-06-2005	
		US 2003236177 A1 25-12-2003	
		US 2005250657 A1 10-11-2005	
		WO 03076555 A1 18-09-2003	
WO 2004074412	A2	02-09-2004	AU 2004213598 A1 02-09-2004
		BR PI0407521 A 14-02-2006	
		CA 2516271 A1 02-09-2004	
		CN 1751115 A 22-03-2006	
		EP 1594943 A2 16-11-2005	
		JP 5057630 B2 24-10-2012	
		JP 2004250504 A 09-09-2004	
		KR 20050098951 A 12-10-2005	
		MX PA05008671 A 18-10-2005	
		RU 2352621 C2 20-04-2009	
		US 2004224860 A1 11-11-2004	
		WO 2004074412 A2 02-09-2004	
		ZA 200506194 A 27-09-2006	