

DESCRIPTION

Title of Invention

RUBBER COMPOUNDING OIL, AROMATIC COMPOUND-
CONTAINING BASE OIL, AND METHODS FOR PRODUCING
SAME

Technical Field

[0001] The present invention relates to a rubber compounding oil, to an aromatic compound-containing base oil, and to methods for producing them.

Background Art

[0002] High-aromatic mineral oils have high affinity for rubber components, excellent processability and softening properties as rubber compositions, and excellent economy, and they are therefore used for production of rubber compositions comprising natural rubber, synthetic rubber and the like. For example, extender oils are added to synthetic rubber including SBR during their synthesis, while process oils are added to rubber processed products such as tires to improve their processability or the quality of the rubber processed products (Patent document 1, for example).

[0003] In Europe, restrictions prohibiting the use of substances containing more than minimum amounts of DMSO extracts or indicated carcinogenic polycyclic aromatic compounds in the production of tires or tire parts have been enacted since 2010. Consequently, extracts obtained by extraction of vacuum distillation fractions with polar solvents generally have high polycyclic aromatic contents, and they are gradually becoming unusable directly as rubber compounding oils, in

light of the aforementioned restrictions. A demand therefore exists for rubber compounding oils that can meet such restrictions.

[0004] As a rubber compounding oil that meets the restrictions, Patent document 1 proposes a petroleum-based process oil having an aromatic hydrocarbon content (C_A) of 20-35 mass%, a glass transition temperature T_g of between -55°C and -30°C , a dynamic viscosity (100°C) of 20-50 mm^2/s and a polycyclic aromatic component content (PCA) of no greater than 3 mass% in the petroleum-based process oil. When rubber obtained by adding such a petroleum-based process oil to diene rubber is used in a tire, it is possible to achieve both low fuel consumption and grip properties, and to increase the thermal aging resistance or heat-resistant wearability.

[0005] Incidentally, known common rubber compounding oils include high aromatic compound-containing base oils comprising solvent extracts of vacuum distillation fractions or deasphalted oil (Patent document 2, for example). Consequently, solvent extracts obtained by extraction of vacuum distillation fractions generally have high polycyclic aromatic contents, and they are gradually becoming unusable directly as rubber compounding oils, due to the aforementioned restrictions. Known techniques designed against this situation include methods of reducing aromaticity by hydrogenation treatment of aromatic compounds, or of increasing extract yield through a polycyclic aromatic-diluting effect. However, such methods can potentially lower economy due to the addition of hydrogenation equipment, or can lower aromaticity or impair the yields of lubricant base oil by-products.

[0006] On the other hand, it is desirable for rubber compounding oils to

have high flash points (250°C or higher) to avoid falling within the definition of Level-4 Hazardous Petroleum, for improved manageability and handleability.

5 [0007] In addition, rubber compounding oils must have low glass transition points in order to improve the low-temperature characteristics (such as low-temperature elastic modulus) for rubber. For example, Patent documents 1 and 3 propose rubber compounding oils having low glass transition points of between -55°C and -30°C and between -45 and -20°C, respectively.

10 [0008] However, because the glass transition point and aromatic content are generally reciprocal properties, it has been difficult in most cases to achieve both a high aromatic content and a low glass transition point. For example, it is not been possible to obtain a rubber compounding oil with an aromatic content of 50 mass% or greater according to ASTM D 15 2007 and a glass transition point of no higher than -45°C (see examples and comparative examples of Patent document 3, for example). A lower glass transition point of the rubber compounding oil tends to reduce not only the aromatic content but also the flash point.

20 [0009] Therefore, common high aromatic compound-containing base oils containing unrefined extracts have high pour points and high glass transition points. Lubricant base oils obtained by refining of raffinates obtained by polar solvent extraction of vacuum distillation fractions exhibit low pour points, but also have low aromatic contents and a high aniline points, and are therefore difficult to use in extender oils for 25 production of SBR, in particular.

[0010] Methods for producing non-carcinogenic rubber compounding

oils are known including production method (1): a method of hydrogenation treatment of the extract to reduce polycyclic aromatic compounds, production method (2): a method of increasing the yield of the extract in the extraction step using a polar solvent, to dilute the polycyclic aromatic compounds, and production method (3): a method of carrying out solvent extraction of the vacuum distillation fraction in two stages (Patent document 2).

[0011] Also, if the flash point is 250°C or higher, such aromatic compound-containing base oils do not qualify as level-4 hazardous petroleum and are more easily manageable, and therefore a high flash point is also desirable. Rubber compounding oils with low glass transition points have also been proposed (Patent documents 1 and 3, for example).

[0012] As a rubber compounding oil to be added to diene rubber, Patent document 1 proposes the use of a petroleum-based process oil having an aromatic hydrocarbon content (C_A) of 20-35 wt%, a glass transition temperature T_g of between -55°C and -30°C, a dynamic viscosity (100°C) of 20-50 mm²/s and a polycyclic aromatic component content (PCA) of no greater than 3 wt%. When a rubber obtained by adding such a petroleum-based process oil to diene rubber is used in a tire, it is possible to achieve both low fuel consumption and grip properties, and to increase the thermal aging resistance or heat-resistant wearability.

[Citation List]

[Patent literature]

[0013] [Patent document 1] Japanese Unexamined Patent Application Publication No. 2004-155959

[Patent document 2] Japanese Patent Publication No. 3658155

[Patent document 3] International Patent Publication No. WO97/35462

Summary of the Invention

Technical Problem

5 [0014] In production method (1), however, economy is compromised when hydrogenation equipment is added and the aromatic content of the product also tends to be lowered, while in production method (2), the yield of the lubricant base oil obtained from the raffinate tends to be lowered and its aromatic content also tends to be lowered. In
10 production method (3) as well, despite a density of less than 0.94 g/cm^3 , a low polycyclic aromatic content and a high yield of extract as a high aromatic portion, the raffinate yield is very low and the aromatic content tends to be reduced.

[0015] Thus, no method has been known for accomplishing high-yield
15 production of aromatic compound-containing base oils with high flash points and low glass transition points, as well as high total aromatic contents and sufficient reduction of indicated carcinogenic polycyclic aromatic compounds, from raffinates and extracts obtained by polar solvent extraction processes using vacuum distillation fractions as
20 starting materials. In particular, no method has been known for obtaining non-carcinogenic high aromatic base oils from extracts at high yield, which are useful as rubber compounding oils or their base materials, or for obtaining non-carcinogenic aromatic compound-containing base oils from raffinates, useful as rubber compounding oils
25 or their base materials, and as lubricant base oils.

[0016] It is therefore a first object of the present invention to provide a

rubber compounding oil having a high flash point and a low glass transition point while maintaining a high total aromatic content, and having sufficient reduction in the content of indicated polycyclic aromatic compounds, as well as a method for producing the rubber compounding oil.

[0017] It is a second object of the invention to provide an aromatic compound-containing base oil having a high flash point, a low glass transition point, a high total aromatic content and sufficient reduction in the content of carcinogenic substances, from a raffinate and extract obtained by a polar solvent extraction process using a vacuum distillation fraction as the starting material, as well as a method for producing an aromatic compound-containing base oil which allows high-yield production of the aromatic compound-containing base oil.

Solution to Problem

[0018] According to a first aspect, the invention provides a rubber compounding oil having a total aromatic content of 50 mass% or greater according to ASTM D 2007 or ASTM D 2549, a flash point of 250°C or higher, a difference of at least 45°C between the pour point and glass transition point, a benzo(a)pyrene content of no greater than 1 ppm by mass and a total content of the following indicated aromatic compounds 1) to 8) of no greater than 10 ppm by mass.

[0019] 1) Benzo(a)pyrene (BaP)

2) Benzo(e)pyrene (BeP)

3) Benzo(a)anthracene (BaA)

4) Chrysene (CHR)

5) Benzo(b)fluoranthene (BbFA)

6) Benzo(j)fluoranthene (BjFA)

7) Benzo(k)fluoranthene (BkFA)

8) Dibenzo(a,h)anthracene (DBAhA)

5 [0020] The rubber compounding oil of the invention has a high flash point and a low glass transition point while maintaining a high total aromatic content, and it has sufficient reduction in the content of indicated polycyclic aromatic compounds.

10 [0021] The rubber compounding oil of the invention comprises at least an aromatic compound-containing base oil (b), among an aromatic compound-containing base oil (a) containing a raffinate obtained by separating a vacuum distillation fraction of atmospheric distillation residue oil from crude oil by a solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 60-600 mm²/s, an aniline point of 70°C or higher, a 10% point of 400-500°C and a 90%
15 point of 500-600°C in GC distillation, a %C_A of 3-20 according to ASTM D 3238 and a glass transition point of no higher than -30°C, and an aromatic compound-containing base oil (b) containing an extract obtained by separating the vacuum distillation fraction of atmospheric distillation residue oil by a solvent extraction step, or a refined oil
20 product thereof, and having a 40°C dynamic viscosity of 200 mm²/s or greater, an aniline point of no higher than 90°C, a 15°C density of 0.94 g/cm³ or greater, and a total aromatic content of 30 mass% or greater according to ASTM D 2549, wherein the content of the aromatic compound-containing base oil (a) is no greater than 95 mass% and the
25 content of the aromatic compound-containing base oil (b) is 5 mass% or greater.

[0022] According to the invention, preferably the solvent extraction step includes a first solvent extraction step in which a vacuum distillation fraction is contacted with a polar solvent in a first extraction column having a bottom temperature of 30-90°C and a top temperature that is higher than the bottom temperature, to obtain a first raffinate and a first extract, and a second solvent extraction step in which the first raffinate is contacted with a polar solvent in a second extraction column having a bottom temperature and top temperature that are each at least 10°C higher than those of the first extraction column, to obtain a second raffinate and a second extract, and preferably the aromatic compound-containing base oil (b) includes the second extract or a refined oil product thereof.

[0023] Furthermore, according to the invention there is provided a method for producing a rubber compounding oil, which comprises a blending step of blending an aromatic compound-containing base oil (a) containing a raffinate obtained by separating a vacuum distillation fraction of atmospheric distillation residue oil from crude oil by a solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 60-600 mm²/s, an aniline point of 70°C or higher, a 10% point of 400-500°C and a 90% point of 500-600°C in GC distillation, a %C_A of 3-20 according to ASTM D 3238 and a glass transition point of no higher than -30°C, and an aromatic compound-containing base oil (b) containing an extract obtained by separating the vacuum distillation fraction of atmospheric distillation residue oil by a solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 200 mm²/s or greater, an aniline point of no

higher than 90°C, a 15°C density of 0.94 g/cm³ or greater, and a total aromatic content of 30 mass% or greater according to ASTM D 2549, wherein the total aromatic content is 50 mass% or greater according to ASTM D 2007 or ASTM D 2549, the flash point is 250°C or higher, the difference between the pour point and the glass transition point is at least 45°C, the benzo(a)pyrene content is no greater than 1 ppm by mass and the total content of the following indicated aromatic compounds 1) to 8) is no greater than 10 ppm by mass, and wherein the content of the aromatic compound-containing base oil (a) is greater than 0 and no greater than 95 mass% and the content of the aromatic compound-containing base oil (b) is 5 mass% or greater and less than 100 mass%.

[0024] 1) Benzo(a)pyrene (BaP)

2) Benzo(e)pyrene (BeP)

3) Benzo(a)anthracene (BaA)

4) Chrysene (CHR)

5) Benzo(b)fluoranthene (BbFA)

6) Benzo(j)fluoranthene (BjFA)

7) Benzo(k)fluoranthene (BkFA)

8) Dibenzo(a,h)anthracene (DBAhA)

[0025] A rubber compounding oil obtained by the production method of the invention has a high flash point and a low glass transition point while maintaining a high total aromatic content, and it has sufficient reduction in the content of indicated polycyclic aromatic compounds. Also, since it is a specific feature of the rubber compounding oil comprising the aromatic compound-containing base oil (b) according to the invention that the difference between the pour point and the glass

transition point is at least 45°C, and especially at least 60°C, it is possible to obtain a rubber compounding oil with a glass transition point of no higher than -45°C even if the total aromatic content is 50 mass% or greater and the pour point is 15°C or higher. The economy is also excellent since there is no need for refining of the aromatic compound-containing base oil (b) by dewaxing treatment, hydrogenation treatment or the like.

[0026] According to a second aspect, the invention provides a method for producing an aromatic compound-containing base oil comprising a first solvent extraction step in which a vacuum distillation fraction of atmospheric distillation residue oil from crude oil is contacted with a polar solvent in a first extraction column having a bottom temperature of 30-90°C and a top temperature that is higher than the bottom temperature, to obtain a first raffinate and a first extract, and a second solvent extraction step in which the first raffinate is contacted with a polar solvent in a second extraction column having a bottom temperature and top temperature that are each at least 10°C higher than those of the first extraction column, to obtain a second raffinate and a second extract having a 15°C density of 0.94 g/cm³ or higher and a total aromatic content of 30 mass% or greater, the aromatic compound-containing base oil including at least a portion of the second extract and the second raffinate or a refined oil product thereof, and having a total aromatic content of 30 mass% or greater.

[0027] According to the invention it is possible to accomplish high-yield production of an aromatic compound-containing base oil having a high flash point, a low glass transition point, and a high total aromatic

content while having a sufficiently reduced content of indicated carcinogenic substances. Such an aromatic compound-containing base oil can be suitably used as a rubber compounding oil or a starting material therefor.

5 [0028] Preferably, the invention comprises, after the second solvent extraction step, a base oil preparation step in which the second raffinate is subjected to refining treatment including dewaxing treatment to obtain a refined oil, to produce an aromatic compound-containing base oil comprising the refined oil and having a pour point of no higher than
10 -5°C, an aniline point of 90°C or higher, a viscosity index of 90 or greater and a flash point of 250°C or higher.

[0029] Preferably according to the invention, the second solvent extraction step is followed by a base oil preparation step in which the second raffinate is subjected to refining treatment including dewaxing
15 treatment to obtain a refined oil, to produce an aromatic compound-containing base oil comprising the refined oil and having a 40°C dynamic viscosity of 60-120 mm²/s, and a 10% point of 400-460°C and a 90% point of 500-540°C in gas chromatography distillation.

[0030] Preferably according to the invention, the second solvent extraction step is followed by a base oil preparation step in which the second raffinate is subjected to refining treatment including dewaxing
20 treatment to obtain a refined oil, to produce an aromatic compound-containing base oil comprising the refined oil and having a 40°C dynamic viscosity of 120-250 mm²/s, and a 10% point of 450-520°C and a 90% point of 540-600°C in gas chromatography distillation.

25 [0031] Preferably, the aromatic compound-containing base oil obtained

by the production method of the invention comprises at least a portion of the second extract, and has a 40°C dynamic viscosity of 200 mm²/s or greater, a flash point of 250°C or higher, a pour point of no higher than 30°C, an aniline point of no higher than 90°C, a glass transition point of no higher than -30°C and a difference of at least 50°C between the pour point and the glass transition point. The aromatic compound-containing base oil may also consist of the second extract.

[0032] Also preferably, the aromatic compound-containing base oil obtained by the production method of the invention comprises at least a portion of the second extract, and has a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s, and a glass transition point of between -60 and -40°C. Such an aromatic compound-containing base oil may be most suitably used as a petroleum-based process oil or extender oil to be added to natural rubber (NR), or diene rubbers including various butadiene rubbers (BR), various styrene-butadiene copolymer rubbers (SBR), polyisoprene rubber (IR), butyl rubber (BR) and any blended rubbers thereof, and especially to diene rubbers comprising at least one type of styrene-butadiene copolymer rubber. The aromatic compound-containing base oil may also consist of the second extract.

[0033] Preferably, the aromatic compound-containing base oil obtained by the production method of the invention comprises at least a portion of the second extract, and has a 40°C dynamic viscosity of 500 mm²/s or greater, and a glass transition point of between -50 and -30°C. Such an aromatic compound-containing base oil can be most suitably used, for example, as a petroleum-based process oil or extender oil to be added to

a diene rubber as mentioned above. The aromatic compound-containing base oil may also consist of the second extract.

[0034] Preferably, the aromatic compound-containing base oil obtained by the production method of the invention has a benzo(a)pyrene content of no greater than 1 ppm by mass, and a total content of the following indicated aromatic compounds 1) to 8) of no greater than 10 ppm by mass.

1) Benzo(a)pyrene (BaP)

2) Benzo(e)pyrene (BeP)

3) Benzo(a)anthracene (BaA)

4) Chrysene (CHR)

5) Benzo(b)fluoranthene (BbFA)

6) Benzo(j)fluoranthene (BjFA)

7) Benzo(k)fluoranthene (BkFA)

8) Dibenzo(a,h)anthracene (DBAhA)

[0035] Such an aromatic compound-containing base oil can be most suitably used as a petroleum-based process oil or extender oil, or a base material therefor, in a rubber processed product such as a tire, because it has a sufficiently reduced content of indicated carcinogenic polycyclic aromatic compounds.

[0036] The invention further provides an aromatic compound-containing base oil obtained by the production method having the features described above. The aromatic compound-containing base oil has a high flash point, a low glass transition point and a high total aromatic content while also having sufficient reduction in the content of carcinogenic substances, and it therefore has excellent properties as a

petroleum-based process oil or extender oil, or a base material therefor, as well as sufficiently excellent safety.

[0037] According to a third aspect, the invention further provides a rubber compounding oil comprising an aromatic compound-containing base oil having the features described above.

Effects of the Invention

[0038] According to the invention it is possible to provide a rubber compounding oil having a high flash point and a low glass transition point while maintaining a high total aromatic content, and having sufficient reduction in the content of indicated polycyclic aromatic compounds, as well as a method for producing the rubber compounding oil.

[0039] Because the rubber compounding oil of the invention has a high aromatic content, it has extremely high compatibility with rubber and rubber materials including styrene-butadiene-based rubber. In addition, when it is used as an extender oil or process oil in a rubber or rubber material such as styrene-butadiene-based rubber having a glass transition point of between about -57 and -44°C, it is possible to produce rubber with excellent low-temperature characteristics. Furthermore, because its flash point is high and it has a sufficiently reduced content of carcinogenic polycyclic aromatic compounds, it is also highly safe. In particular, by preparing a rubber compounding oil with an aromatic hydrocarbon content (C_A) of 20-35 mass% (equivalent to % C_A of 20-35 according to ASTM D3238), a glass transition temperature T_g of between -55°C and -30°C and a dynamic viscosity (100°C) of 20-50 mm²/s, as in Patent document 1, and adding this to, for

example, natural rubber (NR), or a diene rubber such as any of various butadiene rubbers (BR), various styrene-butadiene copolymer rubbers (SBR), polyisoprene rubber (IR), butyl rubber (BR) or any blended rubbers thereof, and especially to a diene rubber comprising at least one type of styrene-butadiene copolymer rubber, and using the obtained rubber in a tire, it is possible to obtain low fuel consumption and satisfactory grip properties, as well as to improve the thermal aging resistance and heat-resistant wearability.

[0040] According to the invention it is also possible to provide an aromatic compound-containing base oil with a high flash point and a low glass transition point, as well as a high total aromatic content and sufficient reduction in the content of carcinogenic substances, from a raffinate and extract obtained by a polar solvent extraction process using a vacuum distillation fraction as starting material. It is also possible to provide a method for producing an aromatic compound-containing base oil, which allows high-yield production of such an aromatic compound-containing base oil.

Brief Description of the Drawings

[0041]

Fig. 1 is a process drawing showing a preferred embodiment of the method for producing a rubber compounding oil according to the invention.

Description of the Embodiments

[0042] Preferred embodiments of the invention will now be explained with reference to the accompanying drawings where necessary. The rubber compounding oil of this embodiment preferably has the

following properties, for excellent affinity for rubber, softening properties, flash point and safety, as well as a high level of properties as a rubber composition including low fuel consumption, grip properties, thermal aging resistance and heat-resistant wearability.

5 [0043] Aromatic content according to ASTM D2007 (Clay-gel method): Generally 50-90 mass%, preferably 55 mass% or greater, even more preferably 57 mass% or greater, most preferably 60 mass% or greater, and preferably no greater than 80 mass% and more preferably no greater than 70 mass%.

10 [0044] Saturated components according to ASTM D2007 (Clay-gel method): Generally 5-50 mass%, preferably 10 mass% or greater, more preferably 20 mass% or greater, and preferably no greater than 40 mass% and more preferably no greater than 30 mass%.

15 [0045] Polar compound content according to ASTM D2007 (Clay-gel method): Generally 1-20 mass%, preferably 2 mass% or greater, more preferably 5 mass% or greater, and preferably no greater than 15 mass%, more preferably no greater than 12 mass% and even more preferably no greater than 10 mass%.

20 [0046] Saturated component/polar compound ratio according to ASTM D2007 (Clay-gel method): Generally 0.25-50, preferably 1 or greater, more preferably 2.5 or greater and even more preferably 3 or greater, and preferably no greater than 20, more preferably no greater than 10 and even more preferably no greater than 5.

25 [0047] The benzo(a)pyrene (BaP) content is no greater than 1 ppm by mass, and the total content of the following indicated aromatic compounds (PAH) 1) to 8) is no greater than 10 ppm by mass. This

will yield a more highly safe rubber compounding oil with adequately minimal concerns of carcinogenicity.

[0048] 1) Benzo(a)pyrene (BaP)

2) Benzo(e)pyrene (BeP)

5 3) Benzo(a)anthracene (BaA)

4) Chrysene (CHR)

5) Benzo(b)fluoranthene (BbFA)

6) Benzo(j)fluoranthene (BjFA)

7) Benzo(k)fluoranthene (BkFA)

10 8) Dibenzo(a,h)anthracene (DBAhA)

[0049] "Benzo(a)pyrene", as used herein, is the benzo(a)pyrene (BaP) listed as 1) above, and an "indicated aromatic compound" is any of the aromatic compounds (PAH) listed as 1) to 8) above. These indicated aromatic compounds can be quantitatively analyzed by GC-MS analysis, after separation and concentration of the components of interest, upon preparing a sample containing an added internal standard substance.

15 [0050] The flash point is 250°C or higher, preferably 260°C or higher and more preferably 280°C or higher, and preferably no higher than 350°C, more preferably no higher than 320°C and even more preferably no higher than 310°C. The "flash point", as used herein, is the flash point based on Cleveland open-cup (COC), measured according to JIS K2265.

20 [0051] The difference between the pour point and the glass transition point is at least 45°C, preferably at least 50°C, more preferably at least 60°C and even more preferably at least 65°C, and preferably no more than 100°C and more preferably no more than 80°C.

[0052] The pour point is preferably no higher than 30°C and more preferably no higher than 25°C, and preferably -10°C or higher, more preferably 5°C or higher, even more preferably +10°C or higher and most preferably +12.5°C or higher. The "pour point", as used herein, is the pour point measured according to JIS K2269.

[0053] The glass transition point (T_g) is preferably no higher than -30°C, more preferably no higher than -40°C, even more preferably no higher than -45°C, particularly preferably no higher than -48°C and more particularly preferably no higher than -50°C, and preferably -80°C or higher, more preferably -60°C or higher and even more preferably -55°C or higher.

[0054] The "glass transition point (T_g)", as used herein, is the glass transition point obtained from the peak of thermal change in the glass transition range, as measured by DSC (differential scanning calorimeter) at a fixed temperature-elevating rate (10°C/min). The initial temperature is usually about 30°C-50°C lower than the anticipated glass transition point, or a lower temperature, and temperature elevation is initiated after maintaining this initial temperature for a prescribed period of time. Specifically, measurement may be conducted under the following conditions for this embodiment.

[0055] Apparatus: DSC Q100 Thermal Analysis System by TA Instruments

Initial temperature: -90°C, maintained for 10 minutes

Temperature-elevating rate: 10°C/min

Final temperature: 50°C, maintained for 10 minutes

[0056] The method of calculating the glass transition point from the peak of thermal change may be the method according to JIS K 7121.

[0057] The 15°C density is generally 0.9 g/cm³-1.0 g/cm³, preferably 0.94 g/cm³ or greater and more preferably 0.945 g/cm³ or greater, and preferably no greater than 0.98 g/cm³ and more preferably no greater than 0.96 g/cm³.

[0058] The 40°C dynamic viscosity is generally 200-3000 mm²/s, preferably 300 mm²/s or greater, more preferably 400 mm²/s and even more preferably 500 mm²/s or greater, and preferably no greater than 2000 mm²/s, more preferably no greater than 1000 mm²/s and even more preferably no greater than 800 mm²/s. The dynamic viscosity at each temperature, as used herein, is the dynamic viscosity at each temperature measured according to JIS K2283.

[0059] The 100°C dynamic viscosity is generally 10-100 mm²/s, more preferably 15 mm²/s or greater and even more preferably 20 mm²/s or greater, and preferably no greater than 60 mm²/s, more preferably 50 mm²/s and even more preferably no greater than 32 mm²/s.

[0060] The aniline point is generally 50-100°C, preferably 60°C or higher, more preferably 65°C or higher and even more preferably 70°C or higher, and preferably no higher than 90°C and more preferably no higher than 85°C. The "aniline point" as used herein is the aniline point measured according to JIS K 2256-1985.

[0061] The nitrogen content is generally 0.01-0.2 mass%, preferably 0.03 mass% or greater and even more preferably 0.05 mass% or greater, and preferably no greater than 0.15 mass% and more preferably no greater than 0.1 mass%. The "nitrogen content", as used herein, is the

nitrogen content measured by chemiluminescence according to JIS K2609.

[0062] The %C_N value is generally 5-30, preferably 10 or greater and more preferably 14 or greater, and preferably no greater than 25 and more preferably no greater than 20. The %C_A value is generally 10-40, preferably 17 or greater and more preferably 20 or greater, and preferably no greater than 35, more preferably no greater than 30 and even more preferably no greater than 25. The %C_P value is generally 30-85, preferably 40 or greater and more preferably 50 or greater, and preferably no greater than 73 and more preferably no greater than 66. The %C_P, %C_N and %C_A values referred to throughout the present specification are, unless otherwise specified, the percentage of paraffinic carbons with respect to total carbon atoms, the percentage of naphthenic carbons with respect to total carbons and the percentage of aromatic carbons with respect to total carbons, respectively, as determined by the method of ASTM D 3238-85 (n-d-M ring analysis).

[0063] The total aromatic content is generally 30-90 mass%, preferably 40 mass% or greater and more preferably 50 mass% or greater, and preferably no greater than 80 mass% and more preferably no greater than 70 mass%. The "total aromatic content", as used herein is, unless otherwise specified, the content of the aromatics fraction measured according to ASTM D 2007 or ASTM D 2549.

[0064] The rubber compounding oil of this embodiment comprises an aromatic compound-containing base oil (a) that includes an aromatic compound-containing base oil containing a raffinate obtained by separating a vacuum distillation fraction of atmospheric distillation

residue oil from crude oil by a solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 60-600 mm²/s, an aniline point of 70°C or higher, a 10% point of 400-500°C and a 90% point of 500-600°C in GC distillation, a %C_A of 3-20 according to ASTM D 3238 and a glass transition point of no higher than -30°C, and an aromatic compound-containing base oil (b) containing an extract obtained by separating the vacuum distillation fraction of atmospheric distillation residue oil by a solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 200 mm²/s or greater, an aniline point of no higher than 90°C, a 15°C density of 0.94 g/cm³ or greater, and a total aromatic content of 30 mass% or greater according to ASTM D 2549. The aromatic compound-containing base oil (a) (hereunder referred to as "base oil (a)") and the aromatic compound-containing base oil (b) (hereunder referred to as "base oil (b)") will now be explained.

[0065] The base oil (a) is preferably an aromatic compound-containing base oil with a total aromatic content of 30 mass% or greater, obtained by refining treatment of a second raffinate obtained by a first solvent extraction step in which a vacuum distillation fraction of atmospheric distillation residue oil from crude oil is contacted with a polar solvent in a first extraction column having a bottom temperature of 30-90°C and a top temperature that is higher than the bottom temperature, to obtain a first raffinate and a first extract, and a second solvent extraction step in which the first raffinate is contacted with a polar solvent in a second extraction column having a bottom temperature and a top temperature that are both at least 10°C higher than those of the first extraction

column, to obtain a second raffinate and a second extract having a 15°C density of 0.94 g/cm³ or greater and a total aromatic content of 30 mass% or greater.

5 [0066] The base oil (a) is an aromatic compound-containing base oil obtained by refining treatment including dewaxing treatment of a second raffinate, and it is preferably a base oil (a1) having a 40°C dynamic viscosity of 60-120 mm²/s, and a 10% point of 400-460°C and a 90% point of 500-540°C in GC distillation and/or a base oil (a2) having a 40°C dynamic viscosity of 120-250 mm²/s and a 10% point of 450-520°C and a 90% point of 540-600°C in GC distillation.

10 [0067] The base oil (b) is preferably an aromatic compound-containing base oil having a 40°C dynamic viscosity of 200 mm²/s or greater, a flash point of 250°C or higher, a pour point of no higher than 30°C, an aniline point of no higher than 90°C, a glass transition point of no higher than -30°C and a difference of at least 45°C between the pour point and the glass transition point.

15 [0068] The base oil (b) is preferably an aromatic compound-containing base oil (b1) having a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s and a glass transition point of between -60 and -40°C and/or an aromatic compound-containing base oil (b2) having a 40°C dynamic viscosity of 500 mm²/s or greater and a glass transition point of between -50 and -30°C.

[0069] The preferred properties for the base oil (a) and base oil (b) will now be described in detail.

25 [0070] The pour point of the base oil (a) is preferably no higher than -10°C, and may even be lower than -20°C. From the viewpoint of

production cost of the rubber compounding oil, however, the pour point of the base oil (a) is more preferably between -10 and -20°C . By using a base oil (a) with a pour point of no higher than -10°C , it is possible to easily obtain a rubber compounding oil having an even lower glass transition point.

[0071] The glass transition point of the base oil (a) is preferably no higher than -30°C and more preferably no higher than -50°C , and preferably -100°C or higher, more preferably -80°C or higher and even more preferably -70°C or higher. If the glass transition point is too high it will tend to be difficult to obtain a rubber compounding oil with a low glass transition point, while if the glass transition point is too low the production cost will tend to increase because the dewaxing conditions will need to be excessively stringent.

[0072] The aniline point of the base oil (a) is preferably 70°C or higher, more preferably 90°C or higher and even more preferably 100°C or higher. From the viewpoint of facilitating production of a rubber compounding oil having a suitable aniline point for excellent compatibility with rubber and to maintain the properties of the rubber composition, it is preferably no higher than 120°C .

[0073] As the composition of the base oil (a), $\%C_A$ is preferably 3-20 and more preferably 5-10, and $\%C_N$ is preferably 15-35 and more preferably 20-30. The $\%C_P$ value of the base oil (a) is determined according to $\%C_A$ and $\%C_N$, and it is preferably 45-82, more preferably 60-75 and even more preferably 65-70. By using base oil (a) having a composition within this range, it will be easy to produce a rubber compounding oil having excellent compatibility with rubber and having

a suitable composition for maintaining the properties of the rubber composition.

[0074] The nitrogen content of the base oil (a) is preferably no greater than 0.01 mass% and more preferably no greater than 0.008 mass%, and it may even be less than 0.001 mass%. However, from the viewpoint of economy, considering that production cost for the rubber compounding oil can be reduced by using a lubricant base oil of low purity, the content is preferably 0.002 mass% or greater and more preferably 0.003 mass% or greater.

[0075] The flash point of the base oil (a) is 250°C or higher and preferably 255°C or higher, for a rubber compounding oil flash point of 250°C or higher, from the viewpoint of not falling within the definition of a level-4 hazardous petroleum material. Because the flash point of the base oil (b) can be high and therefore the flash point of the base oil (a) does not need to be higher than necessary, it is preferably no higher than 290°C and more preferably no higher than 280°C.

[0076] The 90% point of the base oil (a) in GC distillation is 500°C or higher and preferably 500-600°C. For the base oil (a1) as one mode of the base oil (a), this may be 510-550°C, and for the base oil (a2) as another mode of the base oil (a), it may be 550-590°C. There are no particular restrictions on the 10% point of the base oil (a) in GC distillation, but it is preferably 400-510°C and more preferably 440-500°C from the viewpoint of resulting in a flash point of 250°C or higher for the rubber compounding oil and avoiding falling under the definition of a level-4 hazardous petroleum. The base oil (a1) used may be one having a 10% point of 440-470°C in GC distillation, and the

base oil (a2) may be one having a 10% point of 450-500°C in GC distillation.

[0077] The base oil (a) has a content of benzo(a)pyrene (BaP) listed as 1) above of preferably no greater than 1 ppm by mass, and a total content of indicated aromatic compounds (PAH) listed as 1) to 8) above of preferably no greater than 10 ppm by mass. This will allow production of a more highly safe rubber compounding oil with adequately reduced carcinogenicity.

[0078] The 40°C dynamic viscosity of the base oil (a) is preferably 60-600 mm²/s, more preferably 60-300 mm²/s and even more preferably 70-200 mm²/s.

[0079] When a base oil (b) with a 40°C dynamic viscosity of less than 2000 mm²/s is used, it is preferred for the 40°C dynamic viscosity of the base oil (a1) to be 50-500 mm²/s and more preferably 60-80 mm²/s and/or that of the base oil (a2) to be 120-250 mm²/s, in order to obtain a rubber compounding oil with a suitable dynamic viscosity.

[0080] There are no particular restrictions on the total aromatic content of the base oil (a), but it will usually be 20 mass% or greater, preferably 30 mass% or greater and even more preferably 35 mass% or greater, and preferably no greater than 50 mass% and more preferably no greater than 45 mass%. If the total aromatic content of the base oil (a) is less than 20 mass%, it will tend to be difficult to obtain a rubber compounding oil with high aromaticity. If the total aromatic content of the base oil (a) exceeds 50 mass%, on the other hand, the oxidation stability when it is used as a lubricant base oil will be reduced and it will less easily serve as both a lubricant base oil and for a rubber

compounding oil, while the economy of the petroleum refining process as a whole will tend to be reduced.

[0081] The aniline point of the base oil (b) is preferably 40-90°C, more preferably 45-70°C and even more preferably 50-65°C. If the aniline point is within this range, it will be possible to easily produce a rubber compounding oil having excellent compatibility with rubber and having a suitable aniline point for maintaining the properties of the rubber composition, even if it contains a lubricant base oil with a high aniline point.

[0082] As the composition of the base oil (b), %C_A is preferably 25-45 and more preferably 30-40, and %C_N is preferably 5-20 and more preferably 6-12. The %C_P value is determined according to %C_A and %C_N, and it is preferably 35-70 and more preferably 48-64. If the composition of the base oil (b) is within this range, it will be easy to produce a rubber compounding oil having excellent compatibility with rubber and having a suitable composition for maintaining the properties of the rubber composition, even with addition of a base oil (a) with high paraffinicity.

[0083] The nitrogen content of the base oil (b) is preferably 0.01 mass% or greater, more preferably 0.05 mass% or greater, even more preferably 0.1 mass% or greater and most preferably 0.15 mass% or greater. A high nitrogen content of the base oil (b) will result in a lower nitrogen content of raffinate obtained by the solvent extraction step, and higher purity of the lubricant base oil. Thus, using a base oil (b) with a high nitrogen content as the rubber compounding oil is preferred to allow effective utilization of the entire vacuum distillation fraction.

[0084] The pour point of the base oil (b) is preferably no higher than 30°C and more preferably no higher than 25°C. The pour point of the base oil (b) is preferably 5°C or higher, more preferably 10°C or higher, even more preferably 15°C or higher and most preferably 20°C or higher.

[0085] The base oil (b) may be an unrefined extract with a high pour point, but preferably it has a low glass transition point. The difference between the pour point and the glass transition point of the base oil (b) (pour point - glass transition point) is preferably at least 45°C, more preferably at least 50°C, even more preferably at least 55°C, and most preferably at least 60°C, and also preferably no more than 100°C and even more preferably no more than 80°C.

[0086] The glass transition point of the base oil (b) is preferably no higher than -30°C and preferably -60°C or higher. The glass transition point of the aromatic compound-containing base oil (b1), as one mode of the base oil (b), is between -60 and -40°C, while the glass transition point of the aromatic compound-containing base oil (b2) as another mode is between -50 and -30°C.

[0087] The base oil (b) has a content of benzo(a)pyrene (BaP) listed as 1) above of preferably no greater than 1 ppm by mass, and a total content of indicated aromatic compounds (PAH) listed as 1) to 8) above of preferably no greater than 10 ppm by mass. This will allow production of a more highly safe rubber compounding oil with adequately reduced carcinogenicity.

[0088] The flash point of the base oil (b) is not particularly restricted, but in order to avoid falling within the definition of Level-4 Hazardous

Petroleum according to the Japanese Fire Service Law, which specifies a flash point of 250°C or higher for rubber compounding oils, it is preferably 250°C or higher, more preferably 270°C or higher, even more preferably 290°C or higher and most preferably 300°C or higher.

5 [0089] The total aromatic content of the base oil (b) is preferably 30 mass% or greater, more preferably 50 mass% or greater, even more preferably 55 mass% or greater, yet more preferably 60 mass% or greater and most preferably 65 mass% or greater, and preferably no greater than 90 mass%, more preferably no greater than 80 mass% and
10 even more preferably no greater than 75 mass%.

[0090] If the total aromatic content of the base oil (b) is less than 50 mass% it will tend to be difficult to obtain a rubber compounding oil with high aromaticity, while a total aromatic content of greater than 90 mass% is not preferred from the viewpoint of economy since the extract
15 yield will be reduced.

[0091] Preferred embodiments of the method for producing a rubber compounding oil of the invention will now be explained.

[0092] This embodiment comprises a first solvent extraction step and a second solvent extraction step, in which the base oil (a) and base oil (b)
20 are produced, and a blending step in which the produced base oil (a) and base oil (b) are blended. The first solvent extraction step and second solvent extraction step in which the base oil (a) and base oil (b) are produced will be explained first.

[0093] In the first solvent extraction step, a vacuum distillation fraction
25 of atmospheric distillation residue oil from crude oil, and a polar solvent, are contacted in a first extraction column having a bottom temperature

of 30-90°C and a top temperature that is higher than the bottom temperature, to obtain a first raffinate and a first extract. In the second solvent extraction step, the first raffinate and a polar solvent are contacted in a second extraction column having a bottom temperature and top temperature that are both at least 10°C higher than those of the first extraction column, to obtain a second raffinate and a second extract having a 15°C density of 0.94 g/cm³ or greater and a total aromatic content of 30 mass% or greater. Each of these steps will now be explained in detail.

[0094]

(First solvent extraction step)

Fig. 1 is a process drawing for illustration of the first solvent extraction step and second solvent extraction step in the method for producing a rubber compounding oil according to this embodiment. In the first solvent extraction step, first a vacuum distillation fraction of atmospheric distillation residue oil from crude oil, and a polar solvent, are contacted in countercurrent flow in a first extraction column 30 having a bottom temperature of 30-90°C and a top temperature that is higher than the bottom temperature, to separate it into a first raffinate and a first extract. The polar solvent is supplied to the first extraction column 30 through tubing 34. The vacuum distillation fraction is supplied to the first extraction column 30 through tubing 16.

[0095] The vacuum distillation fraction is a fraction obtained by introducing common atmospheric distillation residue oil from crude oil into a vacuum distillation unit. The vacuum distillation fraction is not particularly restricted, and it may be the light lube-oil distillate, middle

lube-oil distillate, heavy lube-oil distillate, or a blend thereof, or the entire vacuum distillation fraction may be used. From the viewpoint of raising the flash point of the finally obtained aromatic compound-containing base oil, while also obtaining an aromatic compound-containing base oil with a suitable viscosity range such that the viscosity is not too high, it is preferred to use, for example, the 200-1500N, preferably 250-1200N and more preferably 300-600N or 600-1200N lube-oil distillate. The denotation "N" as used herein means that it is a neutral oil obtained from the vacuum distillation fraction, and for example, 300N means that the viscosity at 100°F (37.8°C) is 300 Saybolt universal seconds (SUS).

[0096] For this embodiment, the vacuum distillation fraction is preferably selected so that the base oil (a) has a viscosity of 200-1500N, preferably 250-600N or 600-1200N and more preferably 300-450N or 700-1000N.

[0097] The bottom temperature of the first extraction column 30 used in the first solvent extraction step is 30-90°C, preferably 50-70°C and more preferably 55-65°C. The top temperature of the first extraction column 30 is preferably higher, preferably 10-50°C higher, more preferably 15-40°C higher and even more preferably 25-35°C higher, than the bottom temperature. Specifically, the top temperature is preferably 60-120°C, more preferably 80-100°C and even more preferably 85-95°C.

[0098] The solvent ratio in the first solvent extraction step is preferably 0.5-3, more preferably 0.7-2 and even more preferably 1-1.5. The "solvent ratio" referred to herein is the volume ratio of the solvent with

respect to the starting material (solvent volume/starting material volume).

[0099] Under these conditions, the polar solvent and the vacuum distillation fraction are contacted in countercurrent flow inside the first extraction column 30 and a blend of the first extract and the polar solvent is obtained through tubing 38 from the column bottom of the first extraction column 30 while a blend of the first raffinate and the polar solvent is obtained through tubing 36 from the column top. Incidentally, the blend of the first extract and the polar solvent is fractionated into the first extract and polar solvent in a fractional distillation column, which is not shown. The blend of the first raffinate and polar solvent may also be fractionated into the first raffinate and polar solvent in a fractional distillation column, which is not shown, or it may be introduced directly into the second extraction column 40 without fractionation.

[0100] In the fractional distillation columns which are not shown, the polar solvent fractionated from the first extract and first raffinate is recovered and reutilized. The polar solvent may be a polar solvent such as furfural, phenol, cresol, sulfolane, N-methylpyrrolidone, dimethyl sulfoxide, formylmorpholine or a glycol-based solvent. According to this embodiment, furfural is preferably used from the viewpoint of allowing utilization of common solvent extraction equipment for lubricant base oils.

[0101] Thus, the vacuum distillation fraction is separated into the first raffinate and the first extract in the first solvent extraction step. The yield of the first raffinate obtained in the first solvent extraction step is

preferably 50-90 vol%, more preferably 60-85 vol% and even more preferably 70-80 vol%, based on the vacuum distillation fraction. The yield of the first extract obtained in the first solvent extraction step is preferably 10-50 vol%, more preferably 15-40 vol% and even more preferably 20-30 vol%, based on the vacuum distillation fraction.

[0102] Since the indicated aromatic compounds (PAH), mentioned below, are extracted at the first extract end by the first solvent extraction step, it is possible to adequately reduce the content of indicated aromatic compounds (PAH) in the second extract, the second raffinate and the aromatic compound-containing base oil obtained from them, at the subsequent stage. The first extract contains indicated aromatic compounds (PAH) and therefore tends to be unsuitable as a lubricant base oil or rubber compounding oil. Consequently, by limiting the first extract yield to no greater than, for example, 30 vol%, it is possible to obtain a high yield of the second raffinate and second extract, for use as a lubricant base oil or rubber compounding oil, or its base material. For example, the total of the second raffinate and second extract may be 70 vol% or greater based on the vacuum distillation fraction, and therefore the method for producing an aromatic compound-containing base oil of this embodiment can be considered very useful from the viewpoint of effective utilization of resources as well.

[0103] In the second solvent extraction step, the first raffinate or the blend of the first raffinate and polar solvent that are obtained in the first solvent extraction step and the polar solvent are introduced into the second extraction column 40 through tubing 36 and tubing 44, respectively, and the first raffinate and polar solvent are contacted in the

second extraction column 40. The second extraction column 40 has a bottom temperature and top temperature that are respectively at least 10°C higher than those of the first extraction column 30.

[0104] The bottom temperature of the second extraction column 40 used in the second solvent extraction step is at least 10°C higher, preferably 10-50°C higher, more preferably 15-40°C higher and even more preferably 20-30°C higher, than the bottom temperature of the first extraction column 30 in the first solvent extraction step. Specifically, the bottom temperature of the second extraction column 40 is preferably 40-140°C, more preferably 60-100°C and even more preferably 80-95°C.

[0105] The top temperature of the second extraction column 40 is preferably 10-50°C higher, more preferably 15-40°C higher and even more preferably 25-35°C higher, than the bottom temperature. More specifically, the top temperature of the second extraction column 40 is preferably 50-150°C, more preferably 80-140°C and even more preferably 110-130°C.

[0106] The solvent ratio in the second solvent extraction step is preferably 1-4, more preferably 1.3-3.5 and even more preferably 1.5-3.3. The solvent ratio in the second solvent extraction step is preferably at least 1.5 times the solvent ratio in the first solvent extraction step.

[0107] Under these conditions, the polar solvent and the first raffinate are contacted in countercurrent flow inside the second extraction column 40, and a blend of the second extract and the polar solvent is obtained through tubing 48 from the bottom part of the second

extraction column 40 while a blend of the second raffinate and the polar solvent is obtained through tubing 46 from the top part. Incidentally, the blend of the second extract and the polar solvent is fractionated into the second extract and polar solvent in a fractional distillation column, which is not shown. The blend of the second raffinate and polar solvent are also fractionated into the second raffinate and polar solvent in a fractional distillation column, which is not shown. In the fractional distillation columns which are not shown, the polar solvent that has been separated from the second extract and second raffinate is recovered and reutilized.

[0108] Thus, the first raffinate is separated into the second raffinate and second extract in the second solvent extraction step. The yield of the second raffinate obtained in the second solvent extraction step is preferably 50-90 vol%, more preferably 60-85 vol% and even more preferably 70-85 vol%, based on the first raffinate introduced into the second extraction column 40. The yield of the second extract obtained in the second solvent extraction step is preferably 10-50 vol%, more preferably 15-40 vol% and even more preferably 15-30 vol%.

[0109] For this embodiment, there is no need to use separate extraction columns as the first and second extraction columns, and one extraction column may be used as both the first extraction column 30 and second extraction column 40. In this case, the raffinate obtained in the first solvent extraction step (preferably with the polar solvent removed) may be first placed in storage such as a tank and introduced into a second extraction column 40 in which the extraction conditions have been adjusted to the conditions of the second solvent extraction step, and the

second solvent extraction step then carried out. This will allow excessive equipment investment to be avoided.

[0110] In the second solvent extraction step it is possible to obtain a first raffinate, and a second extract having a 15°C density of 0.94 g/cm³ or greater and a total aromatic content of 30 mass% or greater. If the 15°C density of the second extract is 0.94 g/cm³ or greater, it will be useful as a petroleum-based process oil or its base material in a process for producing rubber such as tire that employs diene rubber, or an extender oil or its base material in a process for producing diene rubber, having a high total aromatic content, a sufficiently low aniline point, a high flash point and a large difference between pour point and glass transition point. In addition, an aromatic compound-containing base oil that is useful as a lubricant base oil, a petroleum-based process oil, an extender oil or its base material, having a total aromatic content of 30 mass% or greater, will be obtainable at high yield from the simultaneously obtained second raffinate.

[0111] The 15°C density of the second extract is preferably 0.94 g/cm³ or greater, more preferably 0.95-1 g/cm³ and even more preferably 0.95-0.98 g/cm³. The total aromatic content is preferably 30 mass% or greater, more preferably 60 mass% or greater and even more preferably 80 mass% or greater, and preferably no greater than 90 mass%. The total aromatic content referred to throughout the present specification is the value measured according to ASTM D2549.

[0112] The second extract has a %C_A value of preferably 15-35, more preferably 20-33 and even more preferably 22-32, as measured according to ASTM D2140.

[0113] The second extract preferably has the following properties.

- Flash point: Preferably 250°C or higher, more preferably 260°C or higher and preferably no higher than 310°C.
- Pour point: Preferably no higher than 30°C and more preferably 10-30°C.
- Aniline point: Preferably no higher than 90°C, more preferably 40-80°C and even more preferably 50-70°C.
- Glass transition point: preferably no higher than -30°C, more preferably no higher than -40°C and preferably -60°C or higher.
- Difference between pour point and glass transition point (pour point - glass transition point): Preferably 45°C or higher, more preferably 50°C or higher and even more preferably 55°C or higher, and preferably no higher than 100°C and more preferably no higher than 80°C.
- Benzo(a)pyrene content: Preferably no greater than 1 ppm by mass.
- Total content of indicated aromatic compounds (PAH): Preferably no greater than 10 ppm by mass.

[0114] Next, the second raffinate was subjected to refining treatment consisting of dewaxing treatment with a dewaxing unit 50 and hydrofinishing treatment by a hydrofinishing unit 60, to obtain a lubricant base oil as refined oil. The aromatic compound-containing base oil (lubricant base oil) obtained in this manner may be used as a base oil (a). The base oil (a) may be a blend of two or more aromatic compound-containing base oils (lubricant base oils) obtained as described above.

[0115] The base oil (a) obtained in this manner has a total aromatic content of preferably 30 mass% or greater and more preferably 30-60

mass%. The base oil (a) is preferably a base oil (a1) of 200-1500N, more preferably 250N or greater and less than 600N, and/or a base oil (a2) of 600-1200N, and even more preferably a base oil (a1) of 300-450N and/or a base oil (a2) of 700-1000N.

5 [0116] A vacuum distillation fraction used to obtain a 500N aromatic compound-containing base oil can potentially contain large amounts of the 8 types of indicated aromatic compounds (PAH) mentioned above, and it will tend to be difficult to simultaneously obtain two or more high flash point aromatic compound-containing base oils with different
10 viscosities. Therefore, a base oil (a1) of 300-450N and/or a base oil (a2) of 700-1000N is preferably used to obtain the aromatic compound-containing base oil.

[0117] The method for producing an aromatic compound-containing base oil according to this embodiment may comprise, after the second
15 solvent extraction step, a base oil preparation step in which either or both the second extract and second raffinate are used to obtain an aromatic compound-containing base oil with a total aromatic content of 30 mass% or greater.

[0118] In the base oil preparation step either or both the second extract
20 and second raffinate are used to obtain an aromatic compound-containing base oil with a total aromatic content of 30 mass% or greater. Also, the second raffinate may be subjected to refining treatment including dewaxing treatment, to obtain a refined oil (dewaxing oil) having a pour point of no higher than -5°C, an aniline point of 90°C or
25 higher, a viscosity index of 90 or greater and a flash point of 250°C or higher, which may be used as the aromatic compound-containing base

oil. The refining treatment preferably includes dewaxing treatment and hydrofinishing. This can easily yield an aromatic compound-containing base oil having a total aromatic content of 30 mass% or greater. The refined oil obtained by the refining treatment can be suitably used as a lubricant base oil or rubber compounding oil, or as a base material therefor.

[0119] When a 200-1500N refined oil, for example, is to be obtained from the second raffinate, a vacuum distillation fraction with the corresponding viscosity may be used as starting material for the first and second solvent extraction steps, to obtain a second extract having a 40°C dynamic viscosity of preferably 200 mm²/s or greater and more preferably 250 mm²/s or greater, and preferably no greater than 5000 mm²/s and even more preferably no greater than 2000 mm²/s.

[0120] In this step, it is possible to obtain an aromatic compound-containing base oil with a total aromatic content of 30 mass% or greater (hereunder referred to as "aromatic compound-containing base oil (a)") from the second raffinate and/or a refined oil product thereof, and/or an aromatic compound-containing base oil with a total aromatic content of 30 mass% or greater (hereunder referred to as "aromatic compound-containing base oil (b)") from the second extract.

[0121] In the base oil preparation step, the appropriate fraction may be separated off from the second raffinate and second extract by appropriate distillation, and then a portion of the second raffinate and a portion of the second extract may be used as base oil (a) and base oil (b), respectively. Alternatively, the second raffinate and second extract obtained by the second solvent extraction step may be directly used as

base oil (a) and base oil (b), respectively, without a base oil preparation step.

[0122] The vacuum distillation fraction used as the starting material is preferably selected as appropriate for the desired base oil (a) and base oil (b) to obtain a base oil (a) and base oil (b) having the desired properties, as co-products. For example, when a lubricant base oil of 300N or greater and less than 600N is to be obtained as base oil (a), a vacuum distillation fraction corresponding to this fraction may be used as starting material, to obtain a lubricant base oil of 300N or greater and less than 600N as the base oil (a) and one with a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s, preferably 250-350 mm²/s and more preferably 250-300 mm²/s as the base oil (b), through the first and second solvent extraction steps and in some cases through a refining step.

[0123] For example, when a lubricant base oil of 600-1200N is to be obtained as base oil (a), a vacuum distillation fraction corresponding to this fraction may be used as starting material, to obtain a lubricant base oil of 600-1200N as the base oil (a) and one with a 40°C dynamic viscosity of 500-5000 mm²/s or preferably 800-2000 mm²/s and more preferably 900-1500 mm²/s as the base oil (b), through the first and second solvent extraction steps and in some cases through refining treatment.

[0124] As mentioned above, base oil (a) of this embodiment may be obtained by subjecting the second raffinate to refining treatment including dewaxing and hydrofinishing. The base oil (a) obtained in this manner (lubricant base oil) has a total aromatic content of 30

mass% or greater and preferably 30-60 mass%. The base oil (a) can be obtained as a lubricant base oil of preferably 200-1500N, more preferably 250N or greater and less than 600N, or 600-1200N, and even more preferably 300-450N or 700-1000N.

5 [0125] From the viewpoint of obtaining a base oil (a) and base oil (b) having reduced contents of the aforementioned 8 indicated aromatic compounds (PAH), having mutually different viscosities and high flash points, the base oil (a) is most preferably 300N-450N or 700-1000N.

10 [0126] If base oil (a) is 600-1200N and preferably 700-1000N, it preferably has the following properties in addition to the specific properties mentioned above.

- 40°C dynamic viscosity: 120-250 mm²/s and preferably 150-200 mm²/s.

15 · 10% Point in gas chromatography distillation: 450-520°C and preferably 460-500°C.

- 90% Point in gas chromatography distillation: 540-600°C and preferably 560-590°C.

- Total aromatic content (ASTM D 2549): 30 mass% or greater, preferably 35-60 mass% and more preferably 40-50 mass%.

20 [0127] According to this embodiment, it is possible to obtain a base oil (a) and base oil (b) having a content of indicated aromatic compounds (PAH) that is below the specified level. The term "indicated aromatic compounds (PAH)" as used herein refers to the 8 types of indicated aromatic compounds (PAH) listed below. The base oil (a) and base oil
25 (b) of this embodiment have a content of the 1) benzo(a)pyrene (BaP) of no greater than 1 ppm by mass, and a total content of the 8 indicated

aromatic compounds (PAH) listed as 1) to 8) below of no greater than 10 ppm by mass.

[0128] 1) Benzo(a)pyrene (BaP)

2) Benzo(e)pyrene (BeP)

5 3) Benzo(a)anthracene (BaA)

4) Chrysene (CHR)

5) Benzo(b)fluoranthene (BbFA)

6) Benzo(j)fluoranthene (BjFA)

7) Benzo(k)fluoranthene (BkFA)

10 8) Dibenzo(a,h)anthracene (DBAhA)

[0129] These indicated aromatic compounds (PAH) can be quantitatively analyzed by GC-MS analysis, after separation and concentration of the components of interest, upon preparing a sample containing an added internal standard substance.

15 [0130] The base oil (a) and base oil (b) are suitable as lubricant base oils, rubber compounding oils, or base materials therefor. The base oil (a) has a sufficiently reduced content of indicated aromatic compounds (PAH), and a flash point of 250°C or higher and a pour point of no higher than -5°C, and it is therefore suitable as a lubricant base oil.

20 Furthermore, because the total aromatic content is 30 mass% or greater and the glass transition point is no higher than -30°C, it can be used as a petroleum-based process oil or extender oil, or as a base material therefor.

[0131] The base oil (b) has a sufficiently reduced content of indicated aromatic compounds (PAH), as well as a flash point of 250°C or higher, a total aromatic content of 30 mass% or greater and a glass transition

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point of no higher than -30°C , it can therefore be used as a petroleum-based process oil or extender oil, or as a base material therefor.

[0132] The base oil (a), the base oil (b) or a blend thereof, may be used for preparation, for example, to a glass transition temperature of between -55 and -30°C and a dynamic viscosity (100°C) of $20\text{--}50\text{ mm}^2/\text{s}$, to obtain a petroleum-based process oil or extender oil particularly suited for compounding with diene rubber. A tire produced by adding such a petroleum-based process oil or extender oil to diene rubber can exhibit both low fuel consumption and grip properties, and can increase the thermal aging resistance or wear resistance.

[0133] As explained above, according to the production method of this embodiment it is possible to obtain a high yield of an aromatic compound-containing base oil having a high flash point, a low glass transition point, and a high total aromatic content while having a sufficiently reduced content of indicated carcinogenic substances. It also has properties suitable as a lubricant base oil, petroleum-based process oil or extender oil, or a base material therefor, while allowing simultaneous production of several types of aromatic compound-containing base oils with different viscosities, and it is therefore particularly useful in industry.

[0134] The base oil (a) preferably has the following properties.

- Pour point: Preferably no higher than -5°C , more preferably no higher than -10°C and preferably -20°C or higher.

- Glass transition point: Preferably no higher than -30°C , more preferably no higher than -40°C and even more preferably no higher

than -50°C, and preferably -60°C or higher, more preferably -100°C or higher, even more preferably -80°C or higher and most preferably -70°C or higher.

· Aniline point: Preferably 70°C or higher, more preferably 90°C or higher and even more preferably 105°C or higher, and preferably no higher than 120°C.

· Viscosity index: Preferably 90 or greater and more preferably 95 or greater, and preferably no greater than 120 and more preferably no greater than 105.

· Flash point: Preferably 250°C or higher and preferably no higher than 310°C.

· Base oil composition according to ASTM D 3238: The %C_P is preferably 60-70, the %C_N is preferably 20-30 and the %C_A is preferably 5-10.

· Benzo(a)pyrene content: Preferably no greater than 1 ppm by mass.

· Total content of indicated aromatic compounds (PAH): Preferably no greater than 10 ppm by mass.

[0135] An aromatic compound-containing base oil of 300N or greater and less than 600N, and preferably 300-450N, as the base oil (a1), preferably has the following properties in addition to those mentioned above.

[0136]

· 40°C Dynamic viscosity: Preferably 60-120 mm²/s, more preferably 65-90 mm²/s and even more preferably 70-80 mm²/s.

· 10% Point in GC distillation: Preferably 400-460°C and more preferably 430-450°C.

- 90% Point in GC distillation: Preferably 500-540°C and more preferably 510-530°C.

- Total aromatic content (ASTM D 2549): Preferably 30 mass% or greater and more preferably 30-50 mass%.

5 The value in gas chromatography distillation, throughout the present specification, is the value measured according to ASTM D2887.

[0137] An aromatic compound-containing base oil of 600-1200N and preferably 700-1000N, as the base oil (a2), preferably has the following properties in addition to those mentioned above.

10 [0138]

- 40°C Dynamic viscosity: Preferably 120-250 mm²/s and more preferably 150-200 mm²/s.

- 10% Point in GC distillation: Preferably 450-520°C and more preferably 460-500°C.

15 · 90% Point in GC distillation: Preferably 540-600°C and more preferably 560-590°C.

- Total aromatic content (ASTM D 2549): Preferably 30 mass% or greater, more preferably 35-60 mass% and even more preferably 40-50 mass%.

20 [0139] The base oil (b) may be an aromatic compound-containing base oil which is the second extract or a refined oil product thereof, obtained by carrying out the first and second solvent extraction steps. The base oil (b) may also be a blend of two or more different types of second extracts obtained as described above or refined oil products thereof.

25 [0140] The base oil (b) is preferably a base oil (b1) having a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s and

a glass transition point of between -60 and -40°C and/or a base oil (b2) having a 40°C dynamic viscosity of 500 mm²/s or greater and a glass transition point of between -50 and -30°C.

[0141] The base oil (b1) preferably has the following properties, in addition to those mentioned above.

- 40°C Dynamic viscosity: Preferably 200 mm²/s or greater and less than 500 mm²/s, more preferably no greater than 400 mm²/s, even more preferably no greater than 350 mm²/s and most preferably no greater than 300 mm²/s.

- Glass transition point: preferably between -60 and -40°C, and more preferably between -55 and -48°C.

- Pour point: Preferably 0-30°C, more preferably 15°C or higher and even more preferably 20°C or higher.

- Difference between pour point and glass transition point (pour point - glass transition point): Preferably 60°C or higher, more preferably 65°C or higher and even more preferably 70°C or higher, and preferably no higher than 100°C and more preferably no higher than 80°C.

- Total aromatic content: 50 mass% or greater, preferably 60 mass% or greater, more preferably 70 mass% or greater and even more preferably 80 mass% or greater, and preferably no greater than 90 mass%.

[0142] The base oil (b2) preferably has the following properties, in addition to those mentioned above.

- 40°C Dynamic viscosity: Preferably 500 mm²/s or greater, more preferably 800 mm²/s or greater and even more preferably 1000 mm²/s or greater, and preferably no greater than 5000 mm²/s, more preferably no greater than 2000 mm²/s and even more preferably no greater than

1500 mm²/s.

· Glass transition point: Preferably between -50 and -30°C, and more preferably between -45 and -35°C.

· Total aromatic content: 50 mass% or greater, preferably 60 mass% or greater, more preferably 70 mass% or greater and even more preferably 80 mass% or greater, and preferably no greater than 90 mass%.

[0143] When a 200-1500N aromatic compound-containing base oil is to be obtained as the base oil (a) by the first and second solvent extraction steps, the vacuum distillation fraction corresponding to that fraction may be used as starting material to simultaneously obtain a product as the base oil (b) having a 40°C dynamic viscosity of preferably 200 mm²/s or greater and preferably 250 mm²/s or greater, and preferably no greater than 5000 mm²/s and more preferably no greater than 2000 mm²/s. The base oil (a) and base oil (b) may be obtained by appropriately separating off the desired fractions by distillation. In this case, there is no particular restriction on the viscosity of the vacuum distillation fraction used as the starting material.

[0144] However, because it is preferred to obtain a co-product of the desired base oil (a) and base oil (b), preferably a vacuum distillation fraction matching the properties of the base oil (a) and base oil (b) is selected and the same vacuum distillation fraction is used as the starting material.

[0145] For example, when an aromatic compound-containing base oil of 250N or greater and less than 600N is to be obtained as the base oil (a1), a vacuum distillation fraction corresponding to that fraction may be used as the starting material. Also, in the first and second solvent

extraction steps, preferably an aromatic compound-containing base oil of 250N or greater and less than 600N is obtained as the base oil (a1), and an extract (aromatic compound-containing base oil) with a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s, preferably 250-350 mm²/s and even more preferably 250-300 mm²/s, is obtained as the base oil (b1).

[0146] Also, for example, when a lubricant base oil of 600-1200N is to be obtained as the base oil (a2), a vacuum distillation fraction corresponding to that fraction may be used as the starting material.

Also, in the first and second solvent extraction steps, preferably an aromatic compound-containing base oil of 600-1200N is obtained as the base oil (a2), and an extract (aromatic compound-containing base oil) with a 40°C dynamic viscosity of 500-5000 mm²/s, preferably 800-2000 mm²/s and even more preferably 900-1500 mm²/s, is obtained as the base oil (b2). Batch treatment is preferably carried out in this method to obtain the desired base oil (a) and base oil (b).

[0147] In the blending step, a rubber compounding oil is prepared by blending the base oil (a) and base oil (b) obtained as described above in a prescribed proportion. For example, they may be combined in a proportion of no greater than 95 mass% of the base oil (a) (excluding 0%) and 5 mass% or greater of the base oil (b), based on the total mass of the rubber compounding oil. It will thus be possible to obtain a rubber compounding oil comprising the base oil (a) and the base oil (b).

[0148] The rubber compounding oil may also be prepared with the base oil (b) alone, without the blending step. However, from the viewpoint of obtaining a rubber compounding oil with a suitable dynamic viscosity

(a 100°C dynamic viscosity of 10-70 mm²/s, preferably 15-50 mm²/s and more preferably 20-32 mm²/s), and of obtaining a rubber compounding oil with a difference of at least 50°C between the pour point and glass transition point, the mixing proportion of the base oil (a) is preferably 10-50 mass% and more preferably 20-40 mass%, based on the total rubber compounding oil. From the same viewpoint, the mixing proportion of the base oil (b) is preferably 90-50 mass% and more preferably 80-60 mass%.

[0149] From the viewpoint of obtaining a rubber compounding oil with a difference of at least 50°C between the pour point and glass transition point and a glass transition point of no higher than -50°C, the base oil (a) preferably comprises a base oil (a2), and the base oil (b) preferably comprises a base oil (b1), for a particularly large difference between the pour point and glass transition point, such as 60°C or more, for example.

In order to further increase the yield of the rubber compounding oil having a suitable dynamic viscosity as explained above, the base oil (b) preferably comprises a base oil (b1) and a base oil (b2). From this viewpoint, the content ratios of the base oil (a) or base oil (a2), the base oil (b1) and the base oil (b2) are preferably 10-40 mass%, 5-35 mass% and 85-25 mass%, and more preferably 20-30 mass%, 20-30 mass% and 60-40 mass%, respectively.

[0150] The rubber compounding oil of this embodiment may contain a base material other than the base oil (a) and base oil (b), so long as the effect of the invention is not impeded.

[0151] The embodiments described above are only preferred embodiments of the invention, and the invention is in no way limited

thereto.

Examples

[0152] The present invention will now be explained in greater detail by the following examples and reference examples. However, the present invention is not limited to the examples described below.

[0153] (Example 1)

Atmospheric distillation residue oil from crude oil was subjected to vacuum distillation using a common vacuum distillation unit, and the fuel-corresponding fraction, the fraction up to the 150N-corresponding fraction, the 350N-corresponding fraction and the 900N-corresponding fraction were separated off. The separated 350N-corresponding fraction was treated with a polar solvent extraction unit as shown in Fig. 1. Specifically, a first solvent extraction step was carried out in which the 350N-corresponding fraction was introduced into a first extraction column 30 having a lower bottom temperature than the top temperature, and a polar solvent (furfural) was contacted with the 350N-corresponding fraction. By this first solvent extraction step, the 350N-corresponding fraction was separated into a mixture of a first raffinate and the polar solvent, obtained from the top part of the first extraction column 30, and a mixture of a first extract and the polar solvent, obtained from the bottom part. Next, the polar solvent was recovered by a fractional distillation column (not shown), to obtain the first raffinate and first extract from the mixtures.

[0154] This was followed by a second solvent extraction step in which the first raffinate was introduced into a second extraction column 40 having a lower bottom temperature than the top temperature, and

contacted with a polar solvent (furfural). By this second solvent extraction step, the first raffinate was separated into a mixture of a second raffinate and the polar solvent, obtained from the top part of the column, and a mixture of a second extract and the polar solvent, obtained from the bottom part. Next, the polar solvent was recovered by a fractional distillation column (not shown), to obtain the second raffinate and second extract from the mixtures.

[0155] The second raffinate had a total aromatic content of 30 mass% or greater as measured according to ASTM D2549. The second extract also had a 15°C density of 0.94 g/cm³ or greater, and a total aromatic content of 30 mass% or greater as measured according to ASTM D2549.

[0156] Table 1 shows the production conditions and yields for the first solvent extraction step and second solvent extraction step.

[0157]

[Table 1]

Production step		First solvent extraction step	Second solvent extraction step
Starting material		Vacuum distillation residue (350N)	First raffinate (of previous column)
Polar solvent		Furfural	Furfural
Solvent ratio		1.3	2.0
Column top temperature	°C	90	116
Column bottom temperature	°C	60	85
Raffinate		First raffinate	Second raffinate
Yield (with respect to starting material)	vol%	74	80
Total content of 8 PAH types	ppm by mass.	<10	<10
Extract		First extract	Second extract
Yield (with respect to starting material)	vol%	26	20
Total content of 8 PAH types	ppm by mass.	>10	<10
Yield of useful components	vol%	-	74

[0158] The second raffinate was subjected to refining treatment by MEK dewaxing and hydrofinishing treatment to a pour point of no higher than -10°C, to obtain an aromatic compound-containing base oil (lubricant base oil) with a total aromatic content of 30 mass% or greater.

5 This was used as a base oil (A1). The second extract was also used as base oil (B1) and (B3) components. The properties of the base oil (A1), base oil (B1) and base oil (B3) are shown in Table 2. The second extract was not subjected to refining treatment.

[0159]

10 [Table 2]

		Base oil (A1)	Base oil (B1)	Base oil (B3)
Density (15°C)	g/cm ³	0.8827	0.9539	0.9578
Flash point (COC)	°C	272	263	258
Dynamic viscosity (40°C)	mm ² /s	74.34	236.8	274.8
Dynamic viscosity (100°C)	mm ² /s	9.227	14.81	14.81
Viscosity index		99	37	11
Aniline point	°C	107	61.7	59
Nitrogen content	mass%	0.004	0.040	0.040
Composition (ASTM D3238)				
%C _P		67.6	56.6	54.4
%C _N		26.9	19.6	19.7
%C _A		5.6	23.8	25.9
Composition (ASTM D2140) %C _A		-	22.9	24.2
Composition (ASTM D2549)				
Saturated content	mass%	65.5	15.4	15.4
Total aromatic content	mass%	33.9	82.3	82.3
Benzo(a)pyrene	ppm by mass.	<1	<1	<1
Total content of 8 PAH types	ppm by mass.	<10	<10	<10
Gas chromatography column				
10% point	°C	444	-	-
90% point	°C	518	-	-
Pour point	°C	-12.5	25	25
Glass transition temp.	°C	(Predicted: <-60)	-53.9	-52.4
Pour point - Glass transition temp.	°C	(Predicted: >47.5)	78.9	-

15 [0160] The total content of benzo(a)pyrene and 8 indicated aromatic

compounds (PAH) were measured in the following manner. First, 1 g of each aromatic compound-containing base oil was dissolved in hexane in a 50 ml flask to prepare a 2 mass% sample solution. The sample solution was loaded into 5 mass% hydrous silica gel and rinsed with hexane, and then the target component was eluted with a 1 vol% acetone/hexane solution. After concentrating the eluate, an internal standard substance was added to prepare a sample, and identification and quantitation were performed using a common gas chromatography mass spectrometer (GC-MS).

[0161] (Example 2)

Atmospheric distillation residue oil from crude oil was subjected to vacuum distillation using a vacuum distillation unit, and the fuel-corresponding fraction, the fraction up to the 150N-corresponding fraction, the 350N-corresponding fraction and the 900N-corresponding fraction were separated off. A first and second raffinate, as well as a first and second extract, were produced in the same manner as Example 1, except that the 900N-corresponding fraction was introduced into the first extraction column 30 instead of the 350N-corresponding fraction, and the production conditions for the first and second solvent extraction step were changed as shown in Table 3. The production was carried out several times under the same conditions to obtain multiple lots of products. Table 3 shows the production conditions and yields for the first solvent extraction step and second solvent extraction step.

[0162] The second raffinate of Example 2 also had a total aromatic content of 30 mass% or greater as measured according to ASTM D2549. The second extract had a 15°C density of 0.94 g/cm³ or greater, and a

total aromatic content of 30 mass% or greater as measured according to ASTM D2549.

[0163]

[Table 3]

5

Production step		First solvent extraction step	Second solvent extraction step
Starting material		Vacuum distillation residue (900N)	First raffinate (of previous column)
Polar solvent		Furfural	Furfural
Solvent ratio		1.2	2.1
Column top temperature	°C	90	121
Column bottom temperature	°C	60	90
Raffinate		First raffinate	Second raffinate
Yield (with respect to starting material)	vol%	75	75
Total content of 8 PAH types	ppm by mass.	<10	<10
Extract		First extract	Second extract
Yield (with respect to starting material)	vol%	25	25
Total content of 8 PAH types	ppm by mass.	>10	<10
Yield of useful components	vol%	-	75

[0164] Two different second raffinates from different production lots were subjected to refining treatment by MEK dewaxing and hydrofinishing treatment to a pour point of no higher than -10°C, to obtain two different aromatic compound-containing base oils (lubricant base oils) each with a total aromatic content of 30 mass% or greater. These were used as base oil (A2) and base oil (A3). Also, the second extract obtained in the second solvent extraction step together with the second raffinate used for production of base oil (A2) was used as base oil (B2). In addition, the second extract obtained in the second solvent extraction step together with the second raffinate used for production of base oil (A3) was used as base oil (B4). The properties of the base oil (A2), base oil (A3), base oil (B2) and base oil (B4) are shown in Table 4.

10

15

[0165]

[Table 4]

		Base oil (A2)	Base oil (A3)	Base oil (B2)	Base oil (B4)
Density (15°C)	g/cm ³	0.893	0.8936	0.9768	0.9774
Flash point (COC)	°C	282	298	296	280
Dynamic viscosity (40°C)	mm ² /s	164	170.7	1090	1185
Dynamic viscosity (100°C)	mm ² /s	15.48	15.76	33.77	34.09
Viscosity index		95	94	23	14
Aniline point	°C	112.4	112	59.7	60
Nitrogen content	mass%	0.007	0.009	0.080	0.090
Composition (ASTM D3238)					
%C _P		68.1	68.0	57.5	57.5
%C _N		24.9	25.0	9.7	9.7
%C _A		7.0	7.0	32.8	32.8
Composition (ASTM D2140) %C _A		-	-	29.5	29.5
Composition (ASTM D2549)					
Saturated content	mass%	55.9	55.9	9.0	9.0
Total aromatic content	mass%	43.3	43.3	86.9	86.9
Benzo(a)pyrene	ppm by mass.	<1	<1	<1	<1
Total content of 8 PAH types	ppm by mass.	<10	<10	<10	<10
Gas chromatography column					
10% point	°C	481	481	-	-
90% point	°C	573	573	-	-
Pour point	°C	-15	-12.5	17.5	20
Glass transition temp.	°C	-63	-63	-41.9	-42.7
Pour point - Glass transition temp.	°C	48	-	59.4	-

5 [0166] As shown in Table 1 and Table 3, in Examples 1 and 2, the total yields of the second raffinate and second extract, which are useful as rubber compounding oils or their base materials, were 74-75 vol%, based on the stock oil of the first extraction column (useful component yield). It was confirmed that it is possible, in this manner, to

10 accomplish high-yield production of a lubricant base oil (aromatic compound-containing base oil) useful as a rubber compounding oil or its base material.

[0167] As shown in Table 2 and Table 4, the base oils (A1), (A2), (A3), (B1), (B2), (B3) and (B4) of Examples 1 and 2 all had total aromatic

contents of 30 mass% or greater and flash points of 250°C or higher. That is, it was possible in these examples to produce multiple aromatic compound-containing base oils with excellent properties and different viscosities by using a plurality of vacuum distillation fractions with different viscosities.

[0168] Furthermore, the base oils (A1), (A2), (A3), (B1), (B2), (B3) and (B4) all had sufficiently reduced total contents of the carcinogenic compounds benzo(a)pyrene and 8 indicated aromatic compounds (PAH).

[0169] In addition, base oil (B1) and base oil (B2) had a difference of at least 50°C between the pour point and glass transition point, with base oil (B1) in particular having a unique property with a difference of at least 70°C. In other words, it was confirmed that the base oil (B1) and base oil (B2) both had low glass transition points, despite their high pour points.

[0170] Furthermore, since base oil (B1), base oil (B2), base oil (B3) and base oil (B4) of Examples 1 and 2 each had a high aromatic hydrocarbon content (C_A) of 20-35 mass% and a suitable glass transition temperature T_g , their addition to rubber compositions can improve tensile strength and wear resistance.

[0171] (Example 3)

The base oils (A1), (A2), (B1) and (B2) were compounded in the amounts listed in Table 5 to prepare rubber compounding oils for Examples 3-1 to 3-4. The properties of the rubber compounding oils are summarized in Table 5.

[0172]

[Table 5]

		Example 3-1	Example 3-2	Example 3-3	Example 3-4
Base oil (A1)		-	-	20	-
Base oil (A2)		25	10	-	33
Base oil (B1)		25	40	-	-
Base oil (B2)		50	50	80	67
Density (15°C)	g/cm ³	0.9501	0.9596	0.9585	0.95
Flash point (COC)	°C	272	276	276	294
Dynamic viscosity (40°C)	mm ² /s	415.8	468.0	544.1	501.0
Dynamic viscosity (100°C)	mm ² /s	21.89	22.27	24.97	24.83
Aniline point	°C	75.8	66.1	71.2	78.7
Nitrogen content	mass%	0.05	0.05	0.06	0.06
Composition (ASTM D3238)					
%C _P		60.3	59.1	60.1	61.2
%C _N		16.5	14.4	14.1	15.8
%C _A		23.2	26.6	25.8	23.0
Composition (ASTM D2549)					
Saturated content	mass%	22.3	16.2	22.1	23.1
Total aromatic content	mass%	74.9	80.7	73.5	73.9
Composition (ASTM D2007)					
Saturated content	mass%	25.3	-	-	-
Total aromatic content	mass%	68.9	-	-	-
Polar compound	mass%	5.8	-	-	-
Benzo(a)pyrene	ppm by mass.	<1	<1	<1	<1
Total content of 8 PAH types	ppm by mass.	<10	<10	<10	<10
Pour point	°C	15.0	20.0	-5.0	-2.5
Glass transition temp.	°C	-51.0	-49.3	-50.5	-51.5
Pour point - Glass transition temp.	°C	66.0	69.3	45.5	49.0

[0173] As seen in Table 5, the rubber compounding oils of Examples 3-

5 1, 3-2, 3-3 and 3-4 all had high total aromatic contents and high flash

points, and sufficiently reduced contents of carcinogenic hazardous

substances. The difference between the pour point and the glass

transition point was at least 45°C. In particular, the rubber

compounding oils of Example 3-1 and Example 3-2 had unique

10 properties, with a difference of at least 60°C between the pour point and

the glass transition point, a total aromatic content of 50 mass% or

greater according to ASTM D2549 or ASTM D2007 and a pour point of

15°C or higher, and yet a glass transition point of no higher than -45°C.

[0174] The rubber compounding oils of Examples 3-1 to 3-4 were all petroleum-based process oils with a %C_A of 20-35 according to ASTM D3238, a glass transition temperature T_g of between -55 and -30°C, a
5 dynamic viscosity (100°C) of 20-50 mm²/s, and containing substantially no indicated aromatic compounds (PAH), and therefore when they are added as extender oils to be used in a production step for diene rubber or process oils to be used in processing of diene rubber, it is possible to achieve both low fuel consumption and grip properties, and to exhibit an
10 effect of improving thermal aging resistance and wear resistance.

[0175] (Reference Example 1)

Atmospheric distillation residue oil from crude oil was subjected to vacuum distillation using a common vacuum distillation unit, and the fuel-corresponding fraction, the fraction up to the 150N-corresponding
15 fraction, the 250N-corresponding fraction and the higher fraction (500N-corresponding fraction) were separated off. The separated 500N-corresponding fraction was introduced into an extraction column having a lower bottom temperature than the top temperature, and a solvent extraction step by contact with a polar solvent (furfural) was
20 conducted under conditions such that the raffinate yield was increased and the obtained lubricant base oil had an aromatic content of 30 mass% or greater and an indicated aromatic compound (PAH) content of less than 10 ppm by mass. In this solvent extraction step, the 500N-corresponding fraction was separated into a first raffinate and a first
25 extract. Table 6 shows the production conditions and yield for the solvent extraction step.

[0176]

[Table 6]

Production step		Solvent extraction step
Starting material		Vacuum distillation residue (500N)
Polar solvent		Furfural
Solvent ratio		2
Column top temperature	°C	120
Column bottom temperature	°C	72
Raffinate		First raffinate
Yield (with respect to starting material)	vol%	60
Total content of 8 PAH types	ppm by mass.	<10
Extract		First extract
Yield (with respect to starting material)	vol%	40
Total content of 8 PAH types	ppm by mass.	>18
Yield of useful components	vol%	60

5

[0177] The first raffinate obtained in this manner was subjected to refining treatment by MEK dewaxing and hydrofinishing treatment to a pour point of no higher than -10°C. This yielded 2 types of aromatic compound-containing base oils (lubricant base oils) from different production lots having total aromatic contents of 30 mass% or greater. These were used as base oil (E1) and base oil (E2). The extract was also used as base oil (F). The properties of the base oil (E1), base oil (E2) and base oil (F) are shown in Table 7.

10

[0178]

[Table 7]

Aromatic compound-containing base oil		Base oil (E1)	Base oil (E2)	Base oil (F)
Density (15°C)	g/cm ³	0.8867	0.8867	1.01
Flash point (COC)	°C	270	270	-
Dynamic viscosity (40°C)	mm ² /s	100.4	100.4	-
Dynamic viscosity (100°C)	mm ² /s	11.18	11.18	30.7
Viscosity index		96	96	-
Aniline point	°C	109	109	39
Nitrogen content	mass%	0.004	0.004	-
Composition (ASTM D3238)				
%C _P		66.1	66.1	-
%C _N		27.1	27.1	-
%C _A		6.8	6.8	-
Composition (ASTM D2140) %C _A		-	-	-
Composition (ASTM D2549)				
Saturated content	mass%	62.2	62.2	-
Total aromatic content	mass%	37.3	37.3	-
Benzo(a)pyrene	ppm by mass.	<1	<1	<2
Total content of 8 PAH types	ppm by mass.	<10	<10	>18
Gas chromatography column				
10% point	°C	458	458	-
90% point	°C	537	538	-
Pour point	°C	-12.5	-12.5	-

[0179] As seen in Table 6, in this reference example the first raffinate yield was 60 vol% and the first extract yield was 40 vol%, based on the starting material introduced into the extraction column. Also, as seen in Table 7, the total content of carcinogenic indicated aromatic compounds (PAH) in the aromatic compound-containing base oil (F) exceeded 10 ppm by mass. The aromatic compound-containing base oil (F) is not suitable for direct use as a rubber compounding oil, and even when combined with another lubricant base oil, its blending ratio cannot be over 50 mass% for most purposes.

[0180] (Reference Example 2)

Deasphalted oil of vacuum distillation residue oil of atmospheric distillation residue oil from crude oil was separated into a raffinate and

an extract by the same solvent extraction step as Reference Example 1. The extract had a 100°C dynamic viscosity of 95 mm²/s, a total aromatic content of 69 mass% according to ASTM D2549, a pour point of 12.5°C, a glass transition point of -29.7°C and a difference of 42.2°C between the pour point and glass transition point (pour point - glass transition point).

[0181] This extract was combined with base oil (E1) of Reference Example 1 in a mass ratio of 80:20 to prepare a rubber compounding oil. The pour point of the rubber compounding oil was 0°C, the glass transition point was -44.5°C, and the difference between the pour point and glass transition point was 44.5°C.

[0182] (Reference Example 3)

The solvent extraction step was carried out in the same manner as Reference Example 1, except that the 250N-corresponding fraction was introduced into the extraction column instead of the 500N-corresponding fraction, and the raffinate and extract were obtained. The raffinate was then subjected to refining treatment by MEK dewaxing and hydrofinishing treatment to a pour point of no higher than -10°C, to obtain an aromatic compound-containing base oil. This was used as a base oil (G). The base oil (g) had a flash point of below 250°C. In other words, in this reference example it was only possible to obtain a single aromatic compound-containing base oil (500N base oil) with a flash point of 250°C or higher from the total vacuum distillation fraction (250N-corresponding fraction).

Industrial Applicability

[0183] According to the invention it is possible to provide a rubber

compounding oil having a high flash point and a low glass transition point while maintaining a high total aromatic content, and having sufficient reduction in the content of indicated polycyclic aromatic compounds, as well as a method for producing the rubber compounding oil. According to the invention it is also possible to provide an aromatic compound-containing base oil with a high flash point and a low glass transition point, as well as a high total aromatic content and sufficient reduction in the content of carcinogenic substances, from a raffinate and extract obtained by a polar solvent extraction process using a vacuum distillation fraction as starting material. It is also possible to provide a method for producing an aromatic compound-containing base oil, which allows high-yield production of such an aromatic compound-containing base oil.

Explanation of Symbols

[0184] 30: First extraction column, 40: second extraction column, 50: dewaxing unit, 60: hydrofinishing unit.

CLAIMS

1. A rubber compounding oil comprising

a component (a) that consists of an aromatic compound-containing base oil containing a raffinate obtained by separating a vacuum distillation fraction of atmospheric distillation residue oil from crude oil by a polar solvent extraction step, or a refined oil product thereof, and having a %C_A of 3-20 according to ASTM D 3238 and a pour point of no higher than -10°C; and

a component (b) that consists of an aromatic compound-containing base oil containing an extract obtained by separating the vacuum distillation fraction of atmospheric distillation residue oil by a polar solvent extraction step, or a refined oil product thereof, and having a %C_A of 25-45 according to ASTM D 3238 and a 15°C density of 0.94 g/cm³ or greater, and

wherein the content of the component (a) in the rubber compounding oil is no greater than 95 mass% and the content of the component (b) in the rubber compounding oil is 5 mass% or greater, and

wherein the rubber compounding oil has

a total aromatic content of 50 mass% or greater according to ASTM D 2007 or ASTM D 2549, a flash point of 250°C or higher, a difference of at least 45°C between the pour point and glass transition point,

a benzo(a)pyrene content of no greater than 1 ppm by mass; and

a total content of the following indicated aromatic compounds 1) to 8) of no greater than 10 ppm by mass.

- 1) Benzo(a)pyrene (BaP)
 - 2) Benzo(e)pyrene (BeP)
 - 3) Benzo(a)anthracene (BaA)
 - 4) Chrysene (CHR)
 - 5 5) Benzo(b)fluoranthene (BbFA)
 - 6) Benzo(j)fluoranthene (BjFA)
 - 7) Benzo(k)fluoranthene (BkFA)
 - 8) Dibenzo(a,h)anthracene (DBAhA)
- 10 2. The rubber compounding oil according to claim 1,
wherein the aromatic compound-containing base oil of the
component (a) has a 40°C dynamic viscosity of 60-600 mm²/s, an
aniline point of 70°C or higher, a 10% point of 400-500°C and a 90%
point of 500-600°C in GC distillation and a glass transition point of no
15 higher than -30°C, and
wherein the aromatic compound-containing base oil of the
component (b) has a 40°C dynamic viscosity of 200 mm²/s or greater,
an aniline point of no higher than 90°C, and a total aromatic content of
30 mass% or greater according to ASTM D 2549.
- 20 3. The rubber compounding oil according to claim 1 or 2, wherein
the polar solvent extraction step includes;
a first polar solvent extraction step in which the vacuum
distillation fraction is contacted with a polar solvent in a first extraction
25 column having a bottom temperature of 50-70°C and a top temperature
of 80-100°C, to obtain a first raffinate and a first extract, and

a second polar solvent extraction step in which the first raffinate is contacted with a polar solvent in a second extraction column having a bottom temperature and top temperature that are each at least 10°C higher than those of the first extraction column, to obtain a second raffinate and a second extract,

wherein the component (b) is the second extract or a refined oil product thereof.

4. The rubber compounding oil according to any one of claims 1 to 3, wherein the component (b) comprises

an aromatic compound-containing base oil (b1) having a 40°C dynamic viscosity of 200 mm²/s or greater and less than 500 mm²/s and a glass transition point of between -60 and -40°C, and

an aromatic compound-containing base oil (b2) having a 40°C dynamic viscosity of 500 mm²/s or greater and a glass transition point of between -50 and -30°C.

5. The rubber compounding oil according to any one of claims 1 to 4, wherein the component (a) has a 40°C dynamic viscosity of 120-250 mm²/s.

6. A method for producing a rubber compounding oil, which comprises a blending step of blending;

a component (a) that consists of an aromatic compound-containing base oil containing a raffinate obtained by separating a vacuum distillation fraction of atmospheric distillation residue oil from

crude oil by a polar solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 60-600 mm²/s, an aniline point of 70°C or higher, a 10% point of 400-500°C and a 90% point of 500-600°C in GC distillation, a %C_A of 3-20 according to ASTM D 3238 ,a pour point of no higher than -10°C and a glass transition point of no higher than -30°C, and

a component (b) that consists of an aromatic compound-containing base oil containing an extract obtained by separating the vacuum distillation fraction of atmospheric distillation residue oil by a polar solvent extraction step, or a refined oil product thereof, and having a 40°C dynamic viscosity of 200 mm²/s or greater, an aniline point of no higher than 90°C, a %C_A of 25-45 according to ASTM D 3238, a 15°C density of 0.94 g/cm³ or greater, and a total aromatic content of 30 mass% or greater according to ASTM D 2549,

wherein the rubber compounding oil has the total aromatic content of 50 mass% or greater according to ASTM D 2007 or ASTM D 2549, the flash point of 250°C or higher, the difference between the pour point and the glass transition point of at least 45°C, the benzo(a)pyrene content of no greater than 1 ppm by mass and the total content of the following indicated aromatic compounds 1) to 8) of no greater than 10 ppm by mass, and

wherein the content of the component (a) in the rubber compounding oil is greater than 0 and no greater than 95 mass% and the content of the component (b) in the rubber compounding oil is 5 mass% or greater and less than 100 mass%.

1) Benzo(a)pyrene (BaP)

- 2) Benzo(e)pyrene (BeP)
- 3) Benzo(a)anthracene (BaA)
- 4) Chrysene (CHR)
- 5) Benzo(b)fluoranthene (BbFA)
- 5 6) Benzo(j)fluoranthene (BjFA)
- 7) Benzo(k)fluoranthene (BkFA)
- 8) Dibenzo(a,h)anthracene (DBAhA)