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(54) **Treated labdanum oil, process for preparing the same, novel ketone compound, and perfume composition containing the same.**

(57) A process of preparing a treated labdanum oil is disclosed, which comprises: (a) a step of mixing a labdanum raw material with an aqueous alkali solution and separating the aqueous layer; (b) a step of adding an acidic substance to the aqueous layer separated in step (a) and separating the thus released acidic components of labdanum; and (c) a step of heating the acidic components obtained in step (b) at a temperature of from 200 to 350 °C for a period of from 0.1 to 10 hours. A treated labdanum oil prepared in this process and a perfume composition containing the same are also disclosed. A novel ketone compound of formula (I):

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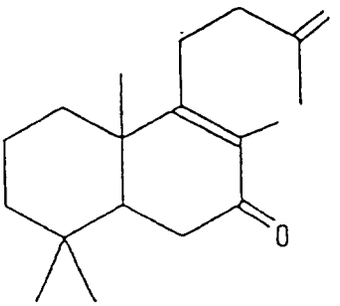

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and a perfume composition containing the ketone compound are further disclosed.

FIELD OF THE INVENTION

This invention relates to a treated labdanum oil with an improved odor which is useful as a material of a perfume composition and a process for preparing the same. It also relates to a novel ketone compound having an animal and amber-like fragrance and a perfume composition containing the same.

BACKGROUND OF THE INVENTION

Labdanum is a black and highly viscous liquid obtained from plants belonging to the genus Cistus. Labdanum, though of plant origin, has an animal and amber-like fragrance and is widely used as a natural amber material in various perfume compositions (see S. Arctander, Perfume and Flavor Chemicals and Osamu Okuda, KORYO KAGAKU SORAN). Labdanum in general use includes labdanum gum obtained by boiling branches and leaves of Cistus Ladaniferus L. in water and then collecting a resin separated therefrom, labdanum gum extract obtained by removing inorganic solids from the labdanum gum, and labdanum gum distillate obtained by distillation or fractional distillation of the labdanum gum extract.

However, these conventional labdanum gum, labdanum gum extract and labdanum gum distillate not only give off an undesired crude woody note like cedarwood oil as well as an amber-like fragrance but assume a black to brown black color. Therefore, where the labdanum product is used as such as a material of perfume compositions for soaps, detergents, etc. to give its characteristic amber animal fragrance, it deteriorates the smell of the scented products due to its rough, woody odor and also colors the products.

While considerable study has been given to the fragrance components present in labdanum, the animal and amber-like fragrance characteristic component of labdanum has not yet been elucidated.

SUMMARY OF THE INVENTION

An object of the present invention is to provide a treated labdanum oil which is free from the crude odor like cedarwood oil possessed by the conventional labdanum gum, labdanum gum extract and labdanum gum distillate, which has an animal and amber-like fragrance with an enhanced animal note, and which has a reduced color and a reduced viscosity.

Another object of the present invention is to provide a process for preparing the above-mentioned treated labdanum oil.

A further object of the present invention is to provide a perfume composition containing the above-mentioned treated labdanum oil.

A still further object of the present invention is to provide a novel perfume component, in particular, a compound having an animal and ambler-like fragrance and useful as a material for perfume compounding.

The present inventors have conducted extensive investigations on the components of labdanum. As a result, they have found that heat treatment of acid components contained in labdanum, such as carboxylic acids and phenols, under atmospheric pressure provides a perfume material having an enhanced animal fragrance and free from the unpleasant woody note and that the resulting heat-treated product can further be purified by distillation under specific conditions to provide a distillate with a reduced color and a reduced viscosity and containing a highly concentrated perfume component, which would be broadly applied to perfume compounding.

As a result of further investigation, the inventors have also found that a ketone compound represented by formula (I) shown below, which is separated from labdanum, has an animal and amber-like fragrance and is useful as a perfume component for various perfume compositions. The present invention has been completed based on these findings.

The present invention provides a process for preparing a treated labdanum oil which comprises:

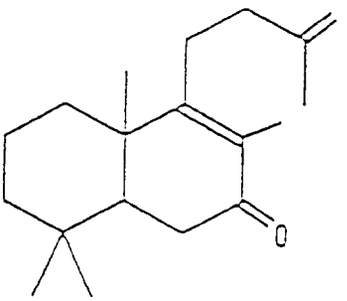
(A) a step of mixing a labdanum raw material with an aqueous alkali solution and separating the aqueous layer;

(B) a step of adding an acidic substance to the aqueous layer separated in step (A) and separating the thus released acidic components of labdanum; and

(C) a step of heating the acidic components obtained in step (B) at a temperature of from 200 to 350 °C for a period of from 0.1 to 10 hours.

The present invention further provides a treated labdanum oil obtainable by the above-mentioned process and a perfume composition containing the thus prepared treated labdanum oil.

The present invention furthermore provides 4-(3-methyl-3-butenyl)-4α,5,6,7,8,8α-hexahydro-3,4α,8,8-tetramethyl-2(1H)-naphthalenone represented by formula (I):



(I)

and a perfume composition containing the same.

DETAILED DESCRIPTION OF THE INVENTION

In the process for preparing the treated labdanum oil according to the present invention, step (C) may preferably be followed by step (D) in which the heat-treated acidic components obtained in step (C) are subjected to further purified by distillation, since step (D) causes further reduction in the color and viscosity, and concentration of perfume components of the resulting product.

The labdanum raw material which can be used in the present invention is not particularly limited but preferably includes (1) labdanum gum, which is a resin separated on boiling of a labdanum-containing plant in water, (2) labdanum gum extract obtained by removing inorganic solids from the labdanum gum, and (3) labdanum gum distillate obtained by distillation or fractional distillation of the labdanum gum extract. These raw materials may be used either individually or in combination of two or more thereof.

The labdanum gum is a resin separated from Cistus ladaniferus L. in boiling water, usually in an aqueous alkali solution. Commercially available labdanum gum products may be used as such. The labdanum gum extract is obtained by dissolving labdanum gum in an organic solvent, such as ethanol, and removing the insoluble inorganic solid content. Commercially available labdanum gum extracts may be used as such. The labdanum gum distillate is obtained by subjecting the labdanum gum extract to distillation or fractional distillation under conditions of, for example, a column inner temperature of 140 to 240 °C and under a pressure of 2 Torr. All of these labdanum raw materials have an unpleasant cedarwood-like woody odor.

Each step is explained below in detail.

Step (a) is a step for extracting acidic components present in a labdanum raw material with an alkali. The extraction is carried out through ordinary neutralization with an alkali. For example, an aqueous alkali solution is added to the labdanum raw material, followed by mixing with stirring.

The alkali which can be used in step (a) is not particularly limited as long as it is capable of neutralizing the acidic components of labdanum, such as carboxylic acids and phenols, to form water-soluble neutral salts. From the economical standpoint, alkali metal hydroxides, alkaline earth metal hydroxides, alkali metal carbonates, and alkali metal hydrogencarbonates are preferred, with alkali metal hydroxides, such as sodium hydroxide and potassium hydroxide being more preferred.

The alkali is used in an amount of from 0.5 to 10 times equivalents, and preferably from 1.0 to 5.0 times equivalents, based on the acid equivalent of the labdanum raw material. The acid equivalent of the labdanum raw material can be determined based on the acid value of the labdanum raw material.

While not limiting, the mixing with stirring is preferably carried out in the presence of an organic solvent at a temperature of from 0 to 150 °C, and preferably from 50 to 100 °C, for a period of from 0.1 to 5 hours, and preferably from 0.5 to 2 hours. The organic solvent to be used is not particularly limited as long as it is stable under an alkaline condition. Examples of suitable organic solvents include hexane, heptane, cyclohexane, benzene, toluene, xylene, and mixtures thereof.

After the alkali extraction, the resulting reaction mixture is subjected to phase separation in a conventional manner, whereby the organic phase is removed to separate the aqueous phase.

Step (B) is a step for subjecting the salts of the acidic components of the labdanum raw material to metathesis with an acid and recovering the thus released acidic components. The operation is carried out by adding an acid to the aqueous alkali solution of the salts, followed by stirring to make the solution acidic, and isolating the thus released acidic components of labdanum.

The acid which can be used in step (b) is not particularly limited as long as it is capable of metathesizing the alkali and the like salt of a carboxylic acid, a phenol, etc., which are the acidic components of labdanum, to release the corresponding acid. From the economical viewpoint, sulfuric acid, hydrochloric acid or phosphoric acid is preferred. The acid is used in an amount of from 1.0 to 5 times, preferably from 1.2 to 3 times, the equivalents of the alkali used.

While conditions for the above mixing and stirring are not restricted, it is preferable, taking heat generation of neutralization into consideration, that the acid is dropwise added to the salt in the presence of an organic solvent at a temperature of from 0 to 50 °C, and particularly from 20 to 40 °C, over a period of from 0.1 to 5 hours, and particularly from 0.3 to 1 hour, and the mixture is then stirred at a temperature of from 0 to 100 °C, and particularly from 50 to 80 °C, for a period of from 0.1 to 5 hours, and particularly from 0.5 to 2 hours. The organic solvent to be used is not particularly limited as long as it is stable under an acidic condition. Examples of suitable organic solvents are hexane, heptane, cyclohexane, benzene, toluene, xylene, and mixtures thereof.

After the re-extraction with an acid, the excess acid can be removed by separating the reaction mixture into an aqueous layer and an organic layer in a usual manner. If desired, the organic layer is washed with water and subjected to distillation in a conventional manner.

Step (C) is a step for heat-treating the acidic components of labdanum recovered in step (B).

The heat treatment is carried out at a temperature of from 200 to 350 °C, and preferably from 250 to 300 °C, for a period of from 0.1 to 10 hours, and preferably from 0.5 to 2 hours while stirring under atmospheric pressure, preferably in a nitrogen atmosphere to avoid generation of an offensive odor or coloring due to oxidation.

The resulting acidic components of labdanum is free from the rough, cedarwood-like unpleasant odor and has an enhanced animal note.

For the purpose of further discoloration, viscosity reduction, and concentration of perfume components, the acidic components obtained in step (C) is preferably subjected to distillation as step (D).

Namely, step (D) is a distillation purification step for discoloration, viscosity reduction and concentration of the heat-treated acidic components of labdanum obtained in step (C).

In this distillation purification step, desired fractions can be obtained efficiently by adding a hydrocarbon to the heat-treated acidic components of labdanum obtained in step (C) and then carrying out distillation, thus preferred. Suitable hydrocarbons include saturated hydrocarbons having a boiling point of from 260 to 400 °C, and preferably from 300 to 350 °C, at 760 mmHg or mixtures thereof, with liquid paraffin being preferred. Distillation is preferably carried out under a reduced pressure of 1 to 3 mmHg while adjusting the highest column temperature at 220 to 250 °C.

The perfume composition according to the present invention comprises the thus obtained treated labdanum oil in a proportion of from 0.1 to 40 % by weight, and preferably from 0.5 to 15 % by weight.

The novel compound according to the present invention, 4-(3-methyl-3-butenyl)-4α,5,6,7,8,8α-hexahydro-3,4α,8,8-tetramethyl-2(1H)-naphthalenone (hereinafter referred to as compound (I)), will be explained below in detail.

Compound (I) is present in the essential oil of Cistus Ladaniferus L. (labdanum) and can be isolated, for example, from labdanum gum as follows.

The labdanum gum which can be used as a starting material is generally obtained by heating Cistus ladaniferus L. in a boiling aqueous alkali solution and collecting the thus released resinous component. Commercially available labdanum gum products may be used as such.

An organic solvent, such as ethyl ether, petroleum ether, methanol, ethanol, hexane or toluene, is added to labdanum gum, and the mixture is allowed to stand or stirred. The resulting extract is distilled to remove the organic solvent to obtain labdanum gum extract. The acidic components of the labdanum gum extract are then extracted with an aqueous solution of an alkali, such as sodium hydroxide or potassium hydroxide. The alkaline extract is neutralized with hydrochloric acid or sulfuric acid and then re-extracted with an organic solvent, such as petroleum ether, hexane or toluene. The solvent was removed from the extract by distillation, and the residual extract is heated to 200 to 350 °C in a nitrogen atmosphere to obtain a treated labdanum oil with enhanced animal note. This treated labdanum oil may be the aforesaid treated labdanum oil according to the present invention.

The treated labdanum oil is passed through a column of silica gel, for example, and developed first with an organic solvent, such as hexane, to elute hydrocarbon components and then with a mixed solvent of hexane and ethyl ether with an increasing concentration of ethyl ether. The effluent is collected into fractions of an adequate amount, and fractions containing compound (I) are combined. The solvent is removed from the combined fraction by distillation, and the residue is purified by high performance liquid chromatography using, for example, a hexane-ethyl acetate mixed solvent as a developing solution to obtain

compound (I).

The thus obtained compound (I) has an animal, phenolic, and amber-like fragrance with a faint tobacco note and is useful as a material for various perfume compositions.

The amount of compound (I) in a perfume composition is not limited, for it varies depending on the kind of the composition and the desired fragrance, and the like. It is preferably used in an amount of from 0.1 to 10 % by weight, and particularly from 1 to 5 % by weight.

When compound (I) is used in combination with a perfume compound selected from phenylethyl alcohol, citronellol and linalool, floral note of these perfume compounds is enhanced to give a more natural and fresh fragrance. For example, compounding of compound (I) and phenylethyl alcohol at a ratio of 1:60 to 1:20 or compounding of compound (I) and citronellol at a ratio of 1:5 to 1:20 or compounding of compound (I) and linalool at a ratio of 1:60 to 1:20 provides a perfume composition with enhanced freshness.

The perfume composition of the present invention essentially comprises the treated labdanum oil or compound (I) according to the present invention. If desired, it may contain base components and other perfume components commonly employed in general perfume composition, such as diethyl phthalate, dipropylene glycol, ethylene diglycol, and Hercolyn D (methyl dihydroabietate). It may also contain natural perfumes, such as sandalwood oil and patchouli oil, in amounts that will not impair the effect of the present invention.

The perfume composition of the present invention is prepared by mixing the treated labdanum oil or compound (I) and other optional components with stirring in a conventional manner.

The perfume composition of the present invention is applicable to a broad range of products to be scented with perfume, such as perfumes, eu de Cologne, soaps, shampoos, rinses, body shampoos, detergents, sprays, bathing preparations, deodorants, and the like.

The present invention will now be illustrated in greater detail with reference to Examples, but the present invention should not be construed as being limited thereto. All the parts and percents are by weight unless otherwise indicated.

EXAMPLE 1

In a 500 cc four-necked flask equipped with a stirrer, a thermometer, and a Dimroth condenser were charged 200 g of a commercially available labdanum extract "Labdanum Resinoid" (a product of Biolandes Technologies), 200 g of a 10 % aqueous solution of sodium hydroxide, and 400 g of toluene, and the mixture was stirred at 90 °C for 30 minutes in a nitrogen atmosphere. The mixture was allowed to stand for phase separation. After the toluene layer was removed, 400 g of toluene was added to the aqueous layer. The mixture was stirred in the same manner as described above and allowed to stand for phase separation. After removing off the toluene layer, 400 g of toluene was further added to the aqueous layer, followed by stirring, and 25 g of concentrated sulfuric acid was slowly added dropwise, giving care to the inner temperature. After the dropwise addition, the temperature was raised up to 90 °C in a nitrogen atmosphere, and the mixture was allowed to stand for phase separation. After removing off the aqueous layer, a 10 % aqueous solution of mirabilite was added to the organic layer, followed by stirring and allowing to stand for phase separation. Toluene was removed from the organic layer to obtain 71 g of the acidic components of labdanum. Fifty grams of the acidic components were heated at 260 °C for 30 minutes in a nitrogen atmosphere with stirring. The resulting heat-treated labdanum oil had and animal and amber note with an enhanced animal note and was free from a cedarwood-like unpolished odor as compared with the starting labdanum gum extract.

After cooling to 100 °C, 50 g of liquid paraffin #60S (produced by Chuo Kasei Co., Ltd.; boiling point: 278-422 °C) was added thereto dropwise, and the mixture was distilled under reduced pressure. The first fraction of distillate (up to 150 °C/2 Torr) was removed. The temperature was elevated to 185 to 240 °C under reduced pressure of 2 Torr to obtain 59 g of a fraction, which showed further improvements in color and viscosity as compared with the above-obtained treated labdanum oil.

EXAMPLE 2 AND COMPARATIVE EXAMPLE 1

A perfume composition was prepared from the components shown in Table 1 below in a conventional manner.

TABLE 1

Component	Example 2 (part by weight)	Comparative Example 1 (part by weight)
Bergamot oil base*	300	300
Petitgrain oil from Paraguay	200	200
Benzyl acetate	100	100
Jasmin oil base*	30	30
Rose oil Bulgaria base*	20	20
Citronellol	100	100
Musk ketone	50	50
Styrax oil	80	80
Styrax Resinoid	20	20
Birch Tar Oil	50	50
Treated labdanum oil of Example 1	50	-
Labdanum resinoid	-	50
Total	1000	1000

Note: *: Synthetic essential oils, all produced by Kao Corporation.

The composition of Example 2 had a leather note with an enhanced animal note as compared with the comparative composition.

EXAMPLE 3

In a 500 cc four-necked flask equipped with a stirrer, a thermometer, and a Dimroth condenser were charged 200 g of a commercially available labdanum gum extract "Labdanum Resinoid" (a product of Biolandes Technologies), 200 g of a 10 % aqueous solution of sodium hydroxide, and 400 g of toluene, and the mixture was stirred at 90 °C for 30 minutes in a nitrogen atmosphere. The mixture was allowed to stand for phase separation. After the toluene layer was removed, 400 g of toluene was added to the aqueous layer. The mixture was stirred in the same manner as described above and allowed to stand for phase separation. After removing off the toluene layer, 400 g of toluene was further added to the aqueous layer, followed by stirring, and 25 g of concentrated sulfuric acid was slowly added dropwise, giving care to the inner temperature. After the dropwise addition, the temperature was raised up to 90 °C in a nitrogen atmosphere, and the mixture was allowed to stand for phase separation. After removing off the aqueous layer, a 10 % aqueous solution of mirabilite was added to the organic layer, followed by stirring and allowing to stand for phase separation. Toluene was removed from the organic layer by distillation to obtain 71 g of the acidic components of labdanum. Fifty grams of the acidic components were heated at 260 °C for 30 minutes in a nitrogen atmosphere while stirring. The inner temperature was cooled to 100 °C, and 50 g of liquid paraffin #60S (produced by Chuo Kasei Co., Ltd.; boiling point: 278-422 °C) was added thereto dropwise. After the first fraction of distillate (up to 150 °C/2 Torr) was removed, the temperature was elevated to obtain 59 g of a fraction (185 to 240 °C/2 Torr).

A 3.0 g portion of the resulting distillate was subjected to column chromatography using silica gel ("WAKOGEL C-300", produced by Wako Pure Chemical Industries, Ltd.) and eluted first with 300 mℓ of hexane and then with 100 mℓ of a 90:10 mixture of hexane and ethyl ether. Then, 100 mℓ of a 90:10 mixture of hexane and ethyl ether was passed through the column to obtain 0.5 g of Fraction 1. Fraction 1 was fractionated by high performance liquid chromatography (HPLC) (hexane/ethyl ether:90/10) to obtain a fraction (160 mg) in which compound (I) was concentrated to a purity of 58 %. The fraction was further subjected to HPLC (hexane/ethyl ether:95/5) to obtain 80 mg of compound (I).

^{1}H-NMR ($CDC\ell_3$) δ ppm: 0.89 (s, 3H), 0.92 (s, 3H), 1.10 (s, 3H), 1.21-1.74 (m, 5H), 1.77 (s, 3H), 1.78 (s, 3H), 2.10-2.58 (m, 8H), 4.75 (s, 2H)

^{13}C-NMR ($CDC\ell_3$) δ ppm: 11.3, 18.1, 18.6, 21.3, 22.4, 28.1, 32.5, 33.2, 35.3, 35.8, 36.7, 41.2, 41.3, 50.3, 110.1, 130.1, 145.3, 167.9, 200.3

MS (70 eV): 274 (M), 259 (M-15), 205 (M-69)

IR: 1695 cm^{-1}(C = O)

EXAMPLE 4 AND COMPARATIVE EXAMPLE 2

A muguet-like perfume composition was prepared from the components shown in Table 2 below in a conventional manner.

TABLE 2

Component	Example 4 (part by weight)	Comparative Example 2 (part by weight)
Phenylethyl alcohol	203	203
Amylcinnamic aldehyde	200	200
2-Methyl-3-(p-isopropylphenyl)propionaldehyde (cyclamen aldehyde)	20	20
Geraniol	100	100
Citronellol	50	50
Linalool	180	180
Benzyl acetate	100	100
Ethyl acetoacetate	2	2
Methyl salicylate	10	10
Methyl cinnamate	10	10
6-Acetyl-1,1,3,4,4,6-hexamethyltetrahydronaphthalenone (Tenterome)	20	20
2-Methyl-3-(p-t-butylphenyl)propionaldehyde	100	100
Compound (I)	5	-
Diethyl phthalate	-	5

The perfume composition of Example 4 had a muguet note with an enhanced freshness, an enhanced sweetness, and an enhanced softness as compared with the comparative composition.

The treated labdanum oil of the present invention is free from a cedarwood-like rough odor and has an animal and amber fragrance with an enhanced animal note. Additionally, it has a greatly reduced color, and thus, is widely applicable as a material of perfume compositions for soaps, detergents, etc.

Compound (I) of the present invention has an animal, ambler-like fragrance and is useful as a material of various perfume compositions. The perfume composition containing compound (I) is suited to perfumes, soaps, and the like.

While the invention has been described in detail and with reference to specific examples thereof, it will be apparent to one skilled in the art that various changes and modifications can be made therein without departing from the spirit and scope thereof.

Claims

1. A process for preparing a treated labdanum oil comprising:
(A) a step of mixing a labdanum raw material with an aqueous alkali solution and separating the aqueous layer;
(B) a step of adding an acidic substance to the aqueous layer separated in step (A) and separating the thus released acidic components of labdanum; and
(C) a step of heating the acidic components obtained in step (B) at a temperature of from 200 to 350 °C for a period of from 0.1 to 10 hours.

2. The process of claim 1, wherein the process further comprises: (D) a step of purifying said acidic components obtained in step (C) by distillation.

3. The process of claim 1 or 2, wherein said labdanum raw material is at least one selected from the group consisting of (1) labdanum gum, which is a resin separated on boiling of a labdanum-containing plant in water, (2) labdanum gum extract obtained by removing inorganic solids from the labdanum gum, and (3) labdanum gum distillate obtained by distillation or fractional distillation of the labdanum gum extract.

4. The process of any of claims 1 to 3, wherein said alkali used in step (A) is at least one selected from the group consisting of an alkali metal hydroxide, an alkaline earth metal hydroxide, an alkali metal carbonate, and an alkali metal hydrogencarbonate.

5. The process of any of claims 1 to 4, wherein said acidic substance used in step (B) is at least one selected from the group consisting of sulfuric acid, hydrochloric acid and phosphoric acid.

6. The process of any of claims 1 to 5, wherein said heating in step (C) is carried out in a nitrogen atmosphere.

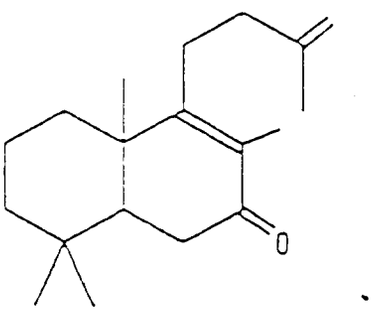
7. The process of claim 2, wherein said distillation in step (D) is carried out by adding a hydrocarbon compound having a boiling point of from 260 to 400 °C at atmospheric pressure to said acidic components and collecting a fraction of up to a boiling point of 250 °C under a pressure of 1 mmHg.

8. The process of claim 7, wherein said hydrocarbon compound is liquid paraffin.

9. A treated labdanum oil obtainable by a process as defined in any one of the claims 1 to 8.

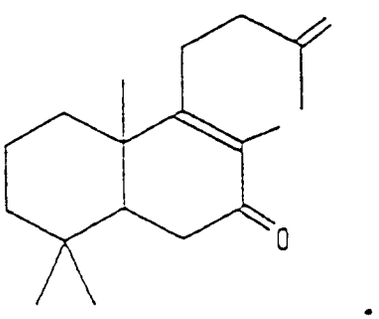
10. A perfume composition containing the treated labdanum oil of claim 9.

11. 4-(3-Methyl-3-butenyl)-4α,5,6,7,8,8α-hexahydro-3,4α,8,8-tetramethyl-2(1H)-naphthalenone represented by formula (I):



(I)

12. A perfume composition containing 4-(3-methyl-3-butenyl)-4α,5,6,7,8,8α-hexahydro-3,4α,8,8-tetramethyl-2(1H)-naphthalenone represented by formula (I):



(I)

13. The perfume composition according to claim 12, which further comprises at least one perfume compound selected from phenylethyl alcohol, citronellol, and linalool.