

Description**BACKGROUND OF THE INVENTION**5 **Field of the Invention:**

The present invention relates to a novel fragrance-imparting compound and more particularly to a compound with a woody or amber odor, having an excellent residual odor property, and a perfume composition containing the same.

10

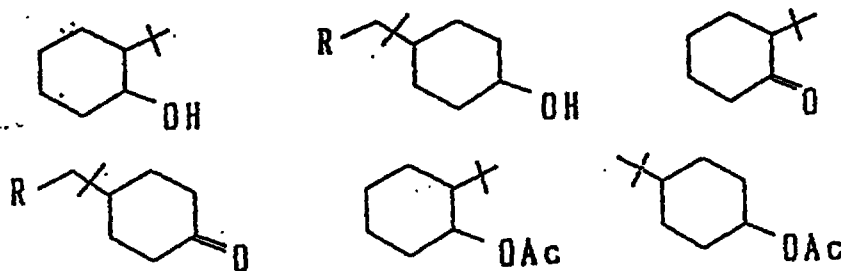
Description of the Background:

Perfumes having a residual odor property among perfumes having a woody or amber odor are used as a base note in compounding perfumes and are important materials for determining odors as a frame of compound perfumes. However, most of these compounds including natural materials are fairly expensive, which places a large restriction on the use of the materials in inexpensive compound perfumes. Hence, it is quite important to develop inexpensive materials having a woody or amber fragrance and a residual odor property.

It has been reported that ortho- and paraalkylcyclohexanols, ortho- and paraalkylcyclohexanones, ortho- and paraalkylcyclohexyl acetates, and the like, which are derived from alkylphenols and are represented by the following formulas,

25

30



35

wherein R is a hydrogen atom or a methyl group, possess a woody odor. The compounds have been widely used as inexpensive materials, as disclosed in "Perfume and Flavor Chemicals", S. Arctander, Elizabeth (1969).

EP-A-0 081 699 discloses cyclohexyloxypentanol derivatives and the use of such compounds as ingredients of perfumes.

40 **SUMMARY OF THE INVENTION**

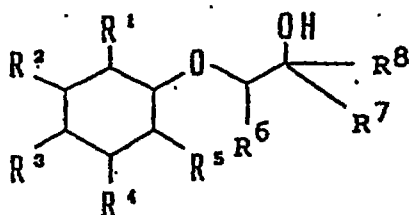
Accordingly, one object of the present invention is to provide a compound having a woody or amber odor and an excellent residual odor property.

Another object of the present invention is to provide a perfume composition containing a compound having a woody or amber odor and an excellent odor property.

Briefly, these objects and other objects of the present invention as hereinafter will become more readily apparent can be attained by an α -(alkylcyclohexyloxy)- β -alkanol of the formula (1)

50

55



(1)

wherein at least one of substituents R^1 , R^2 , R^3 , R^4 and R^5 is a tertiary alkyl group of 4-8 carbon atoms, and the remaining R^1 , R^2 , R^3 , R^4 and R^5 groups are hydrogen atoms or methyl groups, and wherein R^6 , R^7 and R^8 are hydrogen atoms or the same or different alkyl groups having 1-6 carbon atoms, with the proviso that R^6 , R^7 and R^8 are not all hydrogen atoms at the same time.

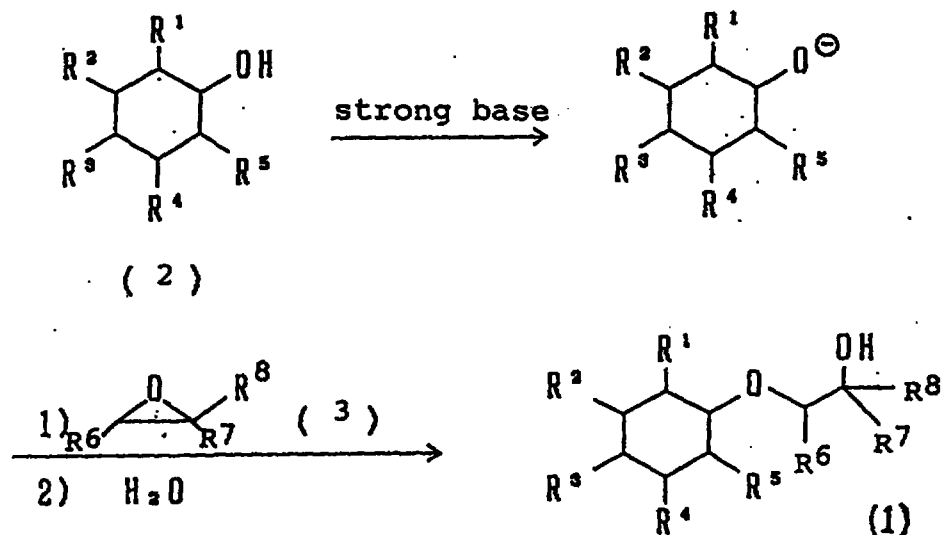
In accordance with another aspect of the present invention, a perfume composition is provided which contains an α -(alkylcyclohexyloxy)- β -alkanol described above.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The inventors have directed research effort and investigation into compounds which exhibit a woody or amber odor, which have an excellent residual odor property and which can be prepared at a low price. It has been found that cyclohexanes obtained by the hydrogenation of epoxide addition products of alkylcyclohexanols or alkylphenols satisfy such requirements. In formula (1) above, methyl, ethyl, n-propyl, iso-propyl, n-butyl, iso-butyl, tert-butyl, n-pentyl, n-hexyl and the like are examples of alkyl groups for R^6 , R^7 and R^8 . In these groups the alkyl groups having 1 to 4 carbon atoms in particular are preferred.

The present compounds of formula (1) can be prepared, for example, by any of the following reaction schemes.

(A) In the following scheme, alkylcyclohexanol (2) is treated in the presence of a strong base to make an alcoholate, and then the alcoholate is reacted with an epoxide (3) to prepare the product compound of formula (1).

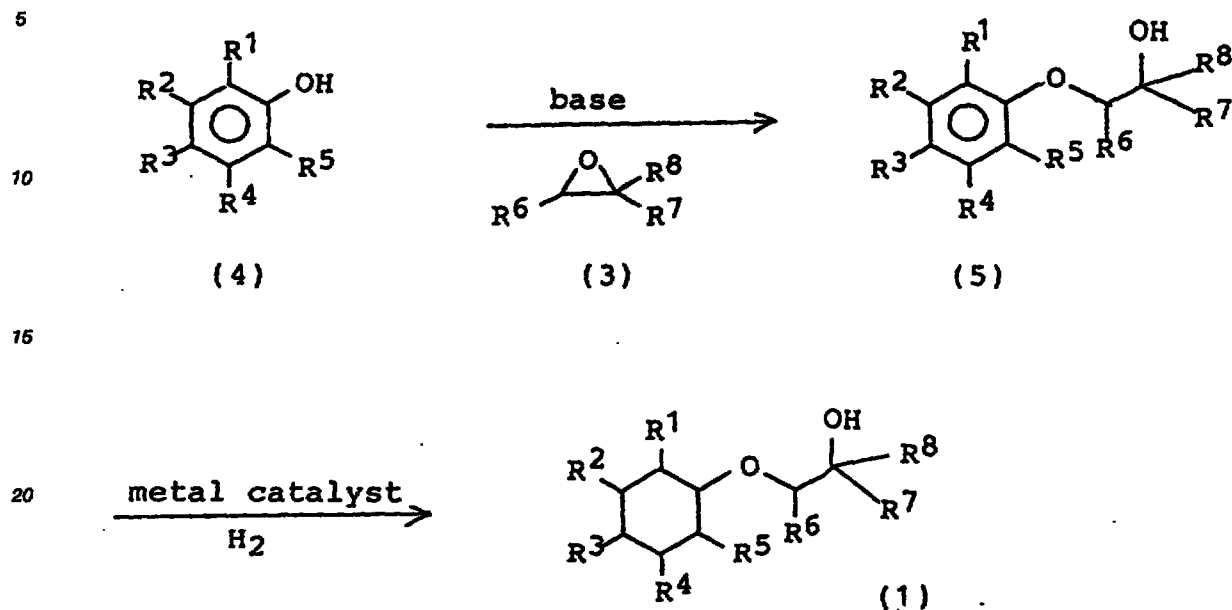


In the formulas, R^1 to R^8 are as defined above.

Solvents which are useful in the reaction include ether solvents such as diethyl ether, dibutyl ether, tetrahydrofuran and the like, and hydrocarbon solvents such as hexane, benzene, toluene, xylene and the like. Suitable strong bases which can be used include alkali metal hydrides such as sodium hydride, lithium hydride, potassium hydride and the like; alkali metal amides such as sodium amide, lithium amide, potassium amide and the like; alkali metals such as sodium, lithium, potassium and the like; and metal alkyl compounds such as alkyl lithium, alkyl magnesium halide and the like. The amount of the strong base used in the above reaction ranges from 0.1 to 2.0 equivalent per equivalent of alkylcyclohexanol (2), and a 1.0 to 1.2 equivalent amount is preferred in particular. The preferred amount of the epoxide (3) used in the above reaction is in a range of a 1.0 to 5.0 equivalent per equivalent of alkylcyclohexanol (2), and a 1.0 to 1.2 equivalent amount in particular is preferred. Both the alcoholate forming reaction and the epoxide addition reaction are carried out at a temperature of 30 to 120 °C, preferably at 50 to 100 °C.

In this case, when alkylcyclohexanol (2), as a raw material, is a mixture of cis-trans isomers, the compound (1) obtained is also a mixture of cis-trans isomers. The mixture of the isomers may be separated by, for example, column chromatography or the like, or can it be used as it is.

(B) In another embodiment, alkylphenol (4) is reacted with the epoxide (3) in the presence of a base catalyst to form α -(alkylphenyleneoxy)- β -alkanol (5), and the obtained compound (5) is hydrogenated in the presence of a metal catalyst to prepare present compound (1).



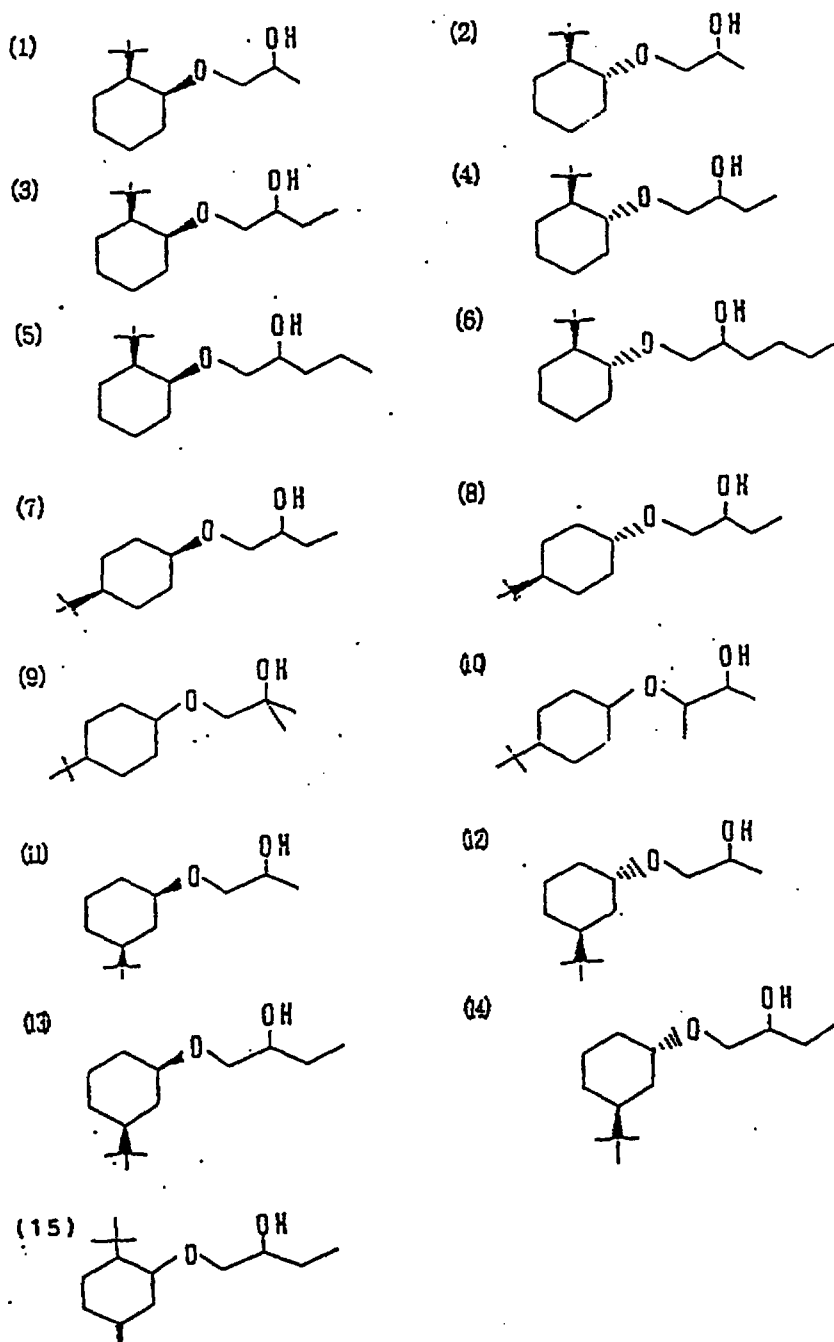
In the formulas, R¹ to R⁸ are as defined above.

Suitable solvents which can be used in the above-described epoxide addition reaction include alcohol solvents such as methanol, ethanol, n-propanol, isopropanol and the like; ether solvents such as diethyl ether, dibutyl ether, tetrahydrofuran and the like; and hydrocarbon solvents such as hexane, benzene, toluene, xylene and the like. The reaction however, can also be carried out without using any solvent. Suitable examples of the base catalyst include alkali metal hydroxides such as sodium hydroxide, lithium hydroxide, potassium hydroxide and the like, and alkali metal carbonates such as sodium carbonate, lithium carbonate, potassium carbonate and the like. The bases can be used in powder form or as a solution containing the same. The amount of the base catalyst-used in the above reaction is 0.01 to 2.0 equivalent per equivalent of alkylphenol (4), and 0.05 to 0.5 equivalent is preferred in particular. The amount of the epoxide (3) normally used in the above reaction is in a range of 1.0 to 5.0 equivalent per equivalent of alkylphenol (4), and 1.0 to 1.2 equivalent amount in particular is preferred. The epoxide addition reaction is carried out at a temperature of 30 to 200 °C, preferably at 50 to 150 °C.

Suitable solvents which can be used in the above-described hydrogenation reaction of the compound (5) include alcohol solvents such as methanol, ethanol, n-propanol, isopropanol and the like, and hydrocarbon solvents such as hexane, heptane, cyclohexane, methylcyclohexane and the like. However, the reaction can also be carried out without using any solvent. Suitable examples of the metal catalyst include palladium catalysts, ruthenium catalysts, rhodium catalysts, platinum catalysts, nickel catalysts and the like. The metal catalyst is used in the reaction in an amount ranging from 0.01 to 10% by weight relative to compound (5), and the range of 0.05 to 5% by weight is preferred. The hydrogenation reaction is carried out at a temperature of 50 to 300 °C, preferably at 100 to 250 °C. The pressure of the hydrogen in this reaction is a range of 1 to 150 atms, preferably a range of 10 to 100 atms.

Compound (1) in the form of a mixture of cis-trans isomers is formed by the hydrogenation of compound (5). The mixture of the isomers may be separated by, for example, column chromatography or the like, or it can be used as it is.

Embodiments of the present compound (1) include those having the following structure:



The odors of these compounds of the present invention are tabulated as follows.

Compound No.	Odor	Residual Odor Property (days) **
(1)	woody, amber, minty	3
(2)	woody, amber, camphor	3
(3)	woody, amber, hay	7
(4)	woody, camphor, earthy, amber	7
(5)	woody, sandalwoody	14
(6)	woody	14
(7)	woody, sandalwoody, rose	7
(8)	woody	7
(9)	woody, amber, green	7
(10)	woody, green	7
(11)	woody	3
(12)	woody	3
(13)	woody	7
(14)	woody, sandalwoody	7
(15)	woody, amber, earthy	14

** Compound is applied to odor paper and the number of days the odor persists is measured.

As described above, the present compounds of formula (1) can be produced by using inexpensive materials and the present compounds having a woody or amber odor possess an excellent residual odor property. Hence, by blending the present compound (1), as a base note, with other odorants, an excellent perfume composition can be obtained. The amount of the present compound (1), blended into the likes of perfume compositions, varies depending on other compounded perfume ingredients, destination odors, and the like and is not limited provided that a woody or amber fragrance can be imparted. Further, the perfume compositions of the present invention can be widely used as a fragrance imparting component for perfumes, soaps, shampoos, rinses, detergents, cosmetics, fragrance imparting agents, and so forth either independently or in combination with other perfume compositions.

Having now generally described this invention, a further understanding can be obtained by reference to certain specific examples which are provided herein for purposes of illustration only and are not intended to be limiting unless otherwise specified.

EXAMPLES

Example 1:

Synthesis of 1-(2-tert-butylcyclohexyloxy)-2-propanol:

[Compounds (1) and (2)]

To a 300 ml round bottom flask provided with a Dimroth condenser and a dropping funnel, 7.5 g (0.194 mol) of sodium hydride (62 weight % mineral oil dispersion type) and 30 ml of tetrahydrofuran were added and heated at 60 °C under a nitrogen gas flow. Then, to this solution a mixture of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans=8:2) and 30 ml of tetrahydrofuran was added dropwise in approximately 30 minutes, and the mixed solution was stirred under reflux for 24 hours until the generation of hydrogen gas had stopped. The reacted mixed solution was cooled to 40 °C, and after dropwise adding 11.1 g (0.192 mol) of propyleneoxide to the mixed solution, the solution was further stirred under reflux for 48 hours. The mixed solution was neutralized by 3 N hydrochloric acid, and the organic layer was separated from the water layer. Distillation was then conducted and 9.9 g of 2-tert-butylcyclohexanol and 24.2 g of 1-(2-tert-butylcyclohexyloxy)-2-propanol (cis:trans=8:2) (bp. 112 to 113 °C/5 mm Hg) were obtained in a 59% yield. Further, the cis-trans isomers of 1-(2-tert-butylcyclohexyloxy)-2-propanol were separated from each other by column chromatography using a developing solvent composed of Kieselgel 60 (trademark) of Merck Corporation and hexane : ethyl acetate = 95 : 5.

1-(cis-2-tert-butylcyclohexyloxy)-2-propanol:

[Compound (1)]

5	IR (film, cm^{-1}):	905, 966, 1089, 1146, 1195, 1368, 2866, 2938, 3406
	NMR (60MHz, CDCl_3 , ppm):	0.95 (s, 9H), 1.16 (d, $J=7\text{Hz}$, 3H), 1.2-2.0 (m, 8H), 2.1-2.5(m, 2H), 2.8-3.6 (m, 2H), 3.6-4.2 (m, 2H)
	GC-MS(M^+):	214

10 1-(trans-2-tert-butylcyclohexyloxy)-2-propanol:

[Compound (2)]

	IR (film, cm^{-1}):	975, 1095, 1371, 1452, 2866, 2932, 3206
15	NMR (60MHz, CDCl_3 , ppm):	0.98 (s, 9H), 1.15 (d, $J=7\text{Hz}$, 3H), 1.1-2.4 (m, 10H), 2.9-4.2 (m, 4H)
	GC-MS(M^+):	214

Example 2:

20 Synthesis of 1-(2-tert-butylcyclohexyloxy)-2-butanol:

[Compounds (3) and (4)]

25 The synthesis was carried out in the same manner as described in Example 1, except that 13.8 g (0.192 mol) of 1,2-butyleneoxide was used instead of 11.1 g (0.192 mol) of propyleneoxide, to obtain 12.3 g of 2-tert-butylcyclohexanol and 24.1 g of 1-(2-tert-butylcyclohexyloxy)-2-butanol (cis:trans=8:2) (bp. 122 to 123°C/5 mm Hg) in a 55% yield. Then, the cis-trans isomers of 1-(2-tert-butylcyclohexyloxy)-2-butanol were separated from each other in the same manner as described in Example 1.

30 1-(cis-2-tert-butylcyclohexyloxy)-2-butanol:

[Compound (3)]

	IR (film, cm^{-1}):	890, 960, 1089, 1180, 1365, 1464, 2855, 2932, 3424
35	NMR (60MHz, CDCl_3 , ppm):	0.93 (s, 9H), 0.97 (t, $J=7\text{Hz}$, 3H), 1.0-2.0 (m, 10H), 2.0-2.4 (m, 2H), 3.0-3.9 (m, 4H)
	GC-MS(M^+):	228

1-(trans-2-tert-butylcyclohexyloxy)-2-butanol:

40

[Compound (4)]

	IR (film, cm^{-1}):	972, 1095, 1368, 1452, 2866, 2932, 3448
45	NMR (60 MHz, CDCl_3 , ppm):	0.93 (s and t, $J=7\text{Hz}$, 9H and 3H), 1.0-1.9 (m, 10H), 2.0-2.4 m, 2H), 3.0-3.8 (m, 4H)
	GC-MS(M^+):	228

Example 3:

50 Synthesis of 1-(2-tert-butylcyclohexyloxy)-2-butanol:

[Compounds (3) and (4)]

55 (a) To a 300 ml round bottom flask provided with a Dimroth condenser and a dropping funnel, 35 g of 48 weight % sodium hydroxide solution and 350 g (2.33 mol) of 2-tert-butylphenol were added and heated at 80°C under a nitrogen gas flow atmosphere. Then, to this solution 176 g (2.45 mol) of 1,2-butyleneoxide was added dropwise in approximately 2 hours, and the mixed solution was stirred at 80°C for 5 hours. After cooling the reacted mixed solution, the lower sodium hydroxide solution layer was

separated from the organic layer. Distillation was then conducted and 497 g of 1-(2-tert-butylphenyloxy)-2-butanol (bp. 130 °C/4 mm Hg) were obtained in a 96% yield.

1-(2-tert-butylphenyloxy)-2-butanol:

[Compound (5a)]

IR (film, cm^{-1}): 744, 975, 1038, 1092, 1134, 1233, 1290, 1362, 1392, 1443, 1491, 1599, 2956, 3058, 3412

NMR (60 MHz, CDCl_3 , ppm): 1.07 (t, $J=7\text{Hz}$, 3H), 1.40 (s, 9H), 1.5-1.8 (m, 2H), 2.15 (s, $J=4\text{Hz}$, 1H), 3.8-4.1 (m, 3H), 6.8-7.4 (m, 4H)

GC-MS(M^+): 222

(b) To a 500 ml autoclave, 50 g (0.23 mol) of 1-(2-tert-butylphenyloxy)-2-butanol, 150 g of isopropanol and 1.0 g of 5 weight % palladium catalyst supported on active carbon containing 50 weight % water (produced by N. E. Chemcat Co.) were added, and the mixture obtained was reacted at 190 °C at a hydrogen pressure of 70 kg/cm^2 for 27 hours until absorption of hydrogen had stopped. After the reaction was finished, the catalyst was filtered to remove it, and the reacted solution was distilled and 38.8 g of 1-(2-tert-butylcyclohexyloxy)-2-butanol (cis:trans=63:37) were obtained in a 74% yield of product.

Example 4:

Synthesis of 1-(2-tert-butylcyclohexyloxy)-2-hexanol:

[Compounds (5) and (6)]

The synthesis was carried out in the same manner as described in Example 1, except that 19.2 g (0.192 mol) of 1,2-hexeneoxide was used instead of 11.1 g (0.192 mol) of propyleneoxide, 12.9 g of 2-tert-butylcyclohexanol and 26.5 g of 1-(2-tert-butylcyclohexyloxy)-2-hexanol (cis:trans=8:2) (bp. 116 to 117 °C/1 mm Hg) were obtained in a 54% yield. Then, the cis-trans isomers of 1-(2-tert-butylcyclohexyloxy)-2-hexanol were separated from each other in the same manner as described in Example 1.

1-(cis-2-tert-butylcyclohexyloxy)-2-hexanol:

[Compound (5)]

IR (film, cm^{-1}): 890, 960, 1089, 1176, 1365, 1464, 2860, 2932, 3430

NMR (60 MHz, CDCl_3 , ppm): 0.93 (br.s, 12H), 1.1-2.5 (m, 16H), 2.9-3.6 (m, 2H), 3.6-4.0 (m, 2H)

GC-MS(M^+): 256

1-(trans-2-tert-butylcyclohexyloxy)-2-hexanol:

[Compound (6)]

IR (film, cm^{-1}): 970, 1098, 1368, 1452, 2866, 2932, 3466

NMR (60 MHz, CDCl_3 , ppm): 0.97 (br.s, 12H), 1.1-2.5 (m, 16H), 1.0-1.9 (m, 10H), 2.9-3.6 (m, 2H), 3.6-4.0 (m, 2H)

GC-MS(M^+): 256

Example 5:

Synthesis of 1-(4-tert-butylcyclohexyloxy)-2-butanol:

[Compounds (7) and (8)]

The synthesis was carried out in the same manner as described in Example 1, except that 30.0 g (0.192 mol) of 4-tert-butylcyclohexanol (cis:trans=8:2) and 13.8 g (0.192 mol) of 1,2-butyleneoxide were used instead of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans=8:2) and 11.1 g (0.192 mol) of

propyleneoxide. 9.6 g of 4-tert-butylcyclohexanol and 17.9 g of 1-(4-tert-butylcyclohexyloxy)-2-butanol (cis:trans = 8:2) (bp. 130 to 133 °C/5 mm Hg) were obtained in a 41% yield. Then, the cis-trans isomers of 1-(4-tert-butylcyclohexyloxy)-2-butanol were separated from each other in the same manner as described in Example 1.

5

1-(cis-4-tert-butylcyclohexyloxy)-2-butanol:

[Compound (7)]

10 IR (film, cm^{-1}): 1032, 1092, 1116, 1182, 1368, 1470, 2872, 2944, 3470
 NMR (60MHz, CDCl_3 , ppm): 0.87 (s, 9H), 0.97 (t, $J=7\text{Hz}$, 3H), 1.0-1.8 (m, 8H), 1.8-2.4 (M, 4H), 3.2-3.9 (m, 4H)
 GC-MS(M^+): 228

15 1-(trans-4-tert-butylcyclohexyloxy)-2-butanol:

[Compound (8)]

20 IR (film, cm^{-1}): 1030, 1104, 1368, 1458, 2870, 2938, 3466
 NMR (60 MHz, CDCl_3 , ppm): 0.87 (s, 9H), 0.97 (t, $J=7\text{Hz}$, 3H), 1.0-2.4 (m, 12H), 3.0-3.8 (m, 4H)
 GC-MS(M^+): 228

Example 6:

25 Synthesis of 1-(4-tert-butylcyclohexyloxy)-2-methyl-2 propanol:

[Compound (9)]

30 The synthesis was carried out in the same manner as described in Example 1, except that 20.0 g (0.128 mol) of 4-tert-butylcyclohexanol (cis:trans = 4:6) and 8.8 g (0.128 mol) of 2-methylpropionoxide were used instead of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans = 8:2) and 11.1 g (0.192 mol) of propyleneoxide. 6.2 g of 4-tert-butylcyclohexanol and 14.6 g of 1-(4-tert-butylcyclohexyloxy)-2-methyl-2-propanol (cis:trans = 4:6) were obtained in a 50% yield.

35 1-(4-tert-butylcyclohexyloxy)-2-methyl-2-propanol (cis:trans = 4:6):

[Compound (9)]

40 IR (film, cm^{-1}): 918, 1098, 1368, 1470, 2938, 3448,
 NMR (200 MHz, CDCl_3 , ppm): 0.84 (s, 9H), 1.03 (s, 3.6H), 1.06 (s, 2.4H), 0.8-2.2 (m, 9H), 2.42 (s, 0.6H), 2.55 (s, 0.4H), 3.1-3.3 (m, 1H), 3.19 (s, 0.8H), 3.28 (s, 1.2H)
 GC-MS(M^+): 228 (cis), 228 (trans)

Example 7:

45

Synthesis of 2-(4-tert-butylcyclohexyloxy)-3-butanol:

[Compound (10)]

50 The synthesis was carried out in the same manner as described in Example 1, except that 20.0 g (0.128 mol) of 4-tert-butylcyclohexanol (cis:trans = 4:6) and 8.8 g (0.128 mol) of 2,3-butyleneoxide were used instead of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans = 8:2) and 11.1 g (0.192 mol) of propyleneoxide. 11.2 g of 4-tert-butylcyclohexanol and 9.3 g of 2-(4-tert-butylcyclohexyloxy)-3-butanol (a mixture of cis-trans and threo-erythro isomers) were obtained in a 32% yield.

55

2-(4-tert-butylcyclohexyloxy)-3-butanol:

[Compound (10)]

IR (film, cm^{-1}): 912, 969, 1098, 1158, 1260, 1320, 1368, 1446, 2938, 3424
 NMR (200 MHz, CDCl_3 , ppm): 0.87 (s, 9H), 1.08 (d, $J=7\text{Hz}$, 3H), 1.12 (d, $J=7\text{Hz}$, 3H), 0.9-2.0 (m, 9H), 2.32 (br.d, $J=3.5\text{Hz}$, 1H), 3.5-3.7 (m, 1H), 3.7-3.9 (m, 1H)
 GC-MS(M^+): 228

Example 8:

Synthesis of 1-(3-tert-butylcyclohexyloxy)-2-propanol:

[Compounds (11) and (12)]

The synthesis was carried out in the same manner as described in Example 1, except that 30.0 g (0.192 mol) of 3-tert-butylcyclohexanol (cis:trans=2:8) was used instead of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans=8:2). 10.2 g of 3-tert-butylcyclohexanol and 15.2 g of 1-(3-tert-butylcyclohexyloxy)-2-propanol (cis:trans=2:8) (bp. 135 to 138°C/9 mm Hg) were obtained in a 37% yield. Then, the cis-trans isomers of 1-(3-tert-butylcyclohexyloxy)-2-propanol were separated from each other in the same manner as described in Example 1.

1-(cis-3-tert-butylcyclohexyloxy)-2-propanol:

[Compound (11)]

IR (film, cm^{-1}): 963, 1089, 1368, 1392, 1452, 2938, 3424
 NMR (200 MHz, CDCl_3 , ppm): 0.83 (s, 9H), 1.00 (d, $J=7\text{Hz}$, 3H), 0.8-2.1 (m, 9H), 2.61 (s, 1H), 3.1-3.2 (m, 1H), 3.3-3.4 (m, 1H), 3.69 (s, 1H), 3.92 (br.s, 1H)
 GC-MS(M^+): 214

1-(trans-3-tert-butylcyclohexyloxy)-2-propanol:

[Compound (12)]

IR (film, cm^{-1}): 966, 1095, 1368, 1395, 1464, 2938, 3448
 NMR (200 MHz, CDCl_3 , ppm): 0.86 (s, 9H), 1.00 (t, $J=7\text{Hz}$, 3H), 0.8-2.2 (m, 9H), 2.5 (s, 1H), 3.1-3.3 (m, 2H), 3.4-3.6 (m, 1H), 3.91 (br.s, 1H)
 GC-MS(M^+): 214

Example 9:

Synthesis of 1-(3-tert-butylcyclohexyloxy)-2-butanol:

[Compounds (13) and (14)]

The synthesis was carried out in the same manner as described in Example 1, except that 30.0 g (0.192 mol) of 3-tert-butylcyclohexanol (cis:trans=2:8) and 13.8 g (0.192 mol) of 1,2-butyleneoxide were used instead of 30.0 g (0.192 mol) of 2-tert-butylcyclohexanol (cis:trans=8:2) and 11.1 g (0.192 mol) of propyleneoxide. 11.3 g of 3-tert-butylcyclohexanol and 9.3 g of 1-(3-tert-butylcyclohexyloxy)-2-butanol (cis:trans=2:8) (bp. 145 to 148°C/10 mm Hg) were obtained in a 32% yield. Then, the cis-trans isomers of 1-(3-tert-butylcyclohexyloxy)-2-butanol were separated from each other in the same manner as described in Example 1.

1-(cis-3-tert-butylcyclohexyloxy)-2-butanol:

[Compound (13)]

5 IR (film, cm^{-1}): 1095, 1368, 1464, 2938, 3454
 NMR (200 MHz, CDCl_3 , ppm): 0.83 (s, 9H), 0.97 (t, $J=7\text{Hz}$, 3H), 0.8-2.1 (m, 11H), 2.50 (s, 1H), 3.1-3.3 (m, 1H), 3.4-3.5 (m, 1H), 3.69 (br.s, 2H)
 GC-MS(M^+): 228

10 1-(trans-3-tert-butylcyclohexyloxy)-2-butanol:

[Compound (14)]

15 IR (film, cm^{-1}): 1095, 1368, 1464, 2938, 3454
 NMR (200 MHz, CDCl_3 , ppm): 0.86 (s, 9H), 0.97 (t, $J=7\text{Hz}$, 3H), 0.8-1.9 (m, 9H), 2.06 (br.s, 2H), 2.42 (s, 1H), 3.1-3.4 (m, 2H), 3.4-3.6 (m, 1H), 3.6-3.8 (m, 1H)
 GC-MS(M^+): 228

Example 10:

20

Synthesis of 1-(2-tert-butyl-5-methylcyclohexyloxy)-2-butanol:

[Compound (15)]

25 (a) The synthesis was carried out in the same manner as described in Example 3a, except that 382 g (2.33 mol) of 2-tert-butyl-5-methylphenol was used instead of 350 g (2.33 mol) of 2-tert-butylphenol. 522 g of 1-(2-tert-butyl-5-methylphenyloxy)-2-butanol (bp. 139 to 140 °C/3.5 mm Hg) were obtained in a 95% yield.

30 1-(2-tert-butyl-5-methylphenyloxy)-2-butanol:

IR (film, cm^{-1}): 808, 1042, 1086, 1144, 1182, 1258, 1294, 1410, 1460, 1502, 1612, 2960, 3404
 NMR (200 MHz, CDCl_3 , ppm): 1.07 (t, $J=7\text{Hz}$, 3H), 1.39 (s, 9H), 1.5-1.8 (m, 2H), 2.14 (d, $J=4\text{Hz}$, 1H), 2.31 (s, 3H), 3.8-4.1 (m, 3H), 6.6-7.3 (m, 3H)
 GC-MS(M^+): 236

(b) The synthesis was carried out in the same manner as described in Example 3b, except that 50 g (0.21 mol) of 1-(2-tert-butyl-5-methylphenyloxy)-2-butanol were used instead of 50 g (0.23 mol) of 1-(2-tert-butylphenyloxy)-2-butanol and the amount of the same palladium catalyst was increased to 2.5 g to effect the reaction for 4 hours, to obtain 32 g of 1-(2-tert-butyl-5-methylcyclohexyloxy)-2-butanol (bp. 139 to 140 °C/3.5 mm Hg) in a 63% yield.

1-(2-tert-butyl-5-methylcyclohexyloxy)-2-butanol:

45 [Compound (15)]

IR (film, cm^{-1}): 1086, 1365, 1461, 2950, 3448
 NMR (200 MHz, CDCl_3 , ppm): 0.94 (s, 9H), 0.8-2.4 (m, 15H), 2.9-3.8 (m, 4H)
 GC-MS(M^+): 242

50

55

Example 11:

Chypre-type Perfume Composition	
	Parts by weight
Bergamot oil	100
Rose base	100
Methyldihydrojasmonate	100
Lirhal *1	100
Eugenol	20
Benzyl salicylate	50
cis-3-hexenyl salicylate	30
γ -Methyl inone	50
Vetiver oil	20
Sandalmysolcore *2	10
Patchouli oil	100
Musk ketone	50
Amber base	50
Acetylcedrene	100
Total	880

*1: Trademark of IFF Co.;

4-(4-hydroxy-4-methyl-pentyl)-3-cyclohexen-1-carboxyaldehyde

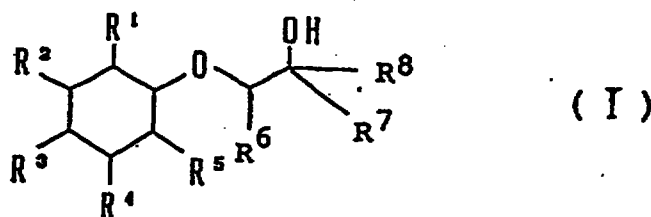
*2: Trademark of Kao Chemicals Co., Ltd.;

2-methyl-4-(2,3,3-trimethyl-3-cyclopentene-1-yl)-2-butene-1-ol

To 880 parts by weight of the above perfume composition 120 parts by weight of 1-(2-tert-butyl-cyclohexyloxy)-2-butanol of the present invention was added to obtain a chypre-type perfume composition having a mild odor with sweetness and bulkiness so as to wrap the wildness of the patchouli oil.

Claims**Claims for the following Contracting States : DE, FR, GB, IT**

1. An α -(alkylcyclohexyloxy)- β -alkanol of formula (1)



wherein at least one of substituents R^1 , R^2 , R^3 , R^4 and R^5 is a tertiary alkyl group of 4-8 carbon atoms, and the remaining R^1 , R^2 , R^3 , R^4 and R^5 groups are hydrogen atoms or methyl groups, and wherein R^6 , R^7 and R^8 are hydrogen atoms or the same or different alkyl groups having 1-6 carbon atoms, with the proviso that R^6 , R^7 and R^8 are not all hydrogen atoms at the same time.

**Example 11:**

Chypre-type Perfume Composition	
	Parts by weight
Bergamot oil	100
Rose base	100
Methyldihydrojasmonate	100
Lirhal *1	100
Eugenol	20
Benzyl salicylate	50
cis-3-hexenyl salicylate	30
γ -Methyl inone	50
Vetiver oil	20
Sandalmysolcore *2	10
Patchouli oil	100
Musk ketone	50
Amber base	50
Acetylcedrene	100
Total	880

*1: Trademark of IFF Co.;

4-(4-hydroxy-4-methyl-pentyl)-3-cyclohexen-1-carboxyaldehyde

*2: Trademark of Kao Chemicals Co., Ltd.;

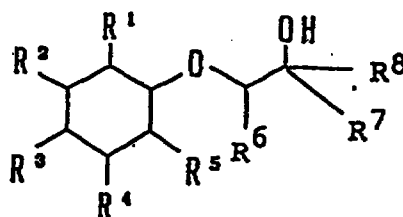
2-methyl-4-(2,3,3-trimethyl-3-cyclopentene-1-yl)-2-butene-1-ol

To 880 parts by weight of the above perfume composition 120 parts by weight of 1-(2-tert-butyl-cyclohexyloxy)-2-butanol of the present invention was added to obtain a chypre-type perfume composition having a mild odor with sweetness and bulkiness so as to wrap the wildness of the patchouli oil.

Claims

Claims for the following Contracting States : DE, FR, GB, IT

1. An α -(alkylcyclohexyloxy)- β -alkanol of formula (1)

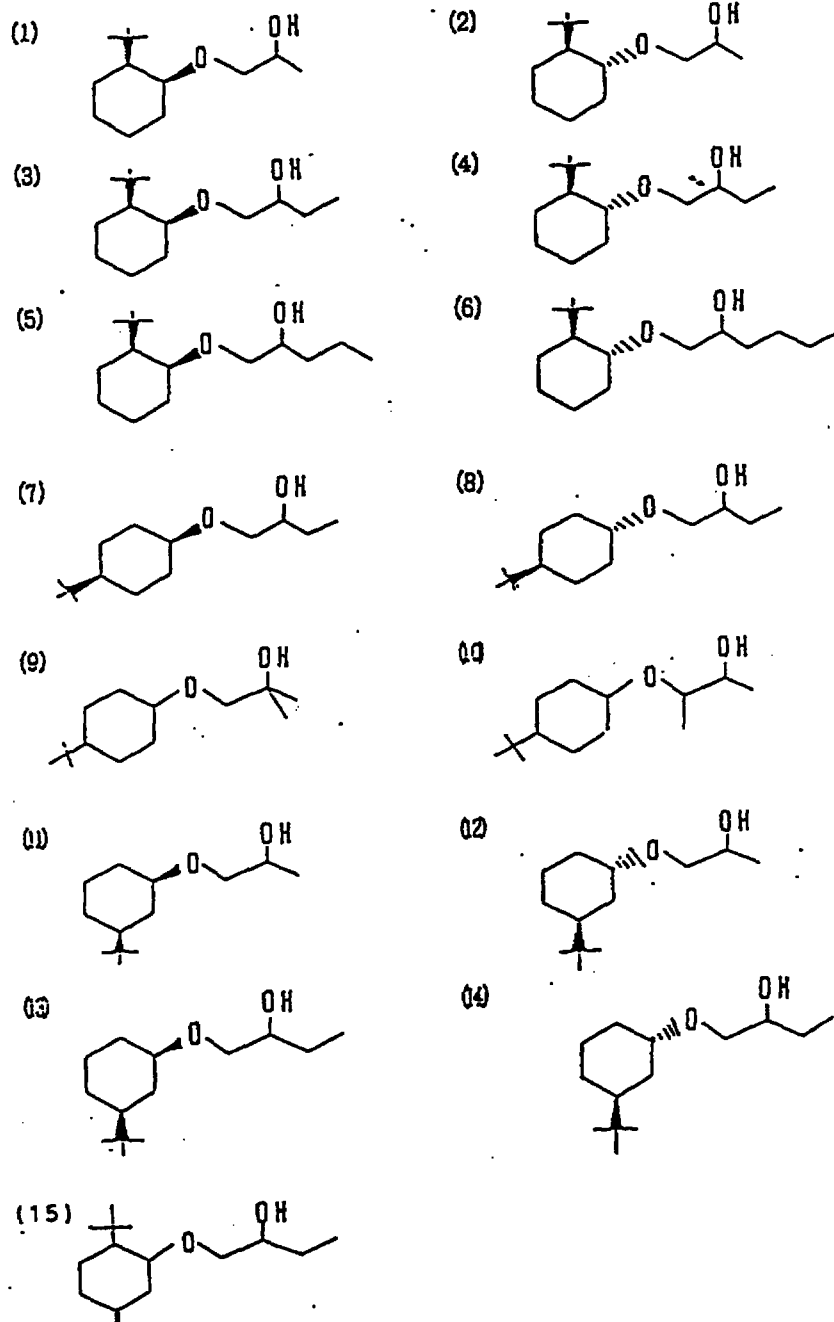


(I)

wherein at least one of substituents R^1 , R^2 , R^3 , R^4 and R^5 is a tertiary alkyl group of 4-8 carbon atoms, and the remaining R^1 , R^2 , R^3 , R^4 and R^5 groups are hydrogen atoms or methyl groups, and wherein R^6 , R^7 and R^8 are hydrogen atoms or the same or different alkyl groups having 1-6 carbon atoms, with the proviso that R^6 , R^7 and R^8 are not all hydrogen atoms at the same time.



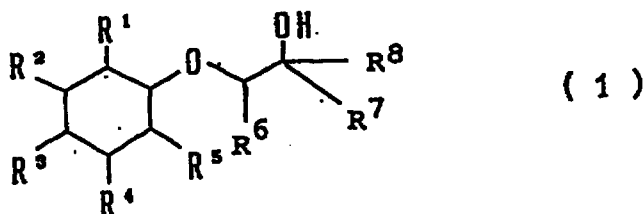
2. The compound of Claim 1, wherein said compound is



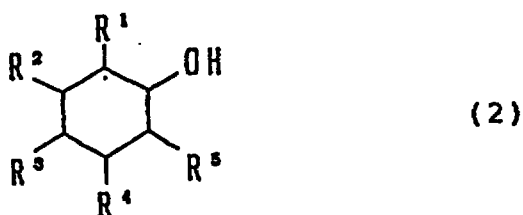
3. A perfume composition containing the α -(alkylcyclohexyloxy)- β -alkanol described in Claim 1 in combination with the other components of the composition.



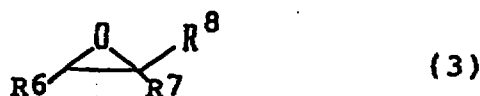
4. A process for preparing an α -(alkylcyclohexyloxy)- β -alkanol represented by formula (1):



wherein R^1 , R^2 , R^3 , R^4 , R^5 , R^6 , R^7 and R^8 are as defined in claim 1, which comprises the steps of reacting an alkylcyclohexanol of formula (2):

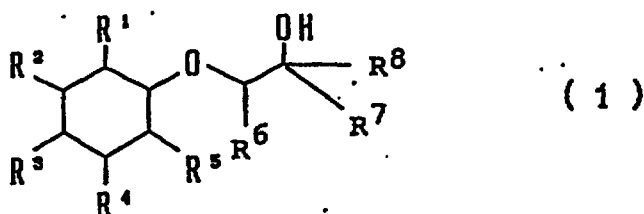


wherein R^1 , R^2 , R^3 , R^4 and R^5 have the same meanings as defined before, with a strong base; and then reacting the product obtained with an epoxide of formula (3):

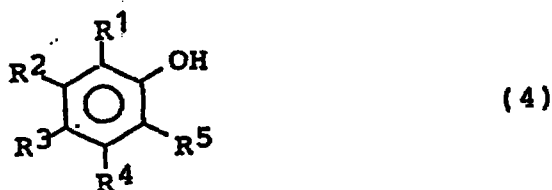


wherein R^6 , R^7 and R^8 have the same meanings as defined above.

- 35
5. A process for preparing an α -(alkylcyclohexyloxy)- β -alkanol represented by formula (1):

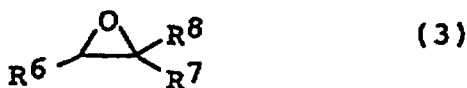


wherein R^1 , R^2 , R^3 , R^4 , R^5 , R^6 , R^7 and R^8 are as defined in claim 1, which comprises the steps of reacting an alkylphenol of formula (4):

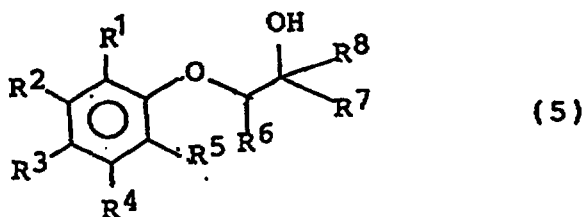




wherein R^1 , R^2 , R^3 , R^4 and R^5 have the same meanings as defined above, with an epoxide of the formula (3):



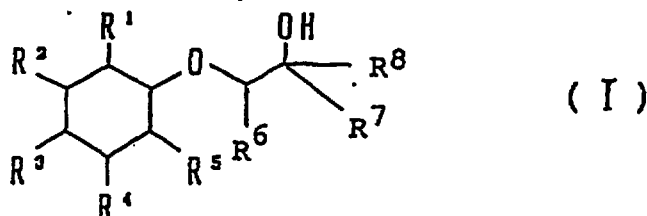
10 wherein R^6 , R^7 and R^8 have the same meanings as defined above in the presence of a base to obtain an α -(alkylphenyloxy)- β -alkanol of the formula (5):



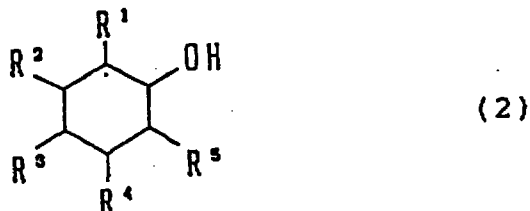
wherein R^1 to R^8 respectively have the same meanings as defined above; and then hydrogenating the α -(alkylphenyloxy)- β -alkanol in the presence of a metal catalyst.

25 **Claims for the following Contracting State : ES**

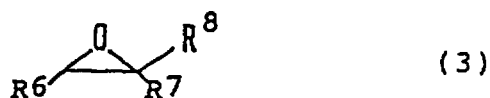
1. A process for preparing an α -(alkylcyclohexyloxy)- β -alkanol of formula (1)



40 wherein at least one of substituents R^1 , R^2 , R^3 , R^4 and R^5 is a tertiary alkyl group of 4-8 carbon atoms, and the remaining R^1 , R^2 , R^3 , R^4 and R^5 groups are hydrogen atoms or methyl groups, and wherein R^6 , R^7 and R^8 are hydrogen atoms or the same or different alkyl groups having 1-6 carbon atoms, with the proviso that R^6 , R^7 and R^8 are not all hydrogen atoms at the same time, which comprises the steps of reacting an alkylcyclohexanol of formula (2):

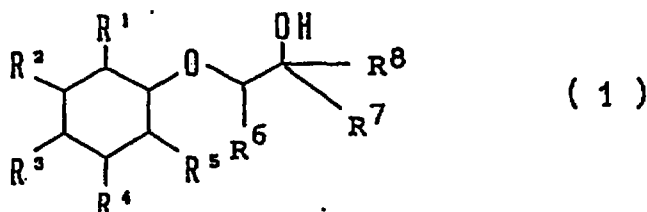


55 wherein R^1 , R^2 , R^3 , R^4 and R^5 have the same meanings as defined before, with a strong base; and then reacting the product obtained with an epoxide of formula (3):

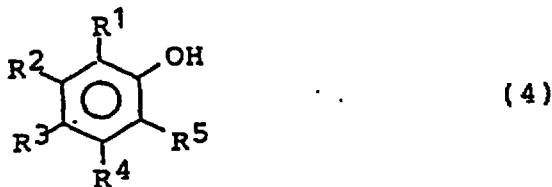


wherein R^6 , R^7 and R^8 have the same meanings as defined above.

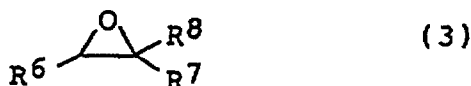
2. A process for preparing an α -(alkylcyclohexyloxy)- β -alkanol represented by formula (1):



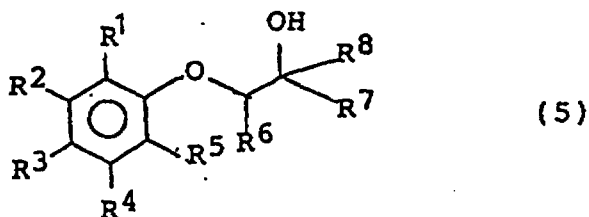
wherein R^1 , R^2 , R^3 , R^4 , R^5 , R^6 , R^7 and R^8 are as defined in claim 1, which comprises the steps of reacting an alkylphenol of formula (4):



wherein R^1 , R^2 , R^3 , R^4 and R^5 have the same meanings as defined above, with an epoxide of the formula (3):

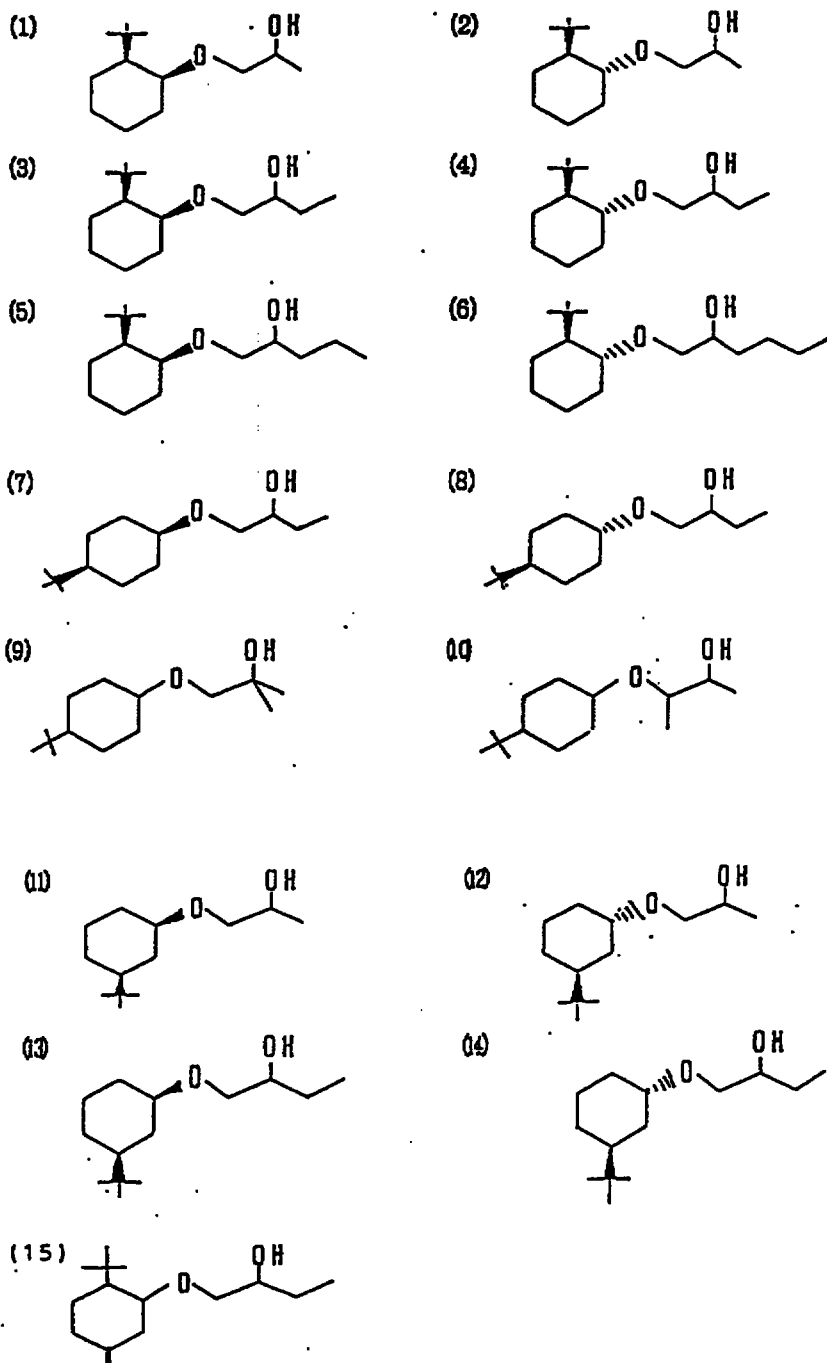


wherein R^6 , R^7 and R^8 have the same meanings as defined above in the presence of a base to obtain an α -(alkylphenyloxy)- β -alkanol of the formula (5):



wherein R^1 to R^8 respectively have the same meanings as defined above; and then hydrogenating the α -(alkylphenyloxy)- β -alkanol in the presence of a metal catalyst.

3. The process according to Claim 1 or 2 which produces a compound of the following formulae (1) to (15):



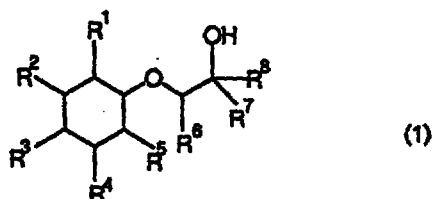
4. A perfume composition containing the α -(alkylcyclohexyloxy)- β -alkanol described in Claim 1 in combination with the other components of the composition.



Patentansprüche

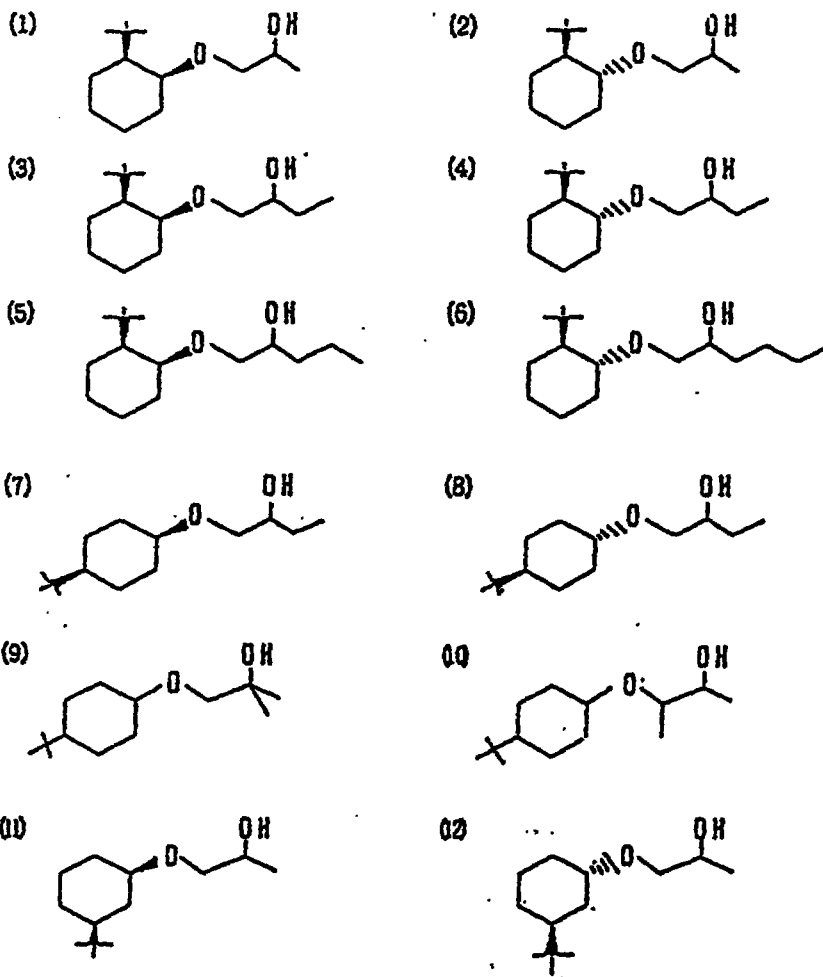
Patentansprüche für folgende Vertragsstaaten : DE, FR, GB, IT

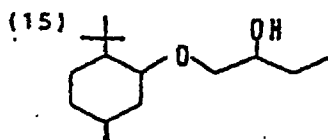
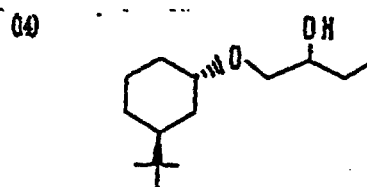
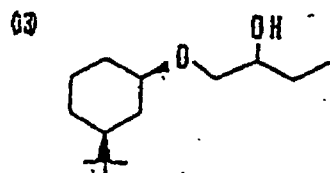
1. α -(Alkylcyclohexyloxy)- β -alkanol der Formel (1)



worin mindestens einer der Substituenten R^1 , R^2 , R^3 , R^4 und R^5 eine tertiäre Alkylgruppe mit 4 bis 8 Kohlenstoffatomen ist, und die übrigen R^1 -, R^2 -, R^3 -, R^4 - und R^5 -Gruppen Wasserstoffatome oder Methylgruppen sind, und worin R^6 , R^7 , und R^8 Wasserstoffatome oder die gleichen oder verschiedene Alkylgruppen mit 1 bis 6 Kohlenstoffatomen sind, unter der Bedingung, daß R^6 , R^7 und R^8 nicht alle gleichzeitig Wasserstoff sein können.

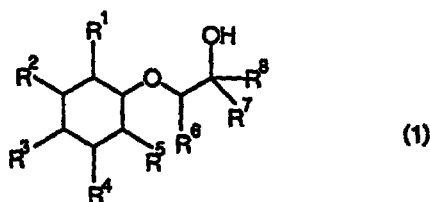
2. Verbindung nach Anspruch 1, worin diese Verbindung ist



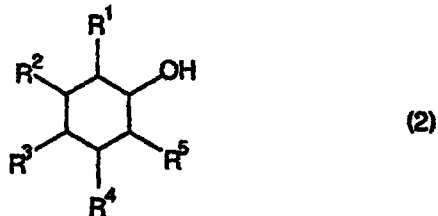


15 3. Parfüm-Zusammensetzung, enthaltend den in Anspruch 1 beschriebenen α -(Alkylcyclohexyloxy)- β -alkanol in Kombination mit den anderen Komponenten der Zusammensetzung.

4. Verfahren zum Herstellen eines α -(Alkylcyclohexyloxy)- β -alkanols der Formel (1):



25 worin R¹, R², R³, R⁴, R⁵, R⁶, R⁷ und R⁸ die in Anspruch 1 angegebene Bedeutung haben, umfassend die Stufen des Umsetzens eines Alkylcyclohexanols der Formel (2):

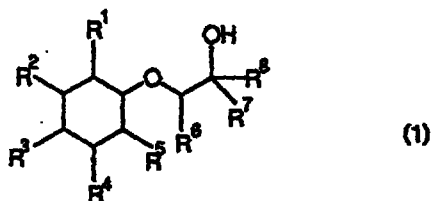


35 worin R¹, R², R³, R⁴ und R⁵ die oben genannten Bedeutungen haben, mit einer starken Base und dann Umsetzen des erhaltenen Produktes mit einem Epoxid der Formel (3):



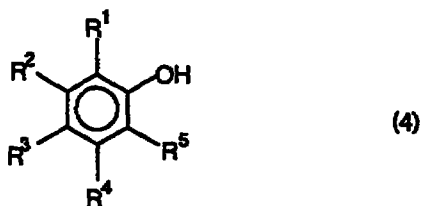
45 worin R⁶, R⁷ und R⁸ die oben genannten Bedeutungen haben.

50 5. Verfahren zum Herstellen eines α -(Alkylcyclohexyloxy)- β -alkanols der Formel (1):





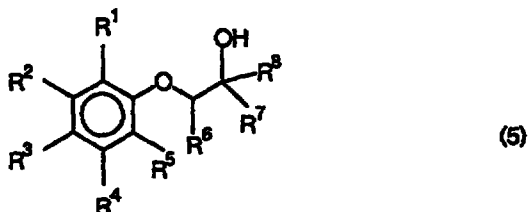
worin R^1 , R^2 , R^3 , R^4 , R^5 , R^6 , R^7 und R^8 die in Anspruch 1 angegebene Bedeutung haben, umfassend die Stufen des Umsetzens eines Alkylphenols der Formel (4):



worin R^1 , R^2 , R^3 , R^4 und R^5 die oben genannten Bedeutungen haben, mit einem Epoxid der Formel (3):



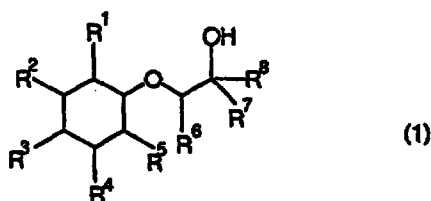
worin R^6 , R^7 und R^8 die oben genannten Bedeutungen haben, in Gegenwart einer Base, um ein α -(Alkylphenyloxy)- β -alkanol der Formel (5) zu erhalten:



worin R^1 bis R^8 die oben genannten Bedeutungen haben, und dann Hydrieren des α -(Alkylphenyloxy)- β -alkanols in Gegenwart eines Metalkatalysators.

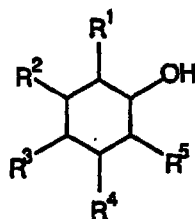
Patentansprüche für folgenden Vertragsstaat : ES

1. Verfahren zum Herstellen eines α -(Alkylcyclohexyloxy)- β -alkanols der Formel (1):



worin mindestens einer der Substituenten R^1 , R^2 , R^3 , R^4 und R^5 eine tertiäre Alkylgruppe mit 4 bis 8 Kohlenstoffatomen ist, und die übrigen R^1 -, R^2 -, R^3 -, R^4 - und R^5 -Gruppen Wasserstoffatome oder Methylgruppen sind, und worin R^6 , R^7 , und R^8 Wasserstoffatome oder die gleichen oder verschiedene Alkylgruppen mit 1 bis 6 Kohlenstoffatomen sind, unter der Bedingung, daß R^6 , R^7 und R^8 nicht alle gleichzeitig Wasserstoff sein können.

umfassend die Stufen des Umsetzens eines Alkylcyclohexanols der Formel (2):



(2)

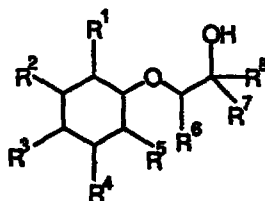
worin R^1 , R^2 , R^3 , R^4 und R^5 die oben genannten Bedeutungen haben, mit einer starken Base und dann Umsetzen des erhaltenen Produktes mit einem Epoxid der Formel (3):



(3)

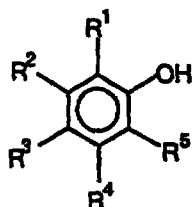
worin R^6 , R^7 und R^8 die oben genannten Bedeutungen haben.

2. Verfahren zum Herstellen eines α -(Alkylcyclohexyloxy)- β -alkanols der Formel (1):



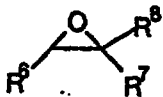
(1)

worin R^1 , R^2 , R^3 , R^4 , R^5 , R^6 , R^7 und R^8 die in Anspruch 1 angegebene Bedeutung haben, umfassend die Stufen des Umsetzens eines Alkylphenols der Formel (4):



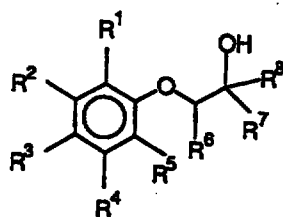
(4)

worin R^1 , R^2 , R^3 , R^4 und R^5 die oben genannten Bedeutungen haben, mit einem Epoxid der Formel (3):



(3)

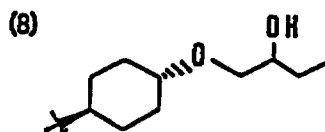
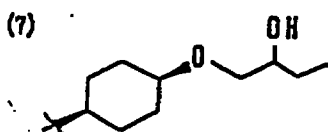
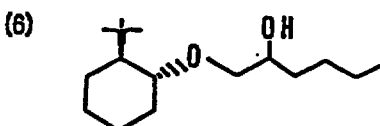
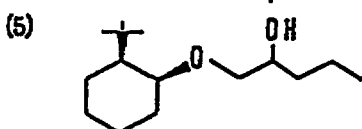
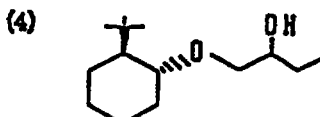
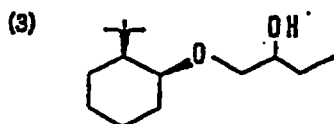
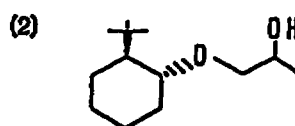
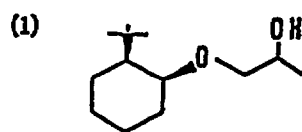
worin R^6 , R^7 und R^8 die oben genannten Bedeutungen haben, in Gegenwart einer Base, um ein α -(Alkylphenyloxy)- β -alkanol der Formel (5) zu erhalten:

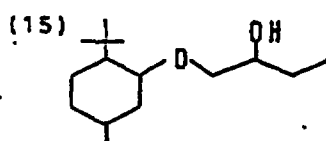
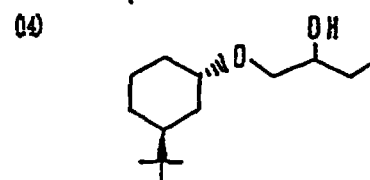
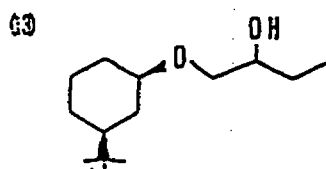
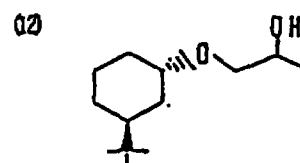
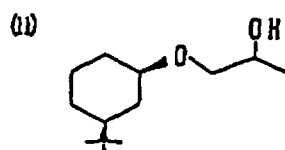
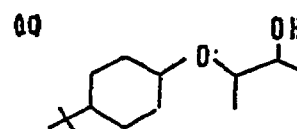
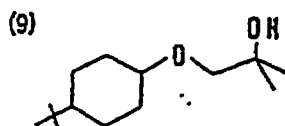


(5)

10 worin R^1 bis R^8 die oben genannten Bedeutungen haben, und dann Hydrieren des α -(Alkylphenoxy)- β -alkanols in Gegenwart eines Metallkatalysators.

3. Verfahren nach Anspruch 1 oder 2, das eine Verbindung der folgenden Formeln (1) bis (15) herstellt:



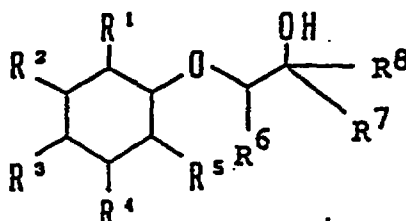


4. Parfüm-Zusammensetzung, enthaltend den in Anspruch 1 beschriebenen α -(Alkylcyclohexyloxy)- β -alkanol in Kombination mit den anderen Komponenten der Zusammensetzung.

Revendications

Revendications pour les Etats contractants suivants : DE, FR, GB, IT

1. α -(Alkylcyclohexyloxy)- β -alkanol représenté par la formule (1) :



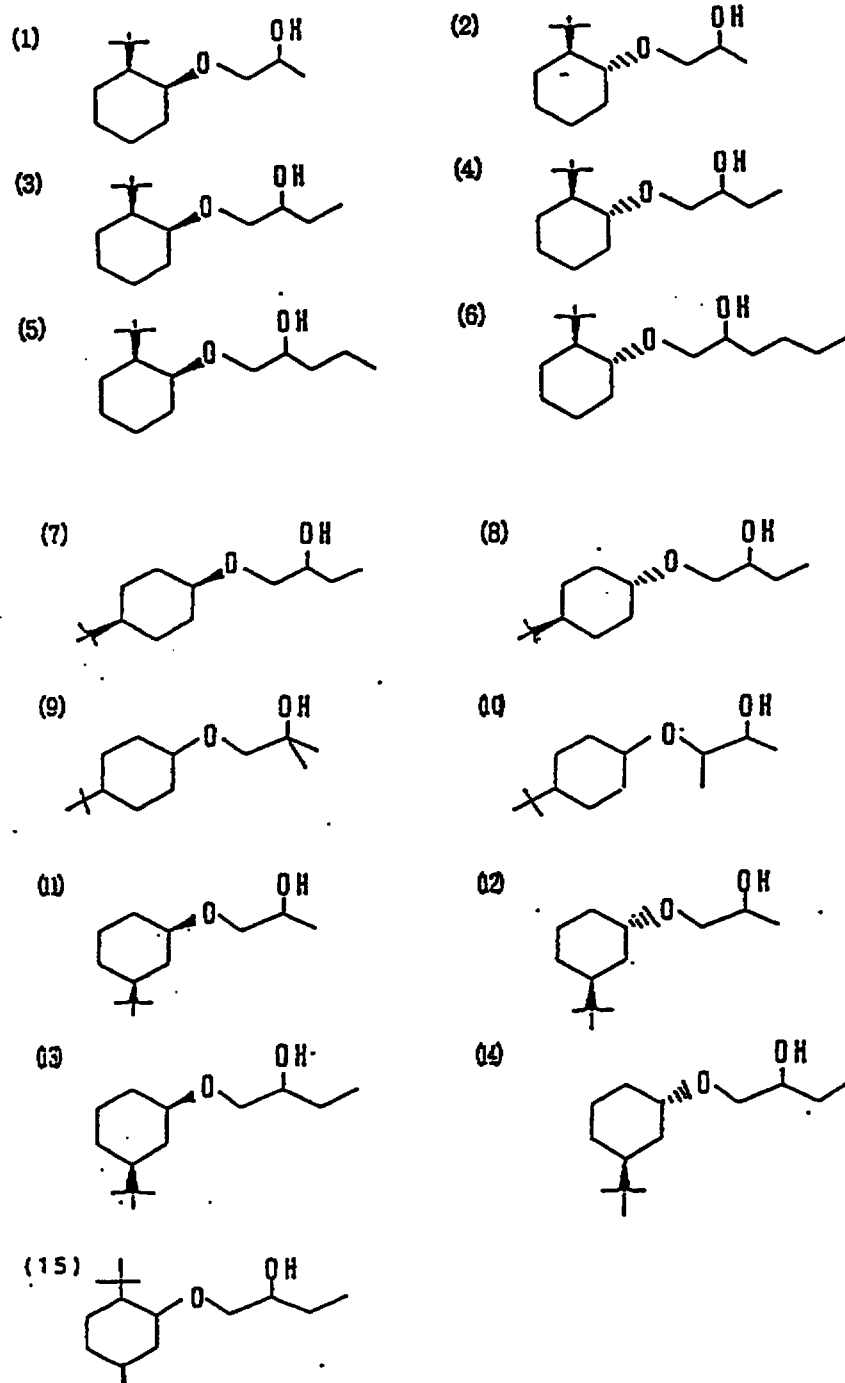
(1)

dans laquelle :

- au moins l'un des substituents R^1 , R^2 , R^3 , R^4 et R^5 représente un groupe alkyle tertiaire, ayant 4-8 atomes de carbone, et les groupes R^1 , R^2 , R^3 , R^4 et R^5 restant représentent des atomes d'hydrogène ou des groupes méthyle ; et
- R^6 , R^7 et R^8 représentent des atomes d'hydrogène ou des groupes alkyle, identiques ou différents, ayant 1-6 atomes de carbones, avec la condition que R^6 , R^7 et R^8 ne représentent pas tous des atomes d'hydrogène en même temps.



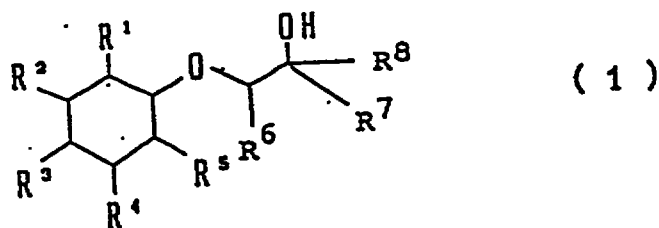
2. Composé selon la revendication 1, lequel composé est :



3. Composition de parfum contenant l' α -(alkylcyclohexyloxy)- β -alcanol décrit à la revendication 1, en combinaison avec les autres composants de la composition.

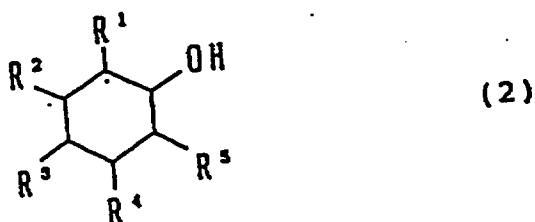


4. Procédé de préparation d'un α -(alkylcyclohexyloxy)- β -alcanol représenté par la formule (1) :



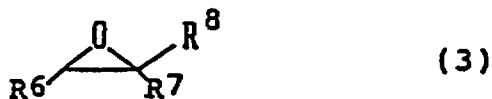
dans laquelle R¹, R², R³, R⁴, R⁵, R⁶, R⁷ et R⁸ sont tels que définis à la revendication 1, qui comprend les étapes consistant à :

- 15
- faire réagir un alkylcyclohexanol de la formule (2) :



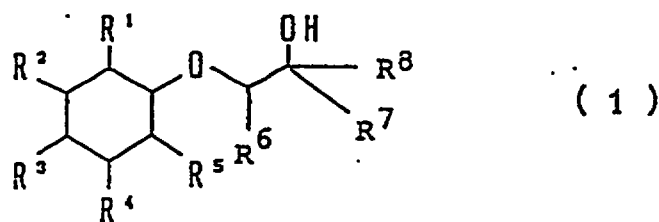
dans laquelle R¹, R², R³, R⁴ et R⁵ ont les mêmes significations que celles définies ci-dessus, avec une base forte ; puis

- 25
- faire réagir le produit obtenu avec un époxyde de la formule (3) :



dans laquelle R⁶, R⁷ et R⁸ ont les mêmes significations que celles définies ci-dessus.

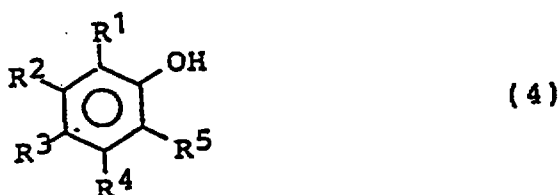
5. Procédé de préparation d'un α -(alkylcyclohexyloxy)- β -alcanol représenté par la formule (1) :



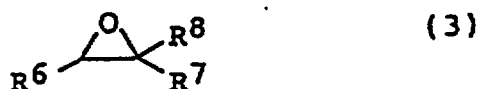
dans laquelle R¹, R², R³, R⁴, R⁵, R⁶, R⁷ et R⁸ sont tels que définis à la revendication 1, qui comprend les étapes consistant à :



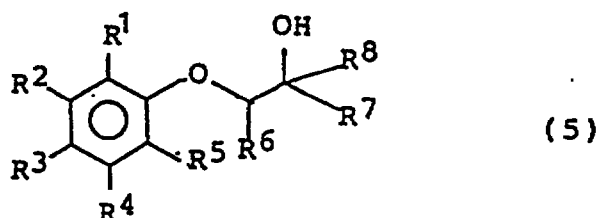
- faire réagir un alkylphénol de la formule (4) :



dans laquelle R¹, R², R³, R⁴ et R⁵ ont les mêmes significations que celles définies ci-dessus, avec un époxyde de la formule (3) :



dans laquelle R⁶, R⁷ et R⁸ ont les mêmes significations que celles définies ci-dessus, en présence d'une base, pour obtenir un α-(alkylphényloxy)-β-alcanol représenté par la formule (5) :

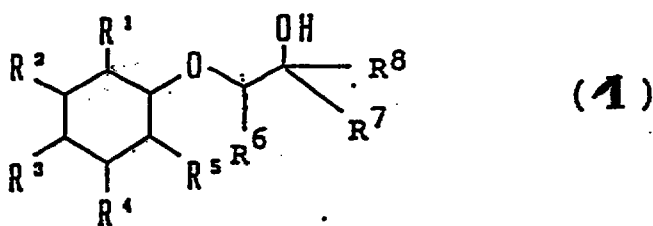


dans laquelle R¹ à R⁸ ont respectivement les mêmes significations que celles définies ci-dessus ; puis

- hydrogéner l'α-(alkylphényloxy)-β-alcanol en présence d'un catalyseur métallique.

35 Revendications pour l'Etat contractant suivant : ES

1. Procédé de préparation d'un α-(alkylcyclohexyloxy)-β-alcanol représenté par la formule (1) :



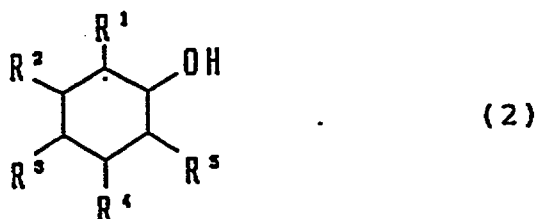
dans laquelle :

- au moins l'un des substituants R¹, R², R³, R⁴ et R⁵ représente un groupe alkyle tertiaire, ayant 4-8 atomes de carbone, et les groupes R¹, R², R³, R⁴ et R⁵ restants représentent des atomes d'hydrogène ou des groupes méthyle ; et
- R⁶, R⁷, et R⁸ représentent des atomes d'hydrogène ou des groupes alkyle, identiques ou différents, ayant 1-6 atomes de carbone, avec la condition que R⁶, R⁷ et R⁸ ne représentent pas tous des atomes d'hydrogène en même temps,

qui comprend les étapes consistant à :

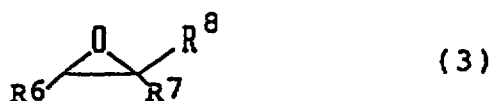


- faire réagir un alkylcyclohexanol de la formule (2) :



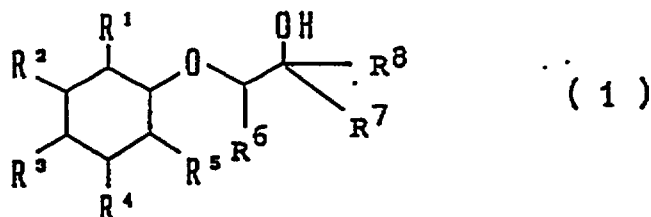
dans laquelle R¹, R², R³, R⁴ et R⁵ ont les mêmes significations que celles définies ci-dessus, avec une base forte ; puis

- faire réagir le produit obtenu avec un époxyde de la formule (3) :



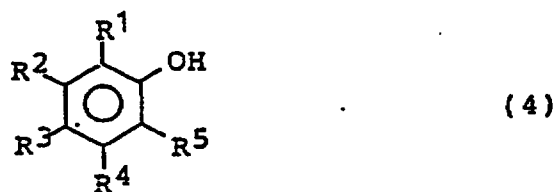
dans laquelle R⁶, R⁷, et R⁸ ont les mêmes significations que celles définies ci-dessus.

2. Procédé de préparation d'un α-(alkylcyclohexyloxy)-β-alcanol représenté par la formule (1) :

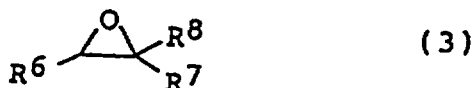


dans laquelle R¹, R², R³, R⁴, R⁵, R⁶, R⁷ et R⁸ sont tels que définis à la revendication 1, qui comprend les étapes consistant à :

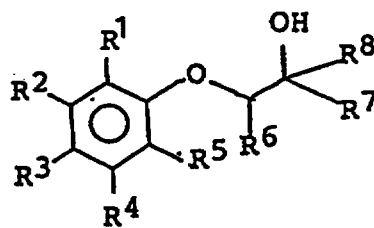
- faire réagir un alkylphénol de la formule (4) :



dans laquelle R¹, R², R³, R⁴ et R⁵ ont les mêmes significations que celles définies ci-dessus, avec un époxyde de la formule (3) :



dans laquelle R⁶, R⁷, et R⁸ ont les mêmes significations que celles définies ci-dessus en présence d'une base, pour obtenir un α-(alkylphényloxy)-β-alcanol représenté par la formule (5) :



(5)

- 10 dans laquelle R¹ à R⁸ ont respectivement les mêmes significations que celles définies ci-dessus ;
puis
- hydrogéner l' α -(alkylphényloxy)- β -alcanol en présence d'un catalyseur métallique.

15

20

25

30

35

40

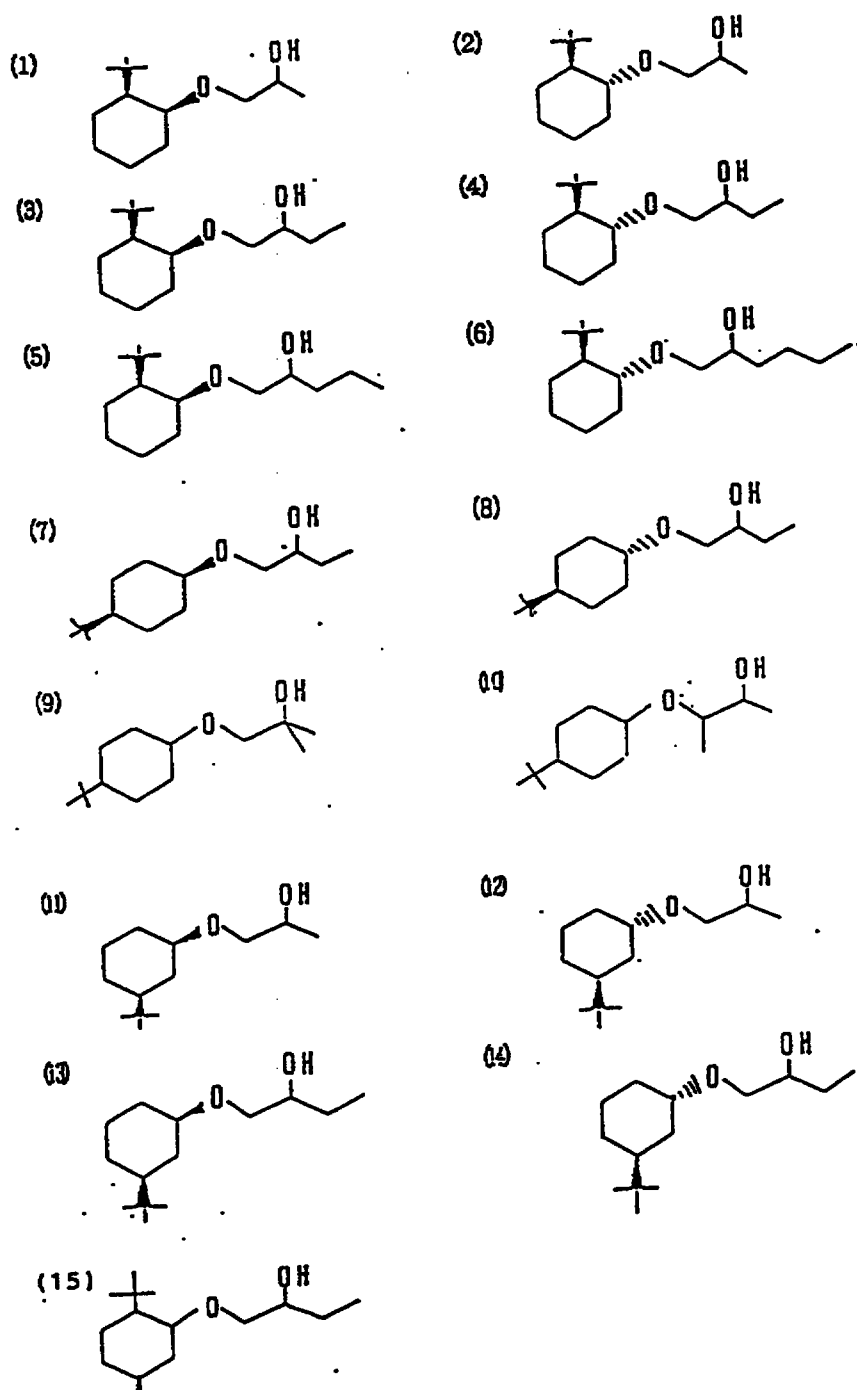
45

50

55



3. Procédé selon la revendication 1 ou 2, qui produit un composé des formules (1) à (15) suivantes :



4. Composition de parfum contenant l' α -(alkylcyclohexyloxy)- β -alcanol décrit à la revendication 1, en combinaison avec les autres composants de la composition.