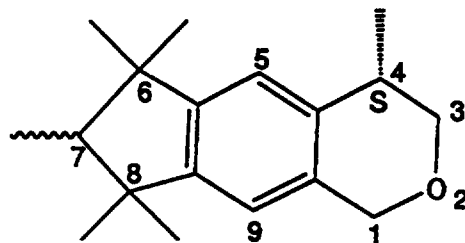




Ref. 6510/262

The invention is concerned with a novel odorant, namely the 7R/7S-diastereoisomer mixture of the formula

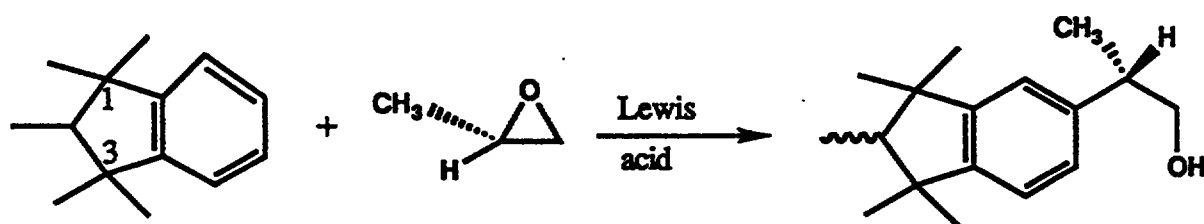
5



in pure form.

10 The invention is also concerned with odorant compositions containing I and with the use of I as an odorant.

Mixture I is accessible according to the following Scheme:

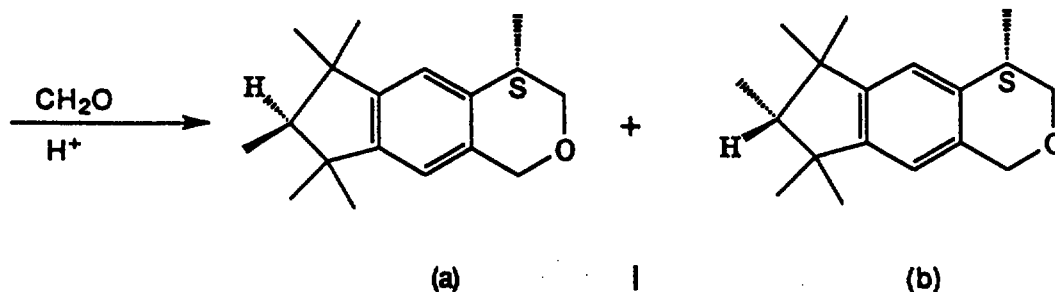


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III

IV

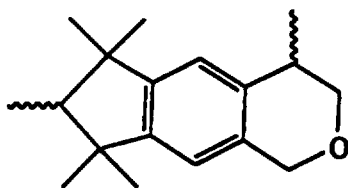
II



20

The isochromane ring closure of II is conveniently carried out in a manner known per se, e.g. using formaldehyde or a synthetic analogue thereof, e.g. the acetal, especially the dimethyl or diethyl acetal or also dihexyl acetal.

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I'

see S. Arctander, Perfume and Flavor Chemicals I, Montclair, N.J. 1969, item 1581,

5 its use is very much preferred, which will be immediately evident from the following comparison of all corresponding olfactory threshold values:

(4S)-cis isomer	0.3 ng/l	air	I (b)	] I'
(4S)-trans isomer	0.6 ng/l	air	I (a)	
(4R)-cis isomer	200 ng/l	air		
(4R)-trans isomer	400 ng/l	air.		

10

By using I the incorporation, entrainment of about 50%, of unnecessary and completely functionless ballast is avoided in all cases. The term "mixture I in pure form" thus serves in the present case to distinguish this mixture I from the commercial product of formula I' above.

15

In other respects, the mixture I can be used as an odorant in exactly the same manner as the known isochroman musk substance, the indene I', although corresponding to the above olfactory threshold values lower concentrations are required.

20

Example

a) 7.5 g of $AlCl_3$ are added to a solution, cooled to -10°C, of 26.2 g of 1,1,2,3,3-pentamethylindane in 15 ml of CS_2 . 2.4 g of S (-)-propylene oxide dissolved in 2.5 ml of CS_2 are now slowly added to the yellow-brown mixture at -5°C to -3°C. After completion of the addition the mixture is stirred at 0°C for a further 30 minutes and then added to ice-water. The working up is effected by taking up in a mixture of hexane-tert.butyl methyl ether in the ratio 1:1.

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The crude product is firstly distilled at 180-200°C and at 0.1 mm Hg, and then purified further by crystallization, namely by crystallization from hexane.

5

The melting point of the product (2S,2'R,S)-2-1',1',2',3',3'-pentamethylindan-5'-yl-propan-1-ol is 73-74°C, $[\alpha]_D^{22}$ (C₂H₅OH, c = 1.0) -6.23°.

- 10 b) The 2-indan-5'-yl-propan-1-ol obtained under a) is treated with paraformaldehyde, 1-hexanol and phosphoric acid exactly according to the procedure of Example 2 of US Patent 3-910 984 and the product, namely (4S,7RS)-1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[g]-2-benzopyran,
- 15 is obtained in good yield. $[\alpha]_D^{22}$ (C₂H₅OH, c = 1.18) +21.4°.

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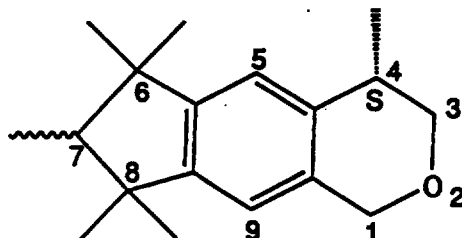


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Claims

1. The 7R/7S-diastereoisomer mixture of the formula

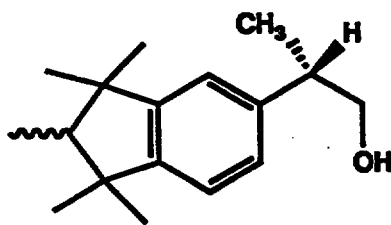


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in pure form.

2. The 2R/2S-diastereoisomer mixture of the formula

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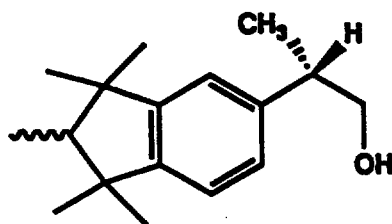


II

in pure form.

3. An odorant composition, which contains the diastereoisomer mixture of formula I according to claim 1.

4. A process for the manufacture of the diastereoisomer mixture of formula I according to claim 1, which process comprises subjecting the 2R/2S-diastereoisomer mixture of the formula



II

- to an isochromane ring closure.

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5. The use of the diastereoisomer mixture of formula I according to claim 1 as an odorant.

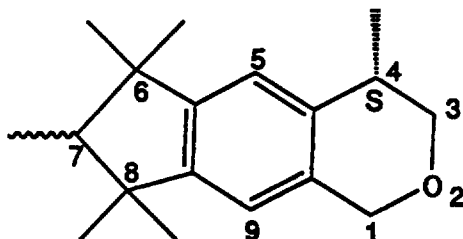
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Abstract

5 The invention is concerned with a novel odorant, namely the
7R/7S-diastereoisomer mixture of the formula



10 in pure form.

 The invention is also concerned with odorant compositions
containing I and with the use of I as an odorant.
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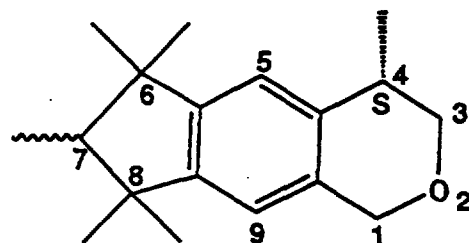
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INDAN

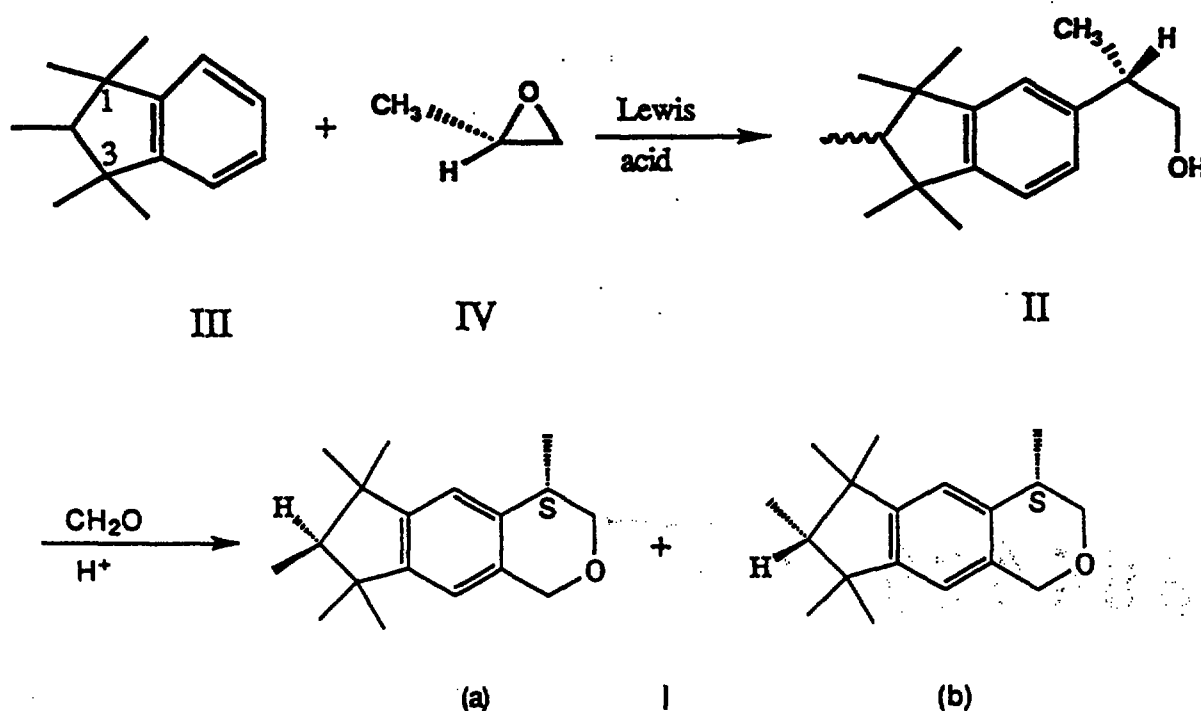
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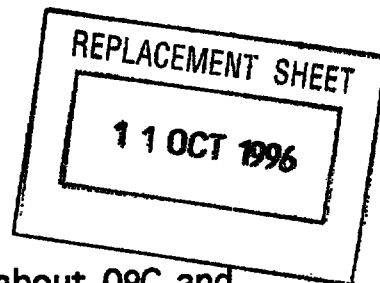
in pure form.

The invention is also concerned with odorant compositions containing I and with the use of I as an odorant.

Mixture I is accessible according to the following Scheme:



The isochromane ring closure of II is conveniently carried out in a manner known per se, e.g. using formaldehyde or a synthetic analogue thereof, e.g. the acetal, especially the dimethyl or diethyl acetal or also dihexyl acetal.



The temperature conveniently lies between about 0°C and about 200°C, preferably between about 85°C and about 150°C.

Catalyst: a protonic acid, for example p-toluenesulphonic acid or phosphoric acid, etc.

Acid concentration: 1 to 100% (wt./wt.) of the amount of II:

Ratio II:CH₂O: about 0.1-about 1.

The acetal can also be prepared in situ from CH₂O and a lower alkanol.

Conditions which are well-known also apply to the Friedel-Crafts reaction of III to II, for example:

Lewis acid: especially AlCl₃, but also SnCl₄, SnBr₄, TiCl₄, BCl₃, etc.

Temperature range: about -30° C for about +30°C, especially about -20° C for about -0°C.

Solvent: saturated aliphatic, optionally halogenated hydrocarbon, for example pentane or hexane, etc., but also the usual, optionally halogenated aromatics, or also CS₂.

Ratio solvent to III: about 1:10 to about 10:1.

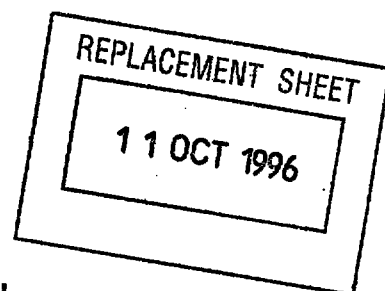
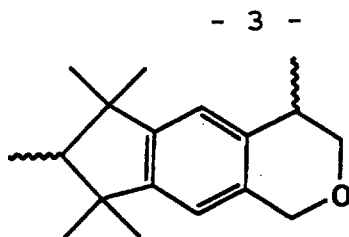
Ratio catalyst:IV: about 1:1 to about 2:1.

Ratio III:IV about 1:1 to about 10:1.

Working up: addition of an alkanol or aliphatic ether or also mixtures of such solvents, etc. to the reaction mixture.

The diastereoisomer mixture I has very surprising properties.

Compared with the commercial mixture of the two racemates of the formula



see S. Arctander, Perfume and Flavor Chemicals I, Montclair, N.J. 1969, item 1581,
its use is very much preferred, which will be immediately evident from the following comparison of all corresponding olfactory threshold values:

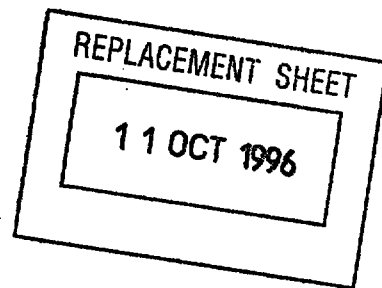
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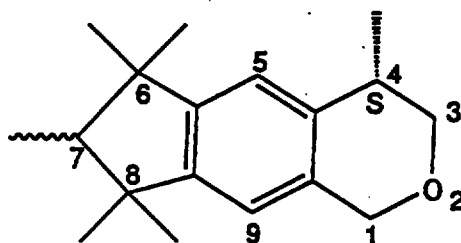
b) The 2-indan-5'-yl-propan-1-ol obtained under a) is treated with paraformaldehyde, 1-hexanol and phosphoric acid exactly according to the procedure of Example 2 of US Patent 3-910 984 and the product, namely (4S,7RS)-1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethyl-cyclopenta[g]-2-benzopyran, is obtained in good yield. $[\alpha]_D^{22}$ (C₂H₅OH, c = 1.18) +21.4°.



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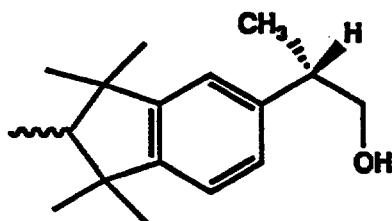
Claims

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in pure form.

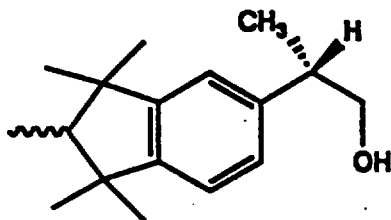
2. The 2R/2S-diastereoisomer mixture of the formula



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to an isochromane ring closure.



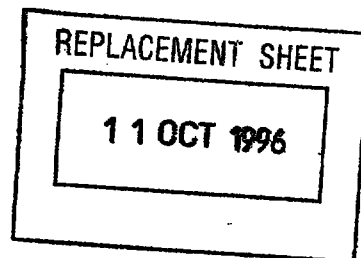
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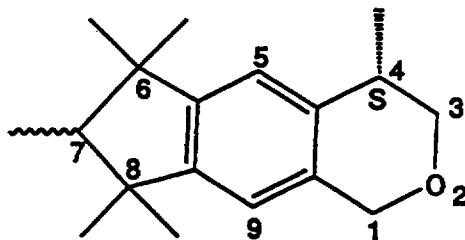
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INDAN



Abstract

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