



(51) International Patent Classification:

A23L 1/22 (2006.01) *A61Q 11/00* (2006.01)
A23L 1/226 (2006.01) *A23G 4/06* (2006.01)
A23L 2/56 (2006.01)

(21) International Application Number:

PCT/IB2011/051665

(22) International Filing Date:

18 April 2011 (18.04.2011)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

10004725.7 5 May 2010 (05.05.2010) EP

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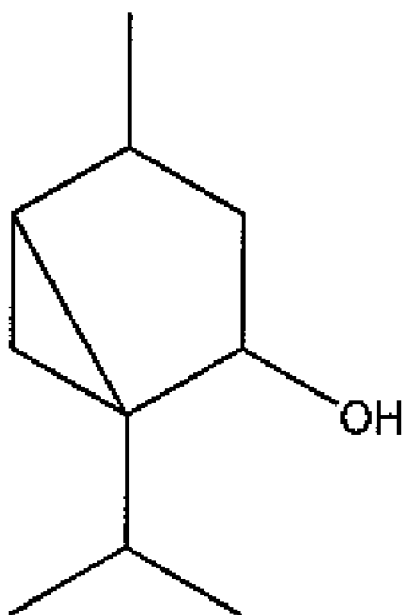
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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

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

[Continued on next page]

(54) Title: COMPOUND HAVING A COOLING EFFECT



(I)

(57) Abstract: The present invention relates to the field of taste-modifiers. More particularly, it concerns the use of certain compounds of Formula (i) as taste-modifiers to impart or reinforce a cooling effect. Furthermore, the present invention concerns also the compositions or articles containing at least one such compound.



Published:

- *without international search report and to be republished
upon receipt of that report (Rule 48.2(g))*

COMPOUND HAVING A COOLING EFFECT

Technical field

The present invention relates to the field of taste-modifiers. More particularly, it
5 concerns the use of certain compounds as taste-modifiers to impart or reinforce a cooling effect.

Furthermore, the present invention concerns also the compositions or articles containing at least one such compound.

Prior art

Dihydroumbellulol (1-isopropyl-4-methylbicyclo[3.1.0]hexan-2-ol, also known under the name 2-thujanol) is described in a number of prior art documents as the product obtained by reduction of dihydroumbellulone (1-isopropyl-4-methylbicyclo[3.1.0]hex-3-en-2-one). For example, In Wienhaus, H. and Todenhofer, K.; *Umbellulone and*
15 *umbellularia oil*, in Schimmel's Report (1929), pp 285-295, dihydroumbellulol is obtained by reduction of dihydroumbellulone. The odor of dihydroumbellulol is described as resembling that of isomenthol, but no taste characteristics of this compound are mentioned in this document. Furthermore, isomenthol is not described in S. Arctander, *Perfume and Flavor Chemicals*, 1969, Montclair, New Jersey, USA, n°1842 as a possible
20 replacement of leavo-menthol as regards its taste properties. It is only described in perfumery where a fresh non-citrusy and non-woody "lift" may be desirable.

Other synthetic ways to obtain dihydroumbellulol are described in the following documents : Klein, E. and Rojahn, W., *The photosensitized O₂ transfer to (+)- α -thujene*, in *Chemische Berichte* (1965), 98(9), pp. 3045-3049 and Holden, C.; Rees, J.C.; Scott, S.P. and Whittaker, D., *Stereochemistry of reduction of umbellulone (thuj-3-en-2-one) and isodihydroumbellulone (4 β H-thujan-2-one)*, in *Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry* (1976), pp. 1342-1345.
25

Dihydroumbellulol has been identified as a natural compound in the essential oil of *hyptis pectinata* (Malan, K.; Pelissier, Y.; Marion, C.; Blaise, A.; and Bessiere, J.M.,
30 *The essential oil of Hyptis pectinata*, in *Planta Medica* (1988), 54(6), pp 531-532). This document suggests antibacterial, antimycotic and antitussive properties of the essential oil as a whole, but it completely silent about the specific properties of dihydroumbellulol.

To the best of our knowledge, no prior art document reports or suggests that dihydroumbellulol has taste modifier properties and in particular that it is capable of imparting or reinforcing a cooling effect, which is highly desirable for many edible and oral care products.

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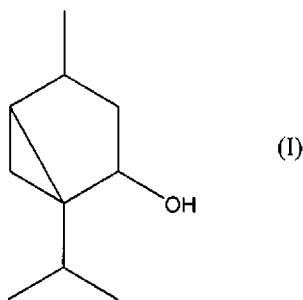
Description of the drawings

Figure 1 represents the results of the sensory analysis carried out to assess the cooling effect of dihydroumbellulol at 50ppm and 100ppm in a mixture of water and ethanol (Example 2).

10

Description of the invention

We have now surprisingly discovered that a compound of formula



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in the form of any one of its stereoisomers or of a mixture thereof, can be used as a taste-modifier, preferably to impart or reinforce the cooling effect of a flavouring composition or of a flavoured article.

According to a preferred aspect of the invention, the compound of formula (I) is in the (1R,2R,4S), (1R,2S,4S), (1R,2R,4R), (1R,2S,4R) configuration or a mixture thereof.

As mentioned above, the invention concerns the use of a compound of formula (I) as taste modifier to impart a cooling effect. Typically, for the purpose of the present invention, the "cooling effect" of a compound is defined as the physiological perception of a refreshing cool or cold sensation when such compound comes into contact with the human body and, in particular, with the mucous membranes of the mouth, nose and throat and preferably those of the mouth and throat. In a preferred aspect of the invention, this cooling effect is achieved by activation of cold trigeminal receptors in the mouth and/or throat, and more preferably by activation of TRPM8 receptors.

In other words it concerns a method to confer, enhance, improve or modify the taste properties, as indicated above, of a flavouring composition or of a flavoured article, which method comprises adding to said composition or article an effective amount of at least a compound of formula (I). The phrase "use of a compound of formula (I)" means
5 also the use of any composition containing the compound of formula (I) and which can be advantageously employed in the flavour industry as active ingredients.

Said compositions, which in fact can be advantageously employed as flavouring compositions having a taste modifying activity as described above, are also an object of the present invention.

10 Therefore, another object of the present invention is a flavouring composition comprising:

- i) as taste-modifier, at least one compound of formula (I) as defined above;
- ii) at least one ingredient selected from the group consisting of a flavour carrier and a flavour base; and
- 15 iii) optionally at least one flavour adjuvant.

According to a particular embodiment of the invention, said flavouring composition comprises:

- i) as taste-modifier, at least one compound of formula (I) as defined above;
- ii) a flavour base; and
- 20 iii) optionally at least one ingredient selected from the group consisting of a flavour carrier and flavour adjuvant.

By "flavour carrier" we mean here a material which is practically neutral from a flavour perspective, i.e. which does not significantly alter the organoleptic properties of flavouring ingredients. Said carrier may be a liquid or a solid.

25 As liquid carrier one may cite, as non-limiting examples, an emulsifying system, i.e. a solvent and a surfactant system, or a solvent commonly used in flavours. A detailed description of the nature and type of solvents commonly used in flavour cannot be exhaustive. However, one can cite as non-limiting example solvents such as propylene glycol, triacetine, triethyl citrate, benzylic alcohol, ethanol, vegetal oils or terpenes.

30 As solid carrier one may cite, as non-limiting examples, absorbing gums or polymers, or yet encapsulating materials. Examples of such materials may comprise wall-forming and plasticizing materials, such as mono, di- or trisaccharides, natural or

modified starches, hydrocolloids, cellulose derivatives, polyvinyl acetates, polyvinylalcohols, proteins or pectins, or yet the materials cited in reference texts such as H. Scherz, Hydrokolloids : Stabilisatoren, Dickungs- und Geliermittel in Lebensmittel, Band 2 der Schriftenreihe Lebensmittelchemie, Lebensmittelqualität, Behr's Verlag GmbH & Co., Hamburg, 1996. Encapsulation is a well known process to a person skilled in the art, and may be performed using any one of the diverse techniques known in the art, such as, for example, spray-drying, agglomeration, extrusion, coating encapsulation and coacervation.

In the context of the present invention, "flavour base" means a composition comprising at least one flavouring co-ingredient.

The flavouring co-ingredient is not a compound of formula (I). In the context of the present invention, "flavouring co-ingredient" means a compound, which is used in flavouring preparations or compositions to impart a hedonic effect. In other words such a co-ingredient, to be considered as being a flavouring one, must be recognized by a person skilled in the art as being able to impart or modify in a desired way the flavour of a composition, and not just as having organoleptic properties.

The nature and type of the flavouring co-ingredient(s) present in the base do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled person being able to select them on the basis of his general knowledge and according to intended use or application and the desired organoleptic effect. In general terms, flavouring co-ingredients belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpenoids, nitrogenous or sulphurous heterocyclic compounds and essential oils, and said co-ingredients can be of natural or synthetic origin. Many of these co-ingredients are in any case listed in reference texts such as the book by S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, New Jersey, USA, or its more recent versions, or in other works of a similar nature, as well as in the abundant patent literature in the field of flavour. It is also understood that said co-ingredients may also be compounds known to release in a controlled manner various types of flavouring compounds.

A flavouring composition comprising at least one compound of formula (I) may also comprise a flavour adjuvant. In the context of the present invention "flavour adjuvant" means an ingredient capable of imparting additional added benefit such as a

color, a particular light resistance, chemical stability, etc. A detailed description of the nature and type of adjuvant commonly used in flavouring bases cannot be exhaustive, but it has to be mentioned that said ingredients are well known to a person skilled in the art.

A flavouring composition comprising at least one compound of formula (I) and at least one flavour carrier represents a particular embodiment of the invention as well as a flavouring composition comprising at least one compound of formula (I), at least one flavour carrier, at least one flavour base, and optionally at least one flavour adjuvant.

Preferably, any mixture resulting directly from a chemical synthesis, e.g. without an adequate purification, in which the compound of the invention would be involved as a starting, intermediate or end-product could not be considered as a flavouring composition according to the invention.

A compound of formula (I) can be advantageously incorporated into flavoured articles to impart a taste to, or modify the taste of said articles. Preferably the compound of formula (I) is incorporated into the flavoured article to impart a cooling effect to or modify the cooling effect of the product. Consequently, the invention also relates to a flavoured article comprising:

- i) as taste-modifier, at least one compound of formula (I), as defined above, or a flavouring composition comprising at least one compound of formula (I); and
- ii) an edible base.

An "edible base" is intended here as an edible product such as a food, a beverage, an oral care product, a tobacco product or a pharmaceutical product. Beverages, iced products such as ice creams or sorbets, sweets, confectionery, dairy products, chewing gums, jams, savoury products, cigarettes, chewing tobacco, pharmaceutical preparations and oral care products such as dental care products are non-limiting examples of suitable edible bases.

Preferred edible bases for the purpose of the present invention are beverages, in particular emulsion-based drinks, fruit juices, soft drinks, cold teas and flavoured waters, chewing gums with and without sugar, sweets with and without sugar, pressed tablets, gelatin-based confectionery, fruit jams and jellies, alcoholic beverages, sorbets, dairy ice creams, tooth gels and toothpastes, mouth-washes and throat-washes.

The nature and type of the constituents of the edible products do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled

person being able to select them on the basis of his general knowledge and according to the nature of said product.

The proportions in which the compounds of formula (I) can be incorporated into the various aforementioned articles or compositions vary within a wide range of values.

- 5 These values are dependent on the nature of the article of which the taste is intended to be modified, as well as the nature of the co-ingredients in a given base when the compound according to the invention is mixed with flavouring co-ingredients, solvents or additives commonly used in the art.

- 10 In the case of taste-modifying compositions, typical concentrations are in the order of 0.0001 % to 1 % by weight of the compound according to formula (I), based on the weight of the consumer product into which they are incorporated. Concentrations below the abovementioned range, such as in the order of 0.001% to 0.5% by weight, can be used when these compounds are incorporated into flavoured articles, percentage being relative to the weight of the article.

- 15 Compounds of formula (I) can be synthesized using any method known in the art and in particular by hydrogenation of dihydroumbellulone, as described in the documents cited above.

Examples

- 20 The invention will now be described in further detail by way of the following examples, wherein the abbreviations have the usual meaning in the art. In all of the following examples, dihydroumbellulol is provided by Firmenich SA, Geneva, Switzerland.

Example 1

Evidence of the cooling effect of dihydroumbellulol

- (1R,2R,4S)-dihydroumbellulol, (1R,2S,4S)-dihydroumbellulol, (1R,2R,4R)-dihydroumbellulol and (1R,2S,4R)-dihydroumbellulol, respectively, were dissolved in a 2.5 g/L solution of ethanol in water, to form a 50 mg/L solution of the compound.
- 30 Each sample was then tasted by a group of 8 experienced panelists. All four samples were described as having a cooling effect.

Example 2

Intensity of the cooling effect of dihydroumbellulol

In a second experiment, the perceived intensity of the cooling effect of dihydroumbellulol was evaluated in a blind test. Given the very similar results obtained with the four samples tested in Example 1, only one isomer ((1R,2R,4S)-dihydroumbellulol) was tested.

A control was prepared consisting of a 2.5 g/L ethanol solution in water (Control 1). Two test samples were then prepared by dissolving dihydroumbellulol in the ethanol-water solution at a concentration of 50ppm (Sample 1) and 100ppm (Sample 2), respectively.

The samples were presented in a random manner on a blind basis to a group of 25 trained panelists. The control was presented three times (Controls 1A, 1B and 1C), Sample 1 was presented once and Sample 2 was presented twice (Sample 2A and 2B). For each sample, the panelists were requested to taste the sample and to continuously rate the intensity of the cooling effect perceived after consumption on a scale ranging from 0 to 10, where 0 means no cooling effect and 10 means very strong cooling effect.

The results are summarized in Figure 1. The average perceived intensity of the cooling effect was clearly stronger with dihydroumbellulol (Samples 1 and 2) than in the controls. Furthermore, the intensity of the perception of the cooling effect was clearly stronger with 100ppm of dihydroumbellulol (Samples 2A and 2B) than with 50ppm (Sample 1). The perception of the cooling effect was also prolonged with Samples 1 and 2 containing dihydroumbellulol compared to the control. This long-lasting effect also increased with the concentration of dihydroumbellulol since a longer effect was perceived with Samples 2A and 2B than with Sample 1. These results also showed that the cooling effect was perceived slightly earlier with dihydroumbellulol than with the control.

Example 3

Sugar free chewing gum comprising dihydroumbellulol

An unflavoured chewing gum base was prepared having the following ingredients :

Table 1 : Composition of the sugar free chewing gum base

Ingredient	Amount (wt%)
Solsona T Gum Base ¹⁾	12.44
Vega Gum Base ²⁾	12.44

Crystalline sorbitol	56.50
Maltitol Syrup	11.50
Glycerin	6.92
Aspartame	0.12
Acesulfame K	0.08

- 1) Origin Cafosa
- 2) Origin Cafosa

A Sigma-blade mixer was pre-heated to 45°C-50°C and half of the crystalline sorbitol and of the maltitol syrup was added. The gum base was pre-heated to 60°C-65°C and added to the mixer. Mixing was carried out for approximately 4 minutes. Finally, the remaining crystalline sorbitol and maltitol syrup, sweeteners and humectants were added and mixing was continued for 4 minutes. This unflavoured chewing gum base was used as a control (Control 2).

To prepare a test sample (Sample 3), dihydroumbellulol was added to a portion of unflavored gum base and mixed for approximately 2 minutes in the Sigma-blade mixer to ensure a homogeneous distribution of dihydroumbellulol within the base. The final concentration of dihydroumbellulol was 0.5% by weight, relative to the total weight of the sample.

The final chewing gums (Control 2 and Sample 3) were laminated and cut into sticks or slabs.

Control 2 and Sample 3 were then evaluated by 8 trained panelists on blind basis. The panelists were asked to chew the chewing gum for 3 minutes and to rate the intensity of the cooling effect of the control and the sample on a scale ranging from 0 to 5, where 0 means no cooling effect and 5 means very strong cooling effect.

The cooling effect of Control 2 was rated at 0 by all panelists, whereas the average rating of the cooling effect intensity of Sample 3 was 2.45. The panelists reported a mild cooling effect of Sample 3, due to the presence of dihydroumbellulol.

25

Example 4

Toothpaste comprising dihydroumbellulol

An unflavored toothpaste base was prepared having the following ingredients :

Table 2 : Composition of the unflavored toothpaste base

Ingredient	Amount (wt%)
Xanthan Gum ¹⁾	0.90
Sorbitol ²⁾	40.0
Sodium fluoride ³⁾	0.22
Sodium Saccharin	0.20
Sodium Benzoate	0.30
Water demineralised	27.94
Hydrated silica ⁴⁾	22.00
Hydrated silica ⁵⁾	5.00
Titanium dioxide	0.50
Sodium lauryl sulfate ⁶⁾	1.26

1) Rhodicare[®] S, origin: Rhodia

5 2) Neosorb[®] 70/70, origin: Roquette Frères

3) Origin: Riedel-deHaën

4) Tixosil[®] 73, origin: Rhodia

5) Tixosil[®] 43, origin: Rhodia

6) Texapon[®] K 12 P, origin: Cognis GmbH

10

The xanthan gum was first dispersed in liquid sorbitol under vigorous stirring in an homogenizer IKA Euro-ST D and the resulting dispersion was transferred into a Mixer for toothpaste ESCO-LABOR EL-1 (Esco Labor AG, Riehen/Switzerland). Then, a solution was prepared by solubilizing the fluoride salt, sodium saccharin and sodium benzoate in water and mixed for 10 minutes. This solution was then added to the dispersion of the gum in sorbitol. The mixture was stirred continuously during about 25 minutes until the gum was fully hydrated to obtain a homogeneous gel. Both hydrated silica and titanium dioxide were then progressively added to the gel and the resulting paste was mixed gently during 40 minutes. Finally, vacuum was applied and sodium lauryl sulfate was added. The final product was stirred again during about 5 minutes under vacuum until full de-aeration was achieved. The unflavoured toothpaste was used as Control (Control 3).

20

To prepare a test sample, a 50% solution of dihydroumbellulol in propylene glycol was

prepared and added to the toothpaste base described above and the resulting toothpaste was mixed until a homogeneous distribution of dihydroumbellulol in the paste was achieved (Sample 4). The final concentration of dihydroumbellulol in Sample 4 was 0.3% by weight, relative to the total weight of the sample.

- 5 The final toothpastes (Control 3 and Sample 4) were transferred into a tube and the tube was sealed.

Control 3 and Sample 4 were then evaluated by 9 trained panelists on a blind basis. The panelists were requested to brush their teeth using 1g of toothpaste during 1 minute and to rinse their mouth with water. Just after rinsing their mouth, they were asked to rate the cooling intensity of Control 3 and Sample 4 on a scale ranging from 0 to 5, where 0 means
10 no cooling effect and 5 means very strong cooling effect.

The cooling effect of Control 3 was rated at 0 by all panelists, whereas the average rating of the cooling effect intensity of Sample 4 was 1.55. The panelists reported a mild cooling effect of Sample 4 on gums and cheeks, due to the presence of dihydroumbellulol.

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Example 5

Mouthwash comprising dihydroumbellulol

An unflavored mouthwash base (without alcohol) was prepared having the following ingredients :

20

Table 3 : Composition of the unflavoured mouthwash base

Ingredient	Amount (wt%)
Water demineralised	82.68
Sodium monofluorophosphate	0.05
Sodium Saccharin	0.03
Sodium Benzoate	0.20
Sorbitol 70% ¹⁾	8.02
Glycerin	8.02
Glycerol-polyethylene glycol oxystearate ²⁾	1.00

1) Neosorb[®] 70/70, origin Roquette Frères

2) Cremophor[®] RH40 (Registered trademark of BASF), origin: Firmenich, Geneva,
25 Switzerland (item n° 936037)

Under magnetic stirring, sodium monofluorophosphate, sodium saccharin and sodium benzoate were added to the water, without heating. Then, sorbitol was added, followed by glycerin. The resulting solution was mixed for 10 minutes until the solutes were well dissolved. Cremophor[®] was added at the end. The thus obtained unflavoured mouthwash was used as control (Control 4).

To prepare a test sample, a 50% solution of dihydroumbellulol in propylene glycol was prepared, added to the mouthwash base described above and the resulting solution was mixed until homogeneous distribution of dihydroumbellulol in the mouthwash is achieved (Sample 5). The final concentration of dihydroumbellulol in Sample 5 was 0.05% by weight, relative to the total weight of the sample.

Control 4 and Sample 5 were then evaluated by 9 trained panelists on a blind basis. The panelists were requested to rinse their mouth during 30 seconds and then spit out the mouthwash. Just after spitting the mouthwash, they were asked to rate the cooling intensity of Control 4 and Sample 5 on a scale ranging from 0 to 5, where 0 means no cooling effect and 5 means very strong cooling effect.

The average rating of the cooling effect intensity of Control 4 was 0.11, whereas the cooling effect intensity of Sample 5 was 3.11. The panelists reported a mild, noticeable cooling effect of Sample 5, due to the presence of dihydroumbellulol.

20

Example 6

Lemon flavoured water comprising dihydroumbellulol

An unflavoured beverage base was prepared having the following ingredients :

25 Table 4 : Composition of the unflavoured beverage base

Ingredient	Amount (wt%)
Water	97.95
Citric acid	0.05
Sugar	2.00

The ingredients were stirred to form a homogeneous beverage base.

One control (Control 5) and two test samples (Sample 6 and Sample 7) were prepared having the following ingredients :

Table 5 : Composition of Control 5 and Samples 6 and 7

Component	Amount in Control 5 (wt%)	Amount in Sample 6 (wt%)	Amount in Sample 7 (wt%)
Unflavoured beverage base	99.90	99.89	99.99
Lemon flavour ¹⁾	0.10	0.10	0.00
Dihydroumbellulol ²⁾	0.00	0.01	0.01

1) Origin: Firmenich SA, Geneva, Switzerland, item n° 505630T

- 5 2) 2% solution of dihydroumbellulol in propylene glycol, origin: Firmenich SA, Geneva, Switzerland

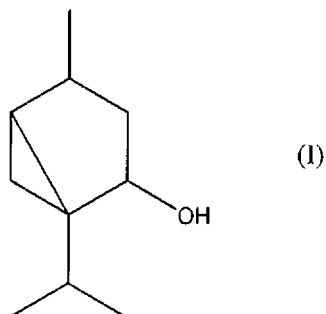
The components of the control and of both samples were stirred to obtain homogeneous beverages.

- 10 Control 5, Sample 6 and Sample 7 were then evaluated by 9 trained panelists on a blind basis. Panelists were requested to taste the three samples and to rate their cooling intensity on a scale ranging from 0 to 5, where 0 means no cooling effect and 5 means very strong cooling effect.

- The average rating of the cooling effect intensity of Control 5 was 0.67. The weak cooling effect observed in the control was probably due to the effect of the temperature of the beverage, which was served cold. Samples 6 and 7 were found more cooling by the panelists, Sample 6 with a cooling intensity average rating of 2.22 and sample 7 with a cooling intensity average rating of 1.8. The slight difference perceived between Samples 6 and 7 is probably due to a minor effect of the lemon flavour. However these results show that dihydroumbellulol is responsible of the main cooling effect perceived in Sample 6 and 7, this compound providing a slight to mild cooling effect in this type of application.
- 15
- 20

Claims

1. Use of a compound of formula



5 in the form of any one of its stereoisomers or of a mixture thereof, as a taste modifier

2. Use according to claim 1, wherein the compound of formula (I) is used to impart or reinforce the cooling effect of a flavouring composition or of a flavoured article.

10 3. Use according to claim 1 or 2, wherein the compound of formula (I) is in the (1R,2R,4S), (1R,2S,4S), (1R,2R,4R), (1R,2S,4R) configuration or a mixture thereof.

4. A flavouring composition comprising:

- 15 i) as taste-modifier, at least one compound of formula (I), as defined in claim 1;
ii) at least one ingredient selected from the group consisting of a flavour carrier and a flavour base; and
iii) optionally at least one flavour adjuvant.

5. A flavoured article comprising:

- 20 i) as taste-modifier, at least one compound of formula (I), as defined in claim 1, or a composition as defined in claim 4; and
ii) an edible base.

25 6. A flavoured article according to claim 5, wherein the edible base is a food, a beverage, an oral care product, a tobacco product or a pharmaceutical product.

7. A flavoured article according to claim 6, wherein the edible base is a beverage, an iced product such as an ice cream or a sorbet, a sweet, a confectionery

product, a dairy product, a chewing gum, a jam, a savoury product, a cigarette, a chewing tobacco, a pharmaceutical preparation or an oral care product such as a dental care product.

- 5 **8.** A flavoured article according to claim 6, wherein the edible base is a beverage, a chewing gum with and without sugar, a sweet with and without sugar, a pressed tablet, a gelatin-based confectionery, a fruit jam or jelly, an alcoholic beverage, a sorbet, a dairy ice cream, a tooth gel, a toothpaste, a mouth-wash or a throat-wash.

Figure 1

Perceived intensity of the cooling effect over time

