

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
24 January 2008 (24.01.2008)

PCT

(10) International Publication Number
WO 2008/009926 A2

(51) International Patent Classification:
C07D 317/62 (2006.01) A23L 1/226 (2006.01)

(21) International Application Number:
PCT/GB2007/002707

(22) International Filing Date: 17 July 2007 (17.07.2007)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/831,230 17 July 2006 (17.07.2006) US
60/828,254 5 October 2006 (05.10.2006) US

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(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH,
CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG,
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IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK,
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TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

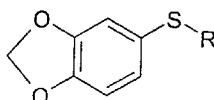
(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),
European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI,
FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL,
PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM,
GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— without international search report and to be republished
upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.

(54) Title: SAFROLE REPLACEMENTS



(I)

(57) Abstract: A compound or mixture of compounds of formula (I): wherein R = straight chain or branched C₁₋₃ alkyl or alkenyl is provided. Methods for their preparation, their use as perfume materials for application to a variety of substrates and their use in flavouring and in articles of manufacture is also provided.



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SAFROLE REPLACEMENTS

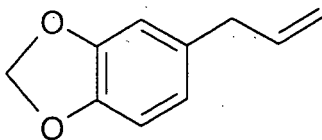
The present invention relates generally to the field of flavours and fragrances. More particularly, the present invention relates to new flavour and fragrance compounds. These compounds find utility in any and all applications requiring flavours and fragrances. The invention also provides mixtures of these compounds, methods for their preparation and their use as perfume materials for application in a variety of substrates and their use in flavouring and articles of manufacture and compositions including the compounds.

10

There are a large number and variety of known flavours and fragrances used as ingredients in perfumes and in a varied range of other products. However, many aromachemicals include double bonds and/or other reactive groups that are potentially susceptible to reaction and may result in a limited useful lifetime. Further, many essential oil fragrances have recently been determined to cause allergic reactions, and it is becoming increasingly difficult to bring products containing such fragrances to market. Some known flavours and fragrances are thought to be carcinogenic.

20 One such compound is Safrole, a phenolic ether obtained primarily from sassafras oil, which is a colourless or slightly yellow liquid, $C_{10}H_{10}O_2$, molecular wt 162.18, with density of 1.096 at 20°C, and melting point about 11°C. The oil is insoluble in water, very soluble in alcohol, miscible with ether and chloroform. The DL_{50} (oral) is 1.950 mg/kg in rats and 2.350 mg/kg in mice (Budavaris 1989). It has the formula:

25



Safrole has a "root beer" type smell, with liquorice and leather notes. Safrole is important to several products. It is used as an ingredient in popular beverages, such as "pinga com sassafras" in Brazil, and was once used as an ingredient for "root beer" in the United States. It has been used as a topical antiseptic and pediculicide. Safrole is an important raw material for the chemical industry

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because of two of its derivatives: heliotropin, which is widely used as a fragrance and flavouring agent, and piperonyl butoxide (PBO), a vital ingredient of pyrethroid insecticides. Natural pyrethrum in particular would not be an economical insecticide without the addition of PBO as a synergist and the future of the natural pyrethrum industry is linked to the continued availability of PBO. Safrole also has many fragrance applications in household products such as floor waxes, polishes, soaps, detergents, and cleaning agents.

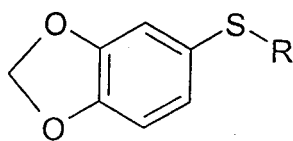
Safrole, however, is strongly suspected to be carcinogenic (see http://www.scorecard.org/chemical-profiles/summary.tcl?edf_substance_id=94-59-7#hazards). It is also used as an ingredient to manufacture Ecstasy, psychoactive drug that affects the brain's use of the naturally-produced chemical serotonin, which regulates mood and aggression. The use of Safrole in many industries has been banned.

There is also a general demand for new flavours and fragrances that have novel or improved fragrance and/or flavour profiles and/or other properties that make them particularly useful for use as fragrances and/or flavours.

The listing or discussion of a prior-published document in this specification should not necessarily be taken as an acknowledgement that the document is part of the state of the art or common general knowledge.

The present invention addresses the foregoing deficiencies by providing the compounds, use, methods, substrate, composition, product, preparation or article, beverage composition, flavouring compositions, food composition, chewing gum composition pharmaceutical composition, orally deliverable matrix and article of manufacture as defined in the claims.

The present invention provides compounds of formula:

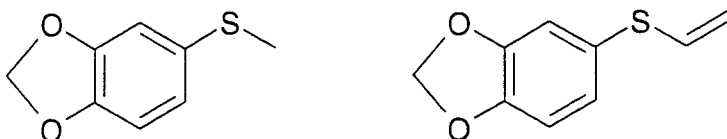


wherein R = straight chain or branched C₁, C₂ or C₃ alkyl or alkenyl. In particular, R may be methyl, ethyl, n-propyl, i-propyl or vinyl (-CH=CH₂). These compounds will be referred to herein as the compounds of the invention.

5

Preferably, R is methyl or vinyl. These compounds, which have the chemical names 5-(methylthio)-1,3-benzodioxole and 5-(vinylthio)-1,3-benzodioxole, are shown below. These compounds are also known as 4-(methylthio)-1,2-(methylenedioxy)benzene and 4-(vinylthio)-1,2-(methylenedioxy)benzene.

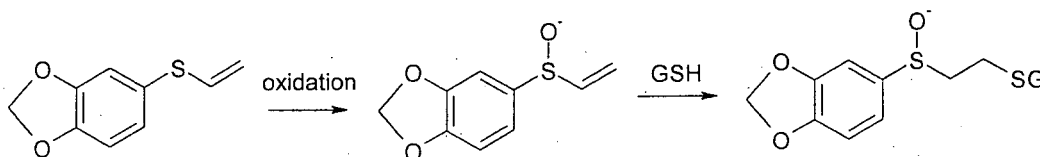
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The compounds of the invention are thought to have Safrole-like flavour and fragrance properties. For example, 4-(methylthio)-1,2-(methylenedioxy)benzene has an odour character which is spicy, leathery and powerful.

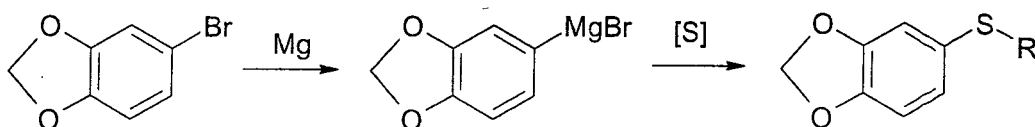
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The compounds of the invention are believed to be non-toxic replacements for Safrole. Alteration of the basic Safrole molecule to remove a benzylic position removes the opportunity for formation of a benzylic/allylic cation, thereby addressing concerns about the potential for the formation of DNA adducts. The metabolism of 4-(vinylthio)-1,2-(methylenedioxy)benzene, for example, should be dominated by the sulfur atom, and it will likely undergo oxidation to the sulfoxide. This substance can be expected to undergo a quasi-1,4-addition with glutathione (GSH, see scheme below), and the resulting mercapturic acid derivative would likely be excreted in the urine. This is common for thiophene derivatives (see
 20
 25 Dansette, P.M., Thang, D.C., Amri, H.El and Mansuy, D. (1992) *Biochem. Biophys. Res. Commun.* 186(3), 1624-1630), and could be expected to operate for 4-(vinylthio)-1,2-(methylenedioxy)benzene as well.



The compounds of the invention may be prepared by standard synthetic methods known in the art. For example, the thioethers may be synthesised by reacting Safrole with an appropriate sulphur reagent (e.g. mercaptan). Depending on the type of thioether desired, its synthesis may be realised either by free-radical and/or
5 a photochemical process or utilising heterogeneous catalysts.

Alternatively, the compounds of the invention may be prepared by forming the Grignards reagent by reaction of 4-bromo-1,2-(methylenedioxy)benzene with magnesium turnings. The Grignards reagent may then be reacted an appropriate
10 sulphur reagent (e.g. dialkyl sulphide or 2-chloroethyl thiocyanate) to produce the compounds of the invention. This reaction scheme is illustrated below.



The present invention provides for the use of the compounds of the invention and
15 mixtures thereof as a flavour and/or fragrance. The compounds of the invention are particularly useful as flavourants.

The present invention also provides compositions, products, preparations or
20 articles containing a compound or mixture of compounds of the invention as described above.

The present invention also provides methods to confer, improve, enhance or
25 modify the taste or flavour property of a composition, product, preparation or article which comprises adding thereto a flavour effective amount of a composition or mixture of compounds of the invention as described above.

A method to confer, improve, enhance or modify the aroma, fragrance or odour
30 characteristics of compositions, products, preparations or articles which comprises adding thereto an aroma, fragrance or odour effective amount of a composition or mixture of compounds of the invention as described above is also provided.

The compounds of the invention can be included in virtually any article of manufacture that can include fragrance or flavourant compounds. Examples include hypochlorite (bleach) compositions, detergents, flavorings and fragrances, beverages, including alcoholic beverages, and the like. The compounds of the invention can be used in applications like soaps, shampoos, denture cleanser tablets, body deodorants and antiperspirants, solid or liquid detergents for treating textiles, fabric softeners, detergent compositions and/or all-purpose cleaners for cleaning dishes or various surfaces, for both household and industrial use. Of course, the use of the compounds is not limited to the above-mentioned products, as they be used in other current uses in perfumery, namely the perfuming of soaps and shower gels, hygiene or hair-care products, as well as of body deodorants, air fresheners and cosmetic preparations, and even in fine perfumery, namely in perfumes and colognes.

The compounds of the invention find utility in foods, flavorings, beverages such as beer and soda, denture cleansers (tablets), flavored orally-delivered products such as lozenges, candies, chewing gums, matrices, pharmaceuticals and the like. These uses are described in more detail below.

The compounds of the invention can be used as perfuming ingredients, as single compounds or as mixtures thereof. The compounds can be used in their pure state or as mixtures, with or without added components. The olfactive characteristics of the individual compounds are also present in mixtures thereof, and mixtures of these compounds can be used as perfuming ingredients. This may be particularly advantageous where separation and/or purification steps can be avoided by using compound mixtures.

In all of the above applications, the compounds of the invention can be used alone, in admixture with each other, or in admixture with other perfuming ingredients, solvents or adjuvants of current use in the art. The nature and the variety of these co-ingredients do not require a more detailed description here, which, moreover, would not be exhaustive, and the person skilled in the art will be able to choose

the latter through their general knowledge and as a function of the nature of the product to be perfumed and of the desired olfactive effect.

5 These perfuming ingredients typically belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitrites, terpene hydrocarbons, sulfur- and nitrogen containing heterocyclic compounds, as well as essential oils of natural or synthetic origin. A large number of these ingredients described in reference textbooks such as the book of S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, N.J., USA, the contents of which are hereby
10 incorporated by reference in its entirety, or its more recent versions, or in other works of similar nature.

The proportions in which the compounds of the invention can be incorporated in the various products vary within a large range of values. These values depend on
15 the nature of the article or product that one desires to perfume and the odor effect searched for, as well as on the nature of the co-ingredients in a given composition when the compounds are used in admixture with perfuming co-ingredients, solvents or adjuvants of current use in the art.

20 As an example, the compounds of the invention are typically present at concentrations between about 0.01 and about 30%, or even more, by weight of these compounds relative to the weight of the composition, product or article in which they are incorporated. It will be appreciated that the amount by weight of a compound of the invention in a particular composition or product will depend on
25 the nature of the composition. For example, a washing powder will typically contain less than 1 % by weight of a compound of the invention while a fine fragrance may contain more than 20 % by weight of a compound of the invention.

The compounds may be used in detergents such as those containing bleaching
30 agents and activators such as, for example, tetraacetylenediamine (TAED), hypochlorites, in particular hypochlorite, peroxygenated bleaching agents such as, for example, perborates, etc. The compounds can also be used in body deodorants

and antiperspirants, for example, those containing aluminum salts. These aspects are described in more detail below.

In addition to the compounds of the invention, the compositions described herein
5 may include a deterative surfactant and optionally, one or more additional
detergent ingredients, including materials for assisting or enhancing cleaning
performance, treatment of the substrate to be cleaned, or to modify the aesthetics
of the detergent composition (e.g. perfumes, colorants, dyes, etc.). Non-limiting
10 examples of synthetic deterative surfactants useful herein typically at levels from
about 0.5% to about 90%, by weight, include the conventional C₁₋₁₈ alkyl benzene
sulfonates ("LAS") and primary, branch-chain and random C₁₀₋₂₀ alkyl sulfates
("AS"), and the like. Preferred compositions incorporating only synthetic
detergents have a detergent level of from about 0.5% to 50%. Compositions
containing soap preferably comprise from about 10% to about 90% soap.

15

The compositions described herein can contain other ingredients such as enzymes,
bleaches, fabric softening agents, dye transfer inhibitors, suds suppressors, and
chelating agents, all well known within the art.

20 The compounds of the invention can be incorporated into beverages and impart
various flavorings to the beverages. The beverage composition can be a cola
beverage composition, and can also be coffee, tea, dairy beverage, fruit juice
drink, orange drink, lemon-lime drink, beer, root beer, malt beverages, or other
flavored beverage. The beverage compositions can be in liquid or powdered form.
25 The beverage compositions can be in ready to drink or "concentrated" form. The
beverage compositions can also include one or more flavoring agents; artificial
colorants; vitamin additives; preservatives; caffeine additives; water; acidulants;
thickeners; buffering agents; emulsifiers; and/or fruit juice concentrates.

30 Artificial colorants that may be used include caramel color, yellow 6 and yellow 5.
Useful vitamin additives include vitamin B2, vitamin B6, vitamin B12, vitamin C
(ascorbic acid), niacin, pantothenic acid, biotin and folic acid. Suitable
preservatives include sodium or potassium benzoate. Salts that may be used

include sodium, potassium and magnesium chloride. Exemplary emulsifiers are gum arabic and purity gum, and a useful thickener is pectin. Suitable acidulants include citric, phosphoric and malic acid, and potential buffering agents include sodium and potassium citrate.

5

The beverage may, for example, be a carbonated beverage such as a cola or root beer beverage. The pH is generally about 2.8 and the following ingredients can be used to make the syrup for these compositions: Flavor Concentrate, including one or more of the compounds of the invention herein (22.22 ml), 80% Phosphoric
10 Acid (5.55 g), Citric Acid (0.267 g), Caffeine (1.24 g), artificial sweetener, sugar or com syrup (to taste, depending on the actual sweetener) and Potassium Citrate (4.07 g). The beverage composition can be prepared, for example, by mixing the foregoing syrup with carbonated water in a proportion of 50 ml syrup to 250 ml of carbonated water.

15

Flavoured food (including sweets such as hard or soft candies) and pharmaceutical compositions including one or more of the compounds of the invention can also be prepared. The compounds of the invention can be incorporated into conventional foodstuffs using techniques well known to those of skill in the art. Alternatively,
20 the compounds can be incorporated within polymeric particles, which can, in turn, be dispersed within and/or over a surface of an orally-deliverable matrix material, which is usually a solid or semi-solid substrate. When used in chewable compositions, the compounds of the invention can be released into the orally-deliverable polymeric matrix material as the composition is chewed and
25 held in the mouth, thus prolonging the flavor of the composition. In the case of dried powders and mixes, the flavor can be made available as the product is consumed or be released into the matrix material as the composition is further processed. When two flavors are combined with the polymeric particles, the relative amounts of the additives can be selected to provide simultaneous release
30 and exhaustion of the compounds.

Flavoured compositions of the invention may include an orally-deliverable matrix material; a plurality of water insoluble polymeric particles dispersed in the

orally-deliverable matrix material, where the polymeric particles individually define networks of internal pores and are non-degradable in the digestive tract; and one or more compounds of the invention entrapped within the internal pore networks. The compounds of the invention are released as the matrix is chewed, dissolved in the mouth, or undergoes further processing selected from the group consisting of liquid addition, dry blending, stirring, mixing, heating, baking, and cooking. The orally-deliverable matrix material can be selected from the group consisting of gums, latex materials, crystallized sugars, amorphous sugars, fondants, nougats, jams, jellies, pastes, powders, dry blends, dehydrated food mixes, baked goods, batters, doughs, tablets, and lozenges.

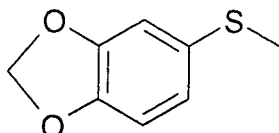
A flavorless gum base can be combined with a compound or a mixture of compounds of the invention to a desired flavor concentration. In one method for producing such gum based products a blade mixer is heated to about 110 °F, the gum base is preheated so that it is softened, and the gum base is then added to the mixer and allowed to mix for approximately 30 seconds. The compound or compounds of the invention are then added to the mixer and mixed for a suitable amount of time. The gum can be then removed from the mixer and rolled to stick thickness on waxed paper while warm.

The compounds of the invention may be incorporated into a system that can release a fragrance in a controlled manner. These include substrates such as air fresheners, laundry detergents, fabric softeners, deodorants, lotions, and other household items. The fragrances are generally one or more derivatives of essential oils as described herein, each present in different quantities. US Patent No. 4,587,129, the contents of which are hereby incorporated by reference in their entirety, describes a method for preparing gel articles that contain up to 90% by weight of fragrance or perfume oils. The gels are prepared from a polymer having a hydroxy (lower alkoxy) 2-alkeneoate, a hydroxy (lower alkoxy) lower alkyl 2-alkeneoate, or a hydroxy poly (lower alkoxy)lower alkyl 2-alkeneoate and a polyethylenically unsaturated crosslinking agent. These materials have continuous slow release properties, i.e. they release the fragrance component continuously over a long period of time. Advantageously, all or a portion of those

derivatives that include an aldehyde group can be modified to include an acetal group, which can cause the formulations to release fragrance over a period of time as the acetal hydrolyzes to form the aldehyde compound.

5 The invention will now be illustrated by the following non-limiting Examples.

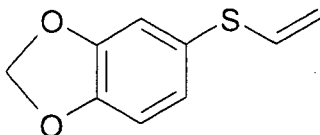
Example 1: Synthesis of 4-(methylthio)-1,2-(methylenedioxy)benzene



Magnesium turnings (3.1 g, 0.127 mol) and a crystal of iodine in anhydrous THF
10 (30 ml) were placed in a 250 ml three-necked round bottom flask fitted with a
condenser and a dropping funnel. A solution of 4-bromo-1,2-
(methylenedioxy)benzene (25 g, 0.124 mol) in anhydrous THF (30 ml) was then
placed in the dropping funnel, 5 ml of this solution was introduced and the
mixture heated with a water bath (45 °C). When the reaction had started the water
15 bath was removed, and the remainder of the 1-bromo-3,4-
(methylenedioxy)benzene solution was added dropwise. When the addition was
complete the solution was stirred for an hour then cooled with an ice bath to 0 °C.
Dimethyl disulfide (13.4 ml, 0.148 mol) in anhydrous THF (15 ml) was added
slowly to the solution of 3,4-methylenedioxyphenylmagnesiumbromide. After the
20 addition the reaction was allowed to warm up to room temperature and the
reaction was stirred for 2 hours. Water (200 ml) was poured into the reaction
mixture and extracted with ethyl acetate (3 x 50 ml). The organic phase was
washed with brine and dried over magnesium sulphate. After evaporation of the
solvent, the crude product was purified by column chromatography on silica gel
25 eluting the ethyl acetate/hexane (2 % / 98 %) to give a mixture of the desired
product and impurities. This mixture was distilled under pressure (25 mbar) and
the impurities were removed at 198°C. to afford safrane.

Example 2: Synthesis of 4-(vinylthio)-1,2-(methylenedioxy)benzene

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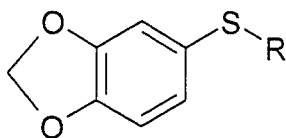


This compound was prepared by adding 3,4-(methylenedioxy)phenylmagnesium bromide (47 cm³, 0.047 mol, see Example 1. for its preparation) to a stirring
5 solution of 2-chloroethyl thiocyanate (5.0 g, 0.041 mol) in anhydrous THF (40 cm³) over 10 mins while keeping the reaction temperature below 40°C. The solution was stirred for a further 15 mins, then potassium t-butoxide (9.2 g, 0.082 mol) in THF (20 cm³) was added in one-shot while the reaction temperature was kept below 55°C with occasional cooling. The reaction mixture was stirred for a
10 further 20 mins. The reaction was quenched with ammonium chloride (sat. aq. 50cm³), the organic layer separated, and the aqueous layer extracted using diethyl ether (3 x 50 cm³). The combined organic extracts were dried (MgSO₄), filtered and concentrated to leave an orange residue, which was purified on silica gel column using petroleum ether/ethyl acetate (100:0 → 98:2) to give the target
15 compound 6 g, 81%) as a light orange oil.

Having hereby disclosed the subject matter of the present invention, it should be apparent that many modifications, substitutions, and variations of the present invention are possible in light thereof. It is to be understood that the present
20 invention can be practiced other than as specifically described. Such modifications, substitutions and variations are intended to be within the scope of the present application.

CLAIMS

1. A compound or a mixture of compounds of formula:

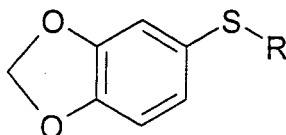


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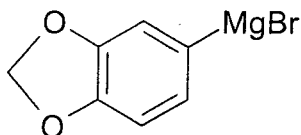
wherein R = straight chain or branched C₁₋₃ alkyl or alkenyl.

2. A compound of claim 1 wherein R is methyl or vinyl.

- 10 3. A method of producing a compound of formula:



wherein R = straight chain or branched C₁₋₃ alkyl or alkenyl, the method comprising reacting a compound of formula:



with an appropriate sulphur-containing reagent.

15

4. A method according to claim 3 wherein the sulphur-containing reagent is selected from dialkyl sulfides and 2-chloroethyl thiocyanate.

5. The use of a compound or a mixture of compounds as defined in claim 1 or
20 2 as a flavour or fragrance.

6. A substrate treated with a compound or mixture of compounds as defined in claim 1 or 2.

- 25 7. A method for treating a substrate to impart flavorant/fragrance releasing characteristics thereto comprising treating the substrate with a compound or mixture of compounds as defined in claim 1 or 2.

8. A composition, product, preparation or article containing as a fragrance component or flavouring ingredient a compound or mixture of compounds as defined in claim 1 or 2 optionally in admixture with other perfuming or flavouring ingredients, solvents, or adjuvants of current use in the art.

9. A beverage composition comprising a compound or mixture of compounds according to claim 1 or 2 and optionally one or more additional beverage ingredients.

10

10. A flavouring composition, product or article comprising a compound or mixture of compounds according to claim 1 or 2 and optionally a solvent, adjuvant and/or another flavouring ingredient.

11. A food composition, product, preparation or article comprising a compound or mixture of compounds according to claim 1 or 2 and optionally a solvent, adjuvant, and/or another food ingredient.

12. A chewing gum composition, product, preparation or article comprising a compound or mixture of compounds according to claim 1 or 2 and optionally a solvent, adjuvant, and/or another chewing gum ingredient.

13. A pharmaceutical composition, product, preparation or article comprising a compound or mixture of compounds according to claim 1 or 2 and a pharmaceutical active ingredient.

14. An orally-deliverable matrix comprising a compound or mixture of compounds according to claim 1 or 2 and at least one matrix material.

15. A method to confer, improve, enhance or modify a taste or flavour property of a composition, product, preparation or article which comprises adding thereto a flavour effective amount of a compound or mixture of compounds as defined in claim 1 or 2.

16. A method according to claim 17, wherein said composition, product, preparation or article is in the form of a beverage, a flavouring, a food, a chewing gum, a pharmaceutical or an orally deliverable matrix.

5

17. A method to confer, improve, enhance or modify an aroma, fragrance or odour characteristics of a composition, product, preparation or article which comprises adding thereto an aroma, fragrance or odour effective amount of a compound or mixture of compounds as defined in claim 1 or 2.

10

18. A method according to claim 19 wherein said composition, product, preparation or article is in the form of a perfume, a body odorant, deodorant or antiperspirant, a detergent, a bleach product or a disinfectant.

15 19. An article of manufacture comprising packaging material and an aroma, odour, fragrance, taste or flavour enhancing agent contained within the packaging material, wherein the agent is effective for the enhancement of the aroma, odour, fragrance, taste or flavour of a composition, preparation, product or article to which it is added, and wherein the packaging material comprises a label which
20 indicates that the agent can be used for enhancing aroma, odour, fragrance, taste or flavour, and wherein the agent is a compound or mixture of compounds as defined in claim 1 or 2.