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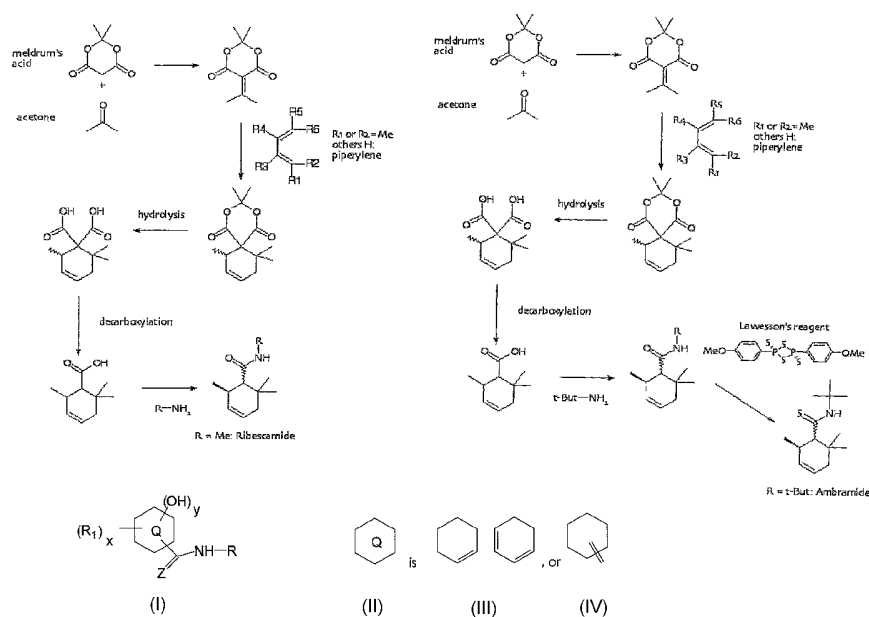
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(54) Title: CYCLOHEXENE CARBOAMIDES AND CARBOTHIOAMIDES



(57) Abstract: A compound or a mixture of compounds comprising an aromachemical compound of the formula: (I) wherein (II) is (III) or (IV); R is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms; R₁ is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms; x is an integer from 1 to 8; y is an integer from 0 to 4, and Z is O or S, as well as compositions, products, preparations or articles having improved aroma, fragrance or odor characteristics containing as active ingredient such compound or mixture of compounds.



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

CYCLOHEXENE CARBOAMIDES AND CARBOTHIOAMIDES

FIELD OF THE INVENTION:

The present invention relates generally to the field of fragrances. More particularly, the present invention relates to novel aromachemicals that provide perfumes and other articles with unique properties and advantages not shared by other aromachemicals. These derivatives find utility in any and all applications requiring certain aroma themes. The invention also relates to mixtures of these derivatives, methods for their preparation and their use as perfume materials for application to a variety of substrates.

BACKGROUND OF THE INVENTION

There are a large number and variety of known fragrances used as ingredients in perfumes and in a varied range of other products. For example, perfumes for application in laundry detergents, fabric softeners, rinse conditioners and other products intended for use on textile fibers primarily contain fragrances.

In recent years there has been much talk about the increasing number of allergies caused by fragrance chemicals in cosmetics and toiletries but as yet there is no legislation to control the use of natural or artificial fragrances. The European Union (EU) Commission now plans to plug this gap in the law.

Under current legislation there are no restrictions on the quantities or combinations of fragrance chemicals that may be used in cosmetics. When fragrance chemicals are added, the law requires only that the label on a cosmetic or toiletry includes the word 'Parfum' in the EU or 'Fragrance' in the USA. There are no requirements to name individual fragrance chemicals in the list of ingredients. This is because a typical perfume contains a mixture of a large

number of fragrance chemicals, often between 50 and 100 and it would be impossible to list them all on a small label. This creates a problem for people who are sensitive or allergic to a particular fragrance chemical. Buying cosmetics becomes a lottery for them because they can never know if the offending chemical has been used in a product or not.

Damascones have been found to be responsible for patient's contact allergy to a number of commercial products. These substances contain chemical structural alerts giving them antigenic ability. [A. Gimenez-Arnau *et al*, "Principles and methodology for identification of fragrance allergens in consumer products", Contact Dermatitis, Volume 47, Issue 6, Page 345, 2002].

Ever since the isolation of .alpha.-Damascone (II) (Tetrahedron letters-1973-381 & Agri.Biol.Chem-1974-38-1351) as an important constituent of Black tea aroma was made, its chemical synthesis has received much attention. However, numerous problems associated with the syntheses of damascones exist [Anthony F. Morris, *et al* - Perfumer & Flavorist (16), 4, 33 (July-August 1991). The problems associated with the syntheses of the damascones have rendered them very expensive [US Patents Nos. 4,113,663; 4,914,229 and US Patent Application Publication No. 20020004615].

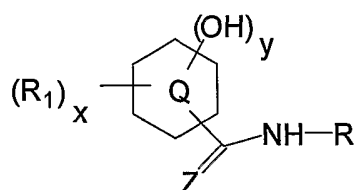
However, the damascones are highly coveted because of their highly desirable fragrance characteristics. It is an object of the invention to provide novel aromachemical compounds and compositions that possess many of the desirable fragrance characteristics of the damascones but which do not cause allergic reactions in most mammals and which are very inexpensive to synthesize.

Novel fragrance and flavor aromachemicals are disclosed herein as well as methods of making the derivatives, uses of the aromachemicals and articles of manufacture including the aromachemicals. These novel derivatives find utility in any and all applications requiring

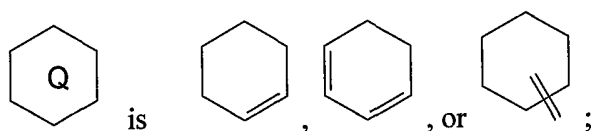
certain aroma themes. The invention also relates to mixtures of these derivatives, methods for their preparation and their use as perfume materials for application to a variety of substrates.

SUMMARY OF THE INVENTION

The invention is directed toward aromachemicals having the formula:



wherein:



R is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms;

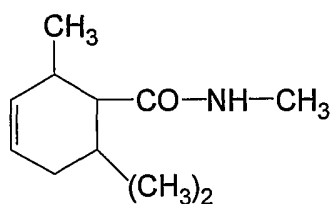
R₁ is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms;

x is an integer from 1 to 8;

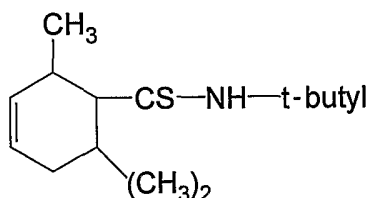
y is an integer from 0 to 4, and

Z is O or S.

A preferred carboamide is 2,6,6-trimethyl-cyclohex-3-enemethylcarboxamide having the formula:



A preferred carbothioamide is 2,6,6-trimethyl-cyclohex-3-ene-t-butylcarboxthioamide having the formula:



A second embodiment of the invention concerns compositions, products, preparations or articles having improved aroma, fragrance or odor characteristics containing as active ingredient a compound described above or a mixture thereof.

Another embodiment of the invention relates to compositions, products, preparations or articles having improved flavor or taste characteristics containing as active ingredient a compound described above or a mixture thereof.

A further embodiment of the invention concerns methods to confer, improve, enhance or modify the taste or flavor property of a composition, product, preparation or article which comprises adding thereto a flavor effective amount of a compound described above or a mixture thereof.

A still further embodiment of the invention relates to a method to confer, improve, enhance or modify the aroma, fragrance or odor characteristics of compositions, products, preparations or articles which comprises adding thereto an aroma, fragrance or odor effective amount of a compound described above or a mixture thereof.

An additional embodiment of the invention concerns articles of manufacture comprising a compound described above or a mixture thereof.

The aromachemicals of the invention may be prepared according to the reaction schemes set forth in the drawing which outlines the syntheses of the above-noted preferred compounds.

The aromachemicals of the invention possess the odor profile: woody, ambery. Examples of suitable articles of manufacture in which the derivatives of the invention may be incorporated include perfumes and colognes, candles, air fresheners, detergent compositions and disinfectants.

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1 and 2 set forth reaction schemes which outline the synthesis of preferred compounds according to the invention.

DETAILED DESCRIPTION OF THE INVENTION

The aromachemicals of the invention can be included in virtually any article of manufacture that can include conventional fragrances, whether natural or artificial. Examples include bleach, detergents, flavorings and fragrances, beverages, including alcoholic beverages, and the like. The derivatives can be used in applications like soaps, shampoos, 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. These uses are described in more detail below.

The compounds can be used as perfuming ingredients, as single compounds or as mixture thereof, preferably at a range of at least about 30% by weight of the perfume composition, more preferably at a range of at least about 60% by weight of the composition. The compounds can even be used in their pure state or as mixtures, 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 cited applications, the derivatives can be used alone 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 its general knowledge and as a function of the nature of the product to be perfumed and of the desired olfactive effect.

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 incorporated by reference in its entirety, or its more recent versions, or in other works of similar nature.

The proportions in which the derivatives can be incorporated in the various products vary within a large range of values. These values depend on 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.

As an example, the derivatives are typically present at concentrations between about 0.1 and about 10%, or even more, by weight of these compounds relative to the weight of the perfuming composition in which they are incorporated. Far lower concentrations than those mentioned above can be used when the compounds are directly applied for perfuming the various consumer products cited beforehand.

The compounds can also be used in body deodorants and antiperspirants, for example, those containing aluminum salts. These embodiments are described in more detail below.

In addition to the derivatives described herein, the compositions herein 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 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.

The compositions 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.

The derivatives described herein 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, malt beverages, or other flavored beverage. The beverages can be in liquid or powdered 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.

Artificial colorants which 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 which 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.

In one embodiment, the beverage is a carbonated cola 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 derivatives described herein (22.22 ml), 80% Phosphoric 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.

Flavored food and pharmaceutical compositions including one or more of the derivatives described herein can also be prepared. The derivatives can be incorporated into conventional foodstuffs using techniques well known to those of skill in the art.

Alternatively, the derivatives 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

derivatives can be released into the orally-deliverable polymeric matrix material as the composition is chewed and 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 and exhaustion of the compounds.

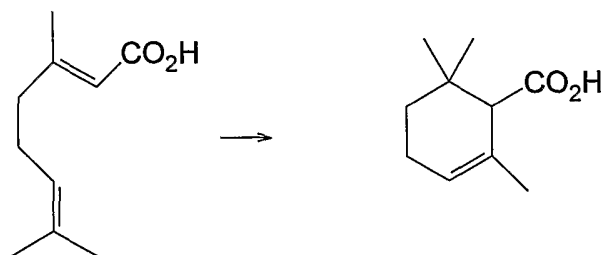
In one embodiment, the flavored composition includes an orally-deliverable matrix material; a plurality of water insoluble polymeric particles dispersed in the orally-deliverable matrix material, where the polymeric particles individually define networks of internal pores and are non-degradable in the digestive tract; and one or more derivatives as described herein entrapped within the internal pore networks. The derivatives 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 suitable derivative as described herein to a desired flavor concentration. Typically, 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 flavored derivative is 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.

In one embodiment, the derivatives described herein are incorporated into a system which can release a fragrance in a controlled manner. These include substrates such as air

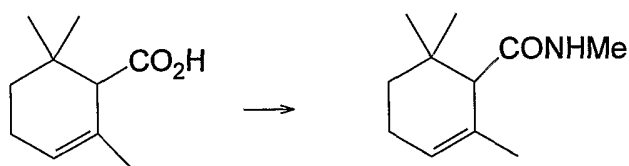
fresheners, laundry detergents, fabric softeners, deodorants, lotions, and other household items. The fragrances are generally one or more derivatives of essential oils as described herein, each present in different quantities. U.S. Pat. No. 4,587,129, the contents of which are hereby incorporated by reference in their entirety, describes a method for preparing gel articles which 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.

EXAMPLE 1



A stirred solution of Geranic acid (15g) in toluene (20mL) was warmed to 100°C and phosphoric acid (0.85mL of 85%) was added dropwise over 5 minutes. Heating was continued for 2 hours, the solution cooled and quenched with sodium hydrogencarbonate

solution. Extraction with diethyl ether gave the cyclised carboxylic acid (11g). This compound was used as such in the next step, it can be recrystallised from hexane if required.



To a stirred powdered solid of the carboxylic acid (11g) was added oxalyl chloride (7mL) carefully over 20 minutes. The solution was stirred for a further 1 hour before evaporation of excess reagents. The acid chloride was taken into dichloromethane (10mL) and added slowly to a stirred solution of methylamine (60mL, aqueous) at 0°C. After the addition was complete the solution was allowed to reach room temperature over 2 hours then extracted with dichloromethane to yield the methylamide. This compound can be further purified by chromatography or recrystallization.

EXAMPLE 2

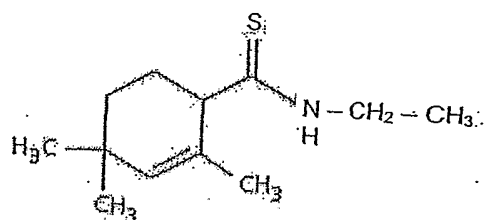
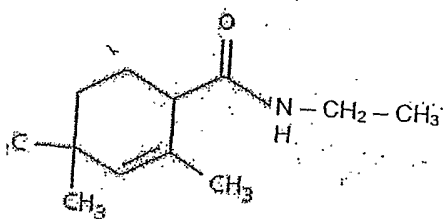
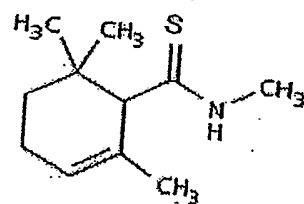
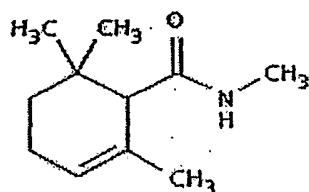
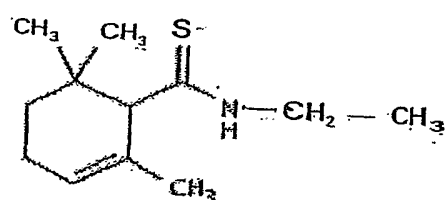
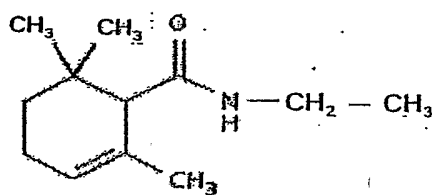
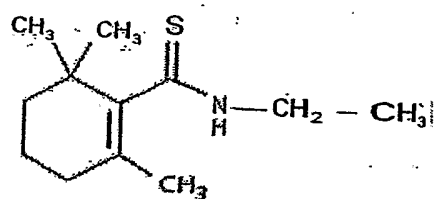
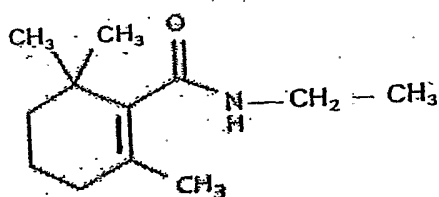
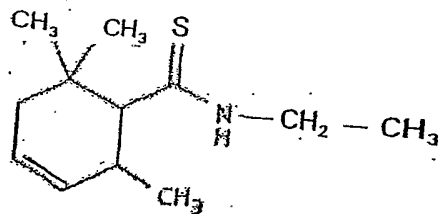
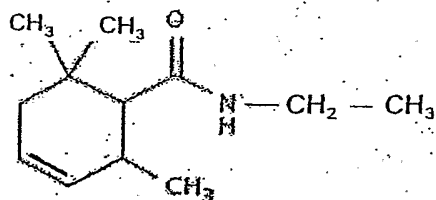
The procedure of Example 1 was repeated utilizing t-butyl amine to prepare the t-butylamide starting material below.



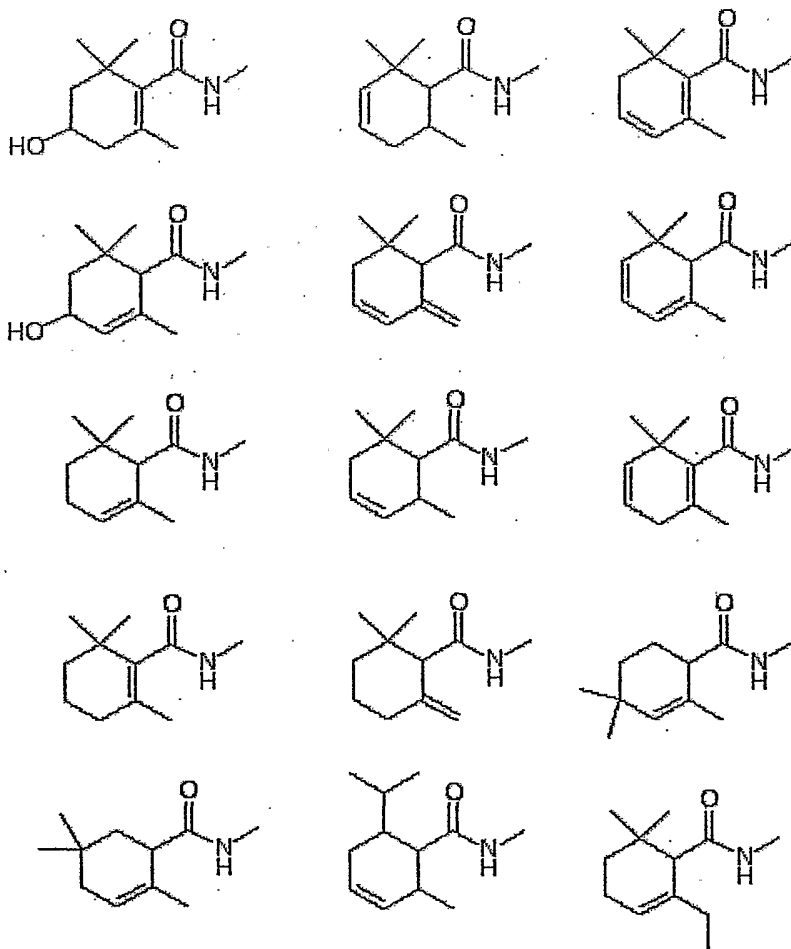
To a solution of the t-butylamide in toluene was added Lawesson's reagent. The reaction was heated to reflux overnight. The reaction was cooled to room temperature and concentrated. Column chromatography yielded the title compound.

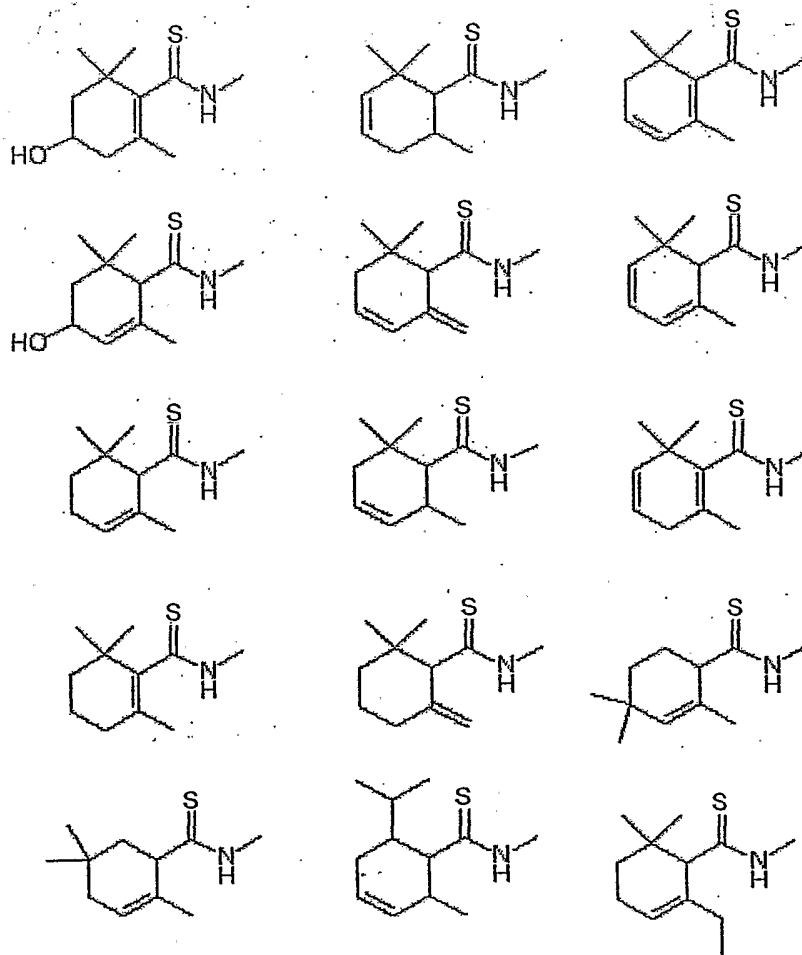
Having hereby disclosed the subject matter of the present invention, it should be apparent that many modifications, substitutions, and variations of the present invention are possible in light thereof. It is to be understood that the present invention can be practiced other than as specifically described.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

Other preferred aromachemicals are those of the formulae:



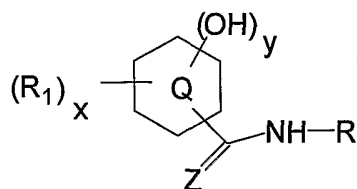
Still other preferred aroma chemicals are those of the formula:



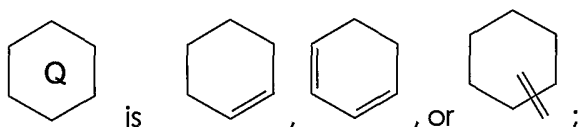


I CLAIM:

1. A compound or a mixture of compounds comprising an aromachemical compound of the formula:



wherein:



R is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms;

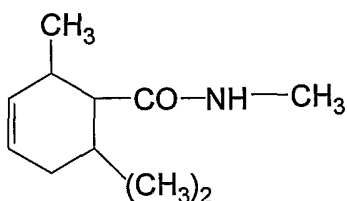
R₁ is a straight or branched chain, saturated or unsaturated hydrocarbyl group; preferably, alkyl or alkenyl, having 1-8 carbon atoms;

x is an integer from 1 to 8;

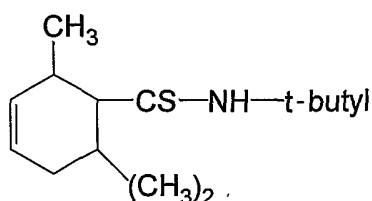
y is an integer from 0 to 4, and

Z is O or S.

2. An aromachemical compound of claim 1 having the formula:



3. An aromachemical compound of claim 1 having the formula:



4. A composition, product, preparation or article having improved aroma, fragrance or odor characteristics containing as active ingredient a compound or mixture of compounds of claim 1.
5. The composition, product, preparation or article of claim 4, wherein the compound or mixture of compounds is present in an amount of at least 30 percent by weight.
6. The composition, product, preparation or article of claim 4, wherein the compound or mixture of compounds is present in an amount of at least 60 percent by weight.
7. A composition, product, preparation or article of claim 4 in the form of a perfume, fragrance or cologne, a soap, a bath or shower gel, a shampoo or other hair care product, a cosmetic preparation, a body odorant, deodorant or antiperspirant, an air freshener, a liquid or solid fabric detergent or softener, bleach product, disinfectant or an all-purpose household or industrial cleaner.
8. A perfuming composition, product, preparation or article of claim 7, wherein the compound or mixture of compounds is present in admixture with other perfuming ingredients, solvents, or adjuvants of current use in the art.
9. A perfumed article according to claim 7, in the form of a perfume or cologne, a soap, a bath or shower gel, a shampoo or other hair care product, a cosmetic preparation, a body deodorant or antiperspirant, an air freshener, a fabric detergent or softener or an all-purpose household cleaner.

10. A composition, product, preparation or article of claim 9 in the form of a perfume wherein the compound or mixture of compounds is in admixture with other perfuming ingredients, solvents or adjuvants.

11. A body deodorant or antiperspirant composition, product, preparation or article, of claim 9.

12. A body deodorant or antiperspirant composition, product, preparation or article, of claim 11, wherein the compound or mixture of compounds is present in admixture with other perfuming ingredients, solvents, or adjuvants of current use in the art.

13. A detergent composition, product, preparation or article of claim 9.

14. A detergent composition, product, preparation or article of claim 13 wherein the compound or mixture of compounds is in admixture with other detergent ingredients, solvents or adjuvants.

15. A bleach composition, product, preparation or article of claim 9.

16. A bleach composition, product, preparation or article of claim 15 wherein the compound or mixture of compounds is in admixture with other bleach ingredients, solvents or adjuvants.

17. A composition, product, preparation or article of claim 9 in the form of a disinfectant.

18. The disinfectant composition, product, preparation or article of claim 17 wherein the compound or mixture of compounds is in admixture with other disinfectant ingredients, solvents or adjuvants.

19. A composition, product, preparation or article having improved flavor or taste characteristics containing as active ingredient a compound or mixture of compounds of claim 1.

20. A composition, product, preparation or article of claim 19 in the form of a beverage.

21. A beverage composition, product, preparation or article of claim 20 wherein the compound or mixture of compounds is in admixture with other beverage ingredients, solvents or adjuvants.

22. A composition, product, preparation or article of claim 19 in the form of a flavoring.

23. A flavoring composition, product, preparation or article of claim 22 wherein the compound or mixture of compounds is in admixture with other flavoring ingredients, solvents or adjuvants.

24. A composition, product, preparation or article of claim 19 in the form of a food.

25. A food composition, product, preparation or article of claim 24 wherein the compound or mixture of compounds is in admixture with other food ingredients, solvents or adjuvants.

26. A composition, product, preparation or article of claim 19 in the form of a chewing gum.

27. A chewing gum composition, product, preparation or article of claim 26 wherein the compound or mixture of compounds is in admixture with other chewing gum ingredients, solvents or adjuvants.

28. A composition, product, preparation or article of claim 19 in the form of a pharmaceutical.

29. A pharmaceutical composition, product, preparation or article of claim 28 wherein the compound or mixture of compounds is in admixture with other pharmaceutical ingredients, solvents or adjuvants.

30. A composition, product, preparation or article of claim 19 in the form of an orally-deliverable matrix material.

31. A composition, product, preparation or article of claim 30 wherein the compound or mixture of compounds is in admixture with other matrix material ingredients, solvents or adjuvants.

32. A method to confer, improve, enhance or modify the taste or flavor property of a composition, product, preparation or article which comprises adding thereto a flavor effective amount of a compound or mixture of compounds of claim 1.

33. The method of claim 21 wherein said composition, product, preparation or article is in the form of a beverage.

34. The method of claim 32 wherein said composition, product, preparation or article is in the form of a flavoring.

35. The method of claim 32 wherein said composition, product, preparation or article is in the form of a food.

36. The method of claim 32 wherein said composition, product, preparation or article is in the form of a chewing gum.

37. The method of claim 32 wherein said composition, product, preparation or article is in the form of a pharmaceutical.

38. The method of claim 32 wherein said composition, product, preparation or article is in the form of an orally deliverable matrix.

39. A method to confer, improve, enhance or modify the aroma, fragrance or odor characteristics of a composition, product, preparation or article which comprises adding thereto an aroma, fragrance or odor effective amount of a compound or mixture of compounds of claim 1.

40. The method of claim 39 wherein said composition, product, preparation or article is in the form of a perfume.

41. The method of claim 39 wherein said composition, product, preparation or article is in the form of a body odorant, deodorant or antiperspirant.

42. The method of claim 39 wherein said composition, product, preparation or article is in the form of a detergent.

43. The method of claim 39 wherein said composition, product, preparation or article is in the form of a bleach product.

44. The method of claim 39 wherein said composition, product, preparation or article is in the form of a disinfectant.

45. An article of manufacture comprising packaging material and an aroma, odor, fragrance, taste or flavor enhancing agent contained within said packaging material, wherein said agent is effective for the enhancement of the aroma, odor, fragrance, taste or flavor of a composition, preparation, product or article to which it is added, and wherein said packaging material comprises a label which indicates that said agent can be used for enhancing aroma, odor, fragrance, taste or flavor, and wherein said agent is a compound or mixture of compounds of claim 1.

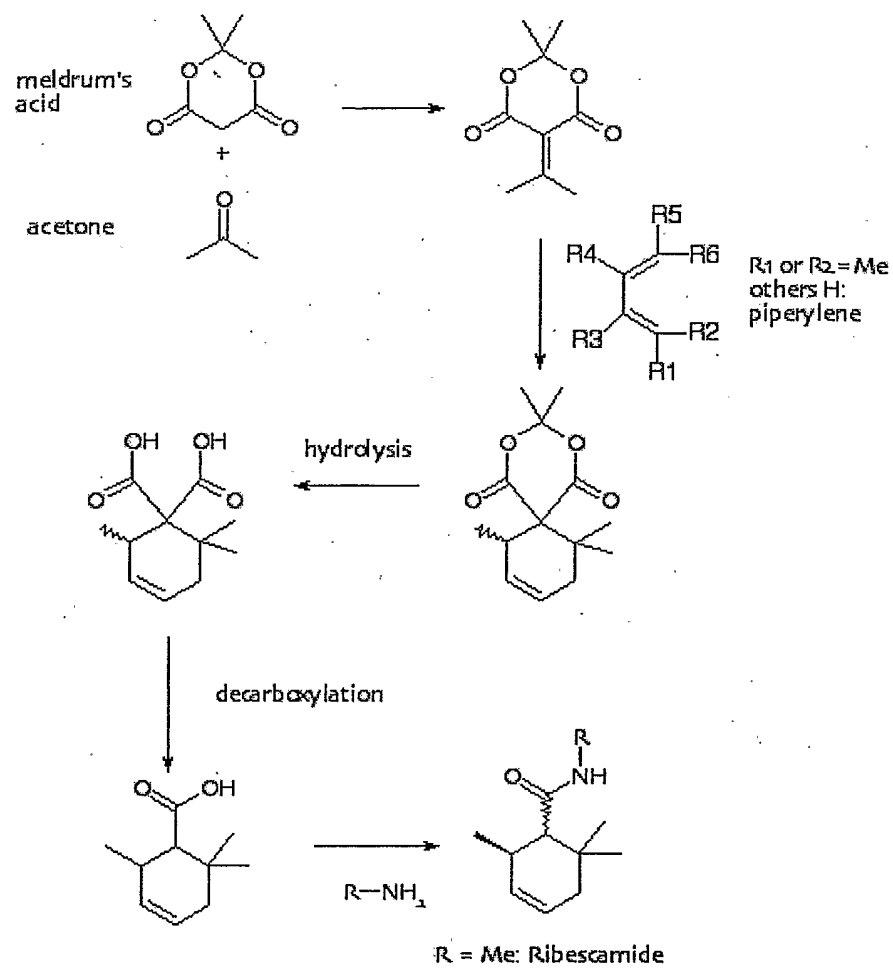


Fig. 1

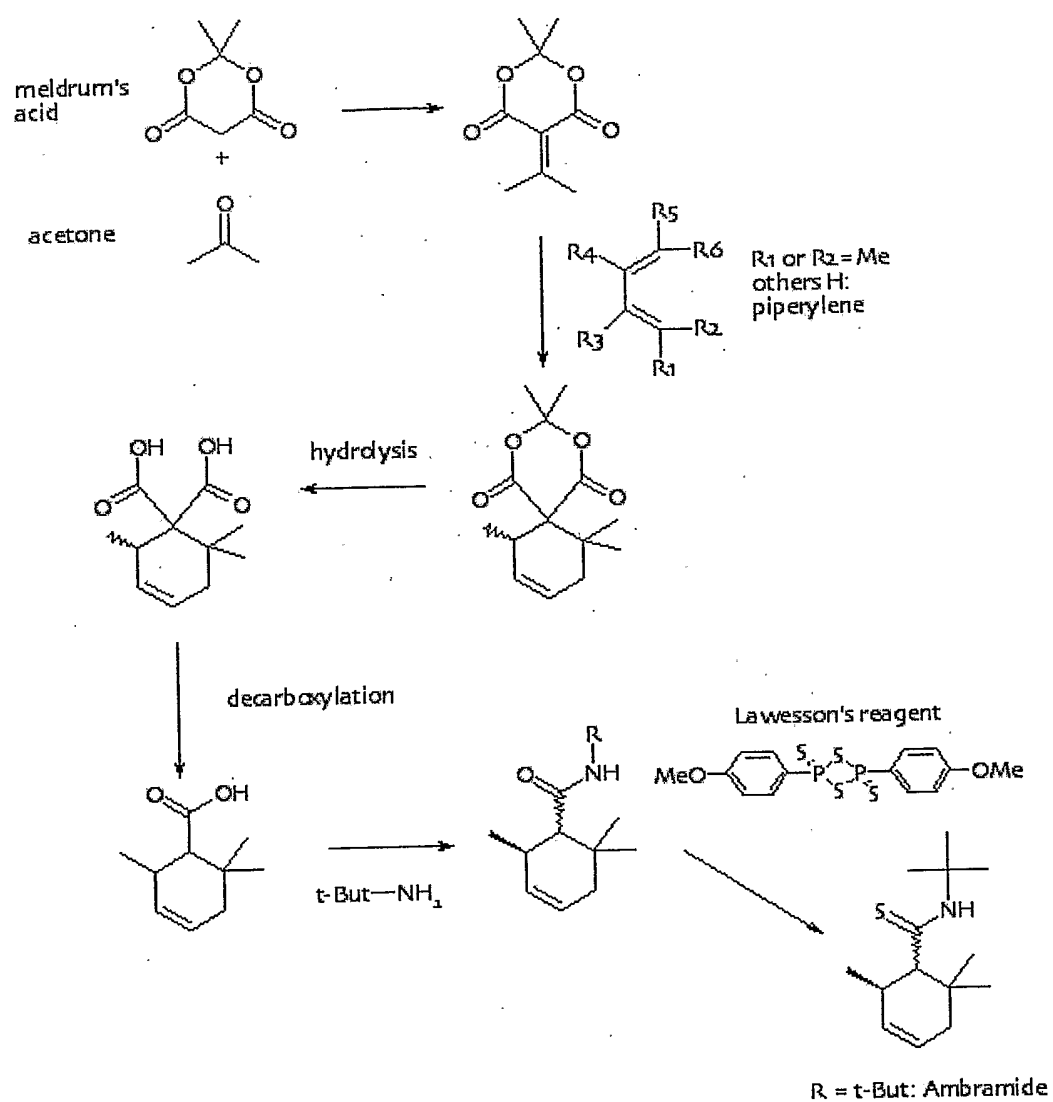


Fig. 2