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(57) Abstract: A liquid fragrance composition consisting essentially of: cyclohexylidene phenyl acetonitrile; and one or more ingredients selected from the group consisting of cyclohexyl salicylate, MEFROSOL™ (3-methyl-5-phenyl-pentan-1-ol), HEDIONE™ (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate; and optionally one or more of 3-methyl-5-phenylpentan-1-ol (MEFROSOL™); an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and dimethyl benzyl carbinol; or a glycol ether.



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IMPROVEMENTS IN OR RELATING TO ORGANIC COMPOUNDS

The present invention relates to stock solutions containing cyclohexylidene phenyl acetonitrile, and to consumer products containing same.

Cyclohexylidene phenyl acetonitrile (known more commonly by the name PEONILETM) is a highly valued perfume ingredient, having an odour that has been characterised as flowery-rosy, green, metallic, and reminiscent of geranium. PEONILETM is powerful, relatively non-volatile and is very stable in almost all media. It has very high substantivity on wet and dry laundry and helps to increase volume and tenacity in functional perfumes. Accordingly, it has been widely used in all manner of consumer products from fine fragrances, through cosmetics, personal care and household care products. The molecule, and its use as an odourant, are described in US 6,069,125.

Despite the considerable commercial success of this molecule, we have found that it is not always a straightforward matter to blend it with other perfume ingredients. In particular, applicant has found that under certain conditions of storage at low temperatures, cyclohexylidene phenyl acetonitrile will crystallise. In particular, crystallisation can occur at temperatures below about 28 degrees centigrade, particularly when the liquid comes into contact with irregular packaging surfaces or solid impurities. Crystallisation leads to very rapid solidification of the bulk material and the only remedy available to the formulator hoping to blend the material with other ingredients is to heat the container in which it is stored. This adds to processing time and costs, and, when the heating step is not carried out carefully, can lead to the development of undesirable burnt off-notes.

The melting point of a specific target substance can be depressed by mixing it with a second substance, which second substance has i) a lower melting point, or ii) forms a eutectic mixture with the target substance. However, whereas there may be many ingredients that can depress the melting point of a target substance, the target substance and the second substance must be thermodynamically compatible (i.e. they should not phase separate) across the entire temperature range within which one requires a liquid state. Still further, liquid mixtures that are thermodynamically compatible below their liquid-solid phase separation temperature, must remain so even if an impurity is

inadvertently or deliberately added to the liquid mixture, or if the liquid mixture is brought into contact with a surface, which could create crystal nucleation sites.

An additional requirement in the field of perfumery is that the mixture of the target substance and the second substance must be olfactively acceptable from a perfumery standpoint, that is, the mixture of substances should have an odour direction and odour intensity that is substantially similar to the pure target substance.

The matter of discovering suitable ingredients to combine with cyclohexylidene phenyl acetonitrile in order to provide a liquid composition, which meet the aforementioned requirements was not straightforward. For example, the skilled person might predict that the fragrance ingredient PETALIATM, which is structurally very similar to cyclohexylidene phenyl acetonitrile, might produce a thermodynamically compatible eutectic mixture, which would remain liquid down to low temperature and which would also be very advantageous because of the similar olfactive direction of cyclohexylidene phenyl acetonitrile and PETALIATM. However, this was found not to be the case. The skilled person might also predict that a broad selection of liquid organic substances, such as liquid fragrance materials and solvents might produce thermodynamically compatible binary mixtures with cyclohexylidene phenyl acetonitrile over broad temperature ranges. However, this was also found not to be the case. In particular, other perfume ingredients having a similar olfactive direction to cyclohexylidene phenyl acetonitrile such as phenyl ethyl alcohol, phenoxy ethyl alcohol, ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methyphenyl ethyl alcohol), ROSAPHENTM (2methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile, were also unable to form thermodynamically compatible liquid binary compositions with cyclohexylidene phenyl acetonitrile. Still further, lower alcohols such as ethanol and isopropyl alcohol; and glycol ethers, such as propylene glycol, dipropylene glycol, DOWANOL

TM PM or DOWANOL TM DPM were also not suitable materials to admix with cyclohexylidene phenyl acetonitrile in compatible binary mixtures.

The present application addresses the potential issues related to the handling and storage of cyclohexylidene phenyl acetonitrile, and overcomes them by providing in a first aspect a

5 liquid fragrance composition consisting essentially of:-

cyclohexylidene phenyl acetonitrile; and

a component A, which is one or more ingredients selected from the group consisting of cyclohexyl salicylate, MEFROSOL TM (3-methyl-5-phenyl-pentan-1-ol), HEDIONE TM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl

10 salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate, phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and optionally

a component B, which is one or more of ROSE OXIDE TM, SUPER MUGUET TM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSE TM (5,6,7-trimethyl-2,5-

15 octadiene-4-one), PIVAROSE TM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSA TM (2-methyphenyl ethyl alcohol), ROSAPHEN TM (2methyl-5-phenyl-pentan-1-ol) PELARGENE TM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLE TM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol
20 comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and dimethyl benzyl carbinol; a glycol ether.

In a particular embodiment of the present invention the liquid fragrance composition consists essentially of:-

25 cyclohexylidene phenyl acetonitrile; and

a component A, which is one or more ingredients selected from the group consisting of cyclohexyl salicylate, MEFROSOL TM (3-methyl-5-phenyl-pentan-1-ol), HEDIONE TM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl

salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate).

In yet another embodiment of the present invention, the liquid fragrance composition
5 consists essentially of:-

cyclohexylidene phenyl acetonitrile; and

a component A, which is a salicylate selected from the group consisting of cyclohexyl salicylate, benzyl salicylate, iso-bornyl salicylate and phenylethyl salicylate.

In yet another embodiment of the present invention, the liquid fragrance composition
10 consists essentially of:-

cyclohexylidene phenyl acetonitrile; and

a component A, which is an ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl salicylate, iso-
15 amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate, phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and

a component B, which is one or more of 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM), ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl
20 carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methylphenyl ethyl alcohol), ROSAPHENTM (2-methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-
25 phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and dimethyl benzyl carbinol; or a glycol ether.

In yet another embodiment of the present invention, the liquid fragrance composition consists essentially of:-

cyclohexylidene phenyl acetonitrile; and

- a component A, which is an ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate, phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and
- 10 a component B, which is 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM) ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methylphenyl ethyl alcohol), ROSAPHENTM (2-methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and
- 15 20 dimethyl benzyl carbinol; or a glycol ether.

As used herein, the term "glycol ether" includes compounds selected from the group consisting of propylene glycol, dipropylene glycol, 1-methoxy-2-propanol DOWANOLTM PM or 2-(2-methoxypropoxy)propan-1-ol DOWANOLTM DPM.

- In a particular embodiment of the present invention, the liquid fragrance composition
- 25 consists essentially of cyclohexylidene phenyl acetonitrile; and cyclohexyl salicylate or benzyl salicylate or iso-bornyl salicylate or phenylethyl salicylate and optionally 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM).

In a particular embodiment of the present invention, the liquid fragrance composition consists essentially of cyclohexylidene phenyl acetonitrile; and cyclohexyl salicylate or

benzyl salicylate or iso-bornyl salicylate or cyclohexyl salicylate or phenylethyl salicylate; and optionally benzyl alcohol or 1-hexanol, 1-heptanol or 1-octanol or cyclohexanol or methyl phenyl carbinol or phenyl ethyl alcohol or dimethyl benzyl carbinol.

In a particular embodiment of the present invention, the liquid fragrance composition
 5 consists essentially of cyclohexylidene phenyl acetonitrile; and cyclohexyl salicylate or benzyl salicylate or iso-bornyl salicylate or phenylethyl salicylate; and optionally a glycol ether, still more particularly, wherein the glycol ether is propylene glycol or dipropylene glycol or 1-methoxy-2-propanol DOWANOLTM PM or 2-(2-methoxypropoxy)propan-1-ol DOWANOLTM DPM.

10 In a particular embodiment of the present invention, the liquid fragrance composition consists essentially of:-

cyclohexylidene phenyl acetonitrile;

and a component A, which is a salicylate selected from the group consisting of cyclohexyl salicylate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3
 15 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate, phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and

one of MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, ROSE OXIDETM, or SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), or dimethyl benzyl carbinol, or
 20 POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), or PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), or PEOMOSATM (2-methyphenyl ethyl alcohol), or ROSAPHENTM (2methyl-5-phenyl-pentan-1-ol), or PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), or diphenyl oxide, or GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), or 3-methyl-2-phenylbut-2-enenitrile, or 3-ethyl-2-
 25 phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile.

Where trivial names or trade names are used to describe perfume ingredients herein, the skilled perfumer will understand that these are commonly used names by perfumers.

However, the skilled perfumer will also understand that these ingredients may also be known by other trivial synonyms, by CAS registry numbers, or by more formal

nomenclature, such as IUPAC nomenclature. Furthermore, the skilled perfumer will be familiar with these synonyms, as well as with more formal nomenclature, or at the least, would be aware of standard reference works, such as The Good Scent Company web site, which contains a comprehensive list of relationships between trivial names, registry

- 5 numbers and more formal nomenclature for all manner of perfume ingredients contained on the perfumers palette.

In a liquid fragrance composition of the present invention, the amount of cyclohexylidene phenyl acetonitrile present may be about 40 to about 95 % by weight, based on the total weight of the composition.

- 10 In a liquid fragrance composition of the present invention, the amount of component A is present between about 5 to about 60 wt % of the liquid fragrance composition, and more particularly 10 and 60 wt %, still more particularly 20 to 60 wt % of the composition, still more particularly 25 to 55 wt % of the composition and still more particularly 45 to 55 wt % .

- 15 In a liquid fragrance composition of the present invention, the amount of component B is present between about 5 to about 25 % by weight of the liquid fragrance composition.

A particular liquid fragrance composition of the present invention consist of cyclohexylidene phenyl acetonitrile (40 to 95 weight % and more particularly 45 to 55 weight %); and cyclohexyl salicylate (20 to 60 weight %, and more particularly 45 to 55
20 weight %).

Yet another particular liquid fragrance composition of the present invention consist of cyclohexylidene phenyl acetonitrile (40 to 95 weight% and more particularly 45 to 55 weight %); and cyclohexyl salicylate (20 to 60 weight %, and more particularly 45 to 55 weight %); and

- 25 benzyl alcohol (5 to 25 weight %, and more particularly 10 weight %).

Yet another particular liquid fragrance composition of the present invention consist of

cyclohexylidene phenyl acetonitrile (40 to 95 weight% and more particularly 45 to 55 weight %); and cyclohexyl salicylate (20 to 60 weight %, and more particularly 45 to 55 weight %); and

3-methyl-5-phenylpentan-1-ol (5 to 20 weight %, and more particularly 10 weight %).

- 5 The ingredients recited hereinabove as representing components of a liquid fragrance composition of the present invention, preferably represent the total mass of said composition. However, it is possible that said liquid fragrance composition may contain minor amounts of impurities or other ingredients commonly found in perfume formulations. Insofar as impurities or other ingredients are present, preferably they
- 10 should not make up more than about 0.0001 to about 5 weight % of the liquid fragrance composition.

- Liquid fragrance compositions of the present invention are flowable, pourable and mixable with other ingredients commonly used in perfumery across all temperature conditions of storage, transportation and use. A liquid fragrance composition should, upon
- 15 inspection with the naked eye, contain no solid or crystalline matter, and in particular no solid or crystalline cyclohexylidene phenyl acetonitrile. In a particular embodiment of the present invention, the liquid fragrance compositions are stable, that is, they do not phase separate over a temperature range of – 20°C to 50 °C, more particularly – 10°C to 50 °C, and still more particularly – 5°C to 50 °C.

- 20 In a particular embodiment of the present invention the liquid fragrance compositions are stable, that is, they do not phase separate over a temperature range of – 20 °C to 50 °C, more particularly – 10°C to 50 °C, and still more particularly – 5°C to 50 °C when stored in a container for a period of one week to one month.

- In a particular embodiment of the present invention the liquid fragrance compositions, in
- 25 25 to 250 Kg quantities, are stable, that is, they do not phase separate over a temperature range of – 20 °C to 50 °C, more particularly – 10°C to 50 °C, and still more particularly – 5°C to 50 °C, when stored in a container for a period of one week to one month.

In a particular embodiment of the present invention, the liquid fragrance composition as herein above defined is in the form of a stock solution.

A stock solution of the present invention is a liquid fragrance composition, as herein defined, which is to be diluted into other perfumery ingredients to form a fragrance composition, which in turn can be used to perfume a consumer product.

In a more particular embodiment of the present invention, the stock solution is provided
5 in containers containing 25 to 250 l of stock solution.

The liquid fragrance composition as herein defined is useful as a fragrance ingredient in a fragrance composition, which in turn can be used to perfume all manner of personal and household care products.

Fragrance compositions, in addition to the liquid fragrance composition, may contain
10 other perfume ingredients, solvents, carrier materials and other auxiliary agents that are commonly employed in the art. Such ingredients are described in "perfume and flavor materials of natural origin", S. Arctander, Ed. Elizabeth, N.J. 1960; "Perfume and Flavor Chemicals", S. Arctander, Ed. Vol I and II, Allured Publishing Corporation, Carol Stream, USA, 1994; and "International Cosmetic Ingredient Dictionary" 6th Ed., the cosmetic,
15 toiletry and fragrance association, Inc. Washington 1995.

Alternatively, the liquid fragrance composition may be employed as the sole fragrance ingredient in personal or household care products.

Liquid fragrance compositions according to the present invention may take the form of an ingredient in a fragrance composition. In this regard, the liquid fragrance composition may
20 be used in amounts substantially similar to those employed by formulators currently using cyclohexylidene phenyl acetonitrile (PEONILETM). In particular, the liquid fragrance compositions may be employed in amounts of 0.2 to 20 % by weight based on the weight of a fragrance composition containing same. Consumer products include, in particular household and personal care products, include but are not limited to solid or liquid
25 detergents and fabric softeners as well as all the other articles common in perfumery, namely perfumes, colognes or after-shave lotions, perfumed soaps, shower or bath salts, mousses, oils or gels, hygiene products or hair care products such as shampoos, body-care products, deodorants or antiperspirants, air fresheners and also cosmetic preparations. As detergents there are intended applications such as detergent compositions or cleaning

products for washing up or for cleaning various surfaces, e. g. intended for textile, dish or hard-surface treatment, whether they are intended for domestic or industrial use. Other perfumed articles include fabric refreshers, ironing waters, papers, wipes or bleaches.

In another aspect of the present invention there is provided a method of rendering
5 cyclohexylidene phenyl acetonitrile in liquid form for a period of up to 1 month at a temperature – 20°C to 50 °C more particularly – 10°C to 50 °C, and still more particularly – 5°C to 50 °C, comprising the step of mixing it with a component A and/or a component B as hereinabove defined, to form a liquid mixture consisting of cyclohexylidene phenyl acetonitrile and a component A and/or a component B.

10 In order to further illustrate the invention and the advantages thereof, the following example is provided.

Example 1

Test samples were prepared by mixing cyclohexylidene phenyl acetonitrile with various amounts of additives, using a Chemspeed mixing robot. The test samples had a size of 25
15 ml and were stored at 5 degrees centigrade for 1 week before being visually inspected.

Samples that remained visibly liquid under these conditions were seeded with a spatula tip of solid cyclohexylidene phenyl acetonitrile, and again stored at 5 degrees centigrade for 1 week.

Finally, those samples that remained visibly liquid under these conditions were submitted
20 to a temperature of minus 20 degrees centigrade for 1 week, before being subjected again to visual inspection.

A binary mixture of cyclohexylidene phenyl acetonitrile and cyclohexyl salicylate (50/50 wt %) remained liquid under all test conditions.

A similar result was achieved with a ternary mixture of cyclohexylidene phenyl
25 acetonitrile, cyclohexyl salicylate and benzyl alcohol.

A similar result was also observed with a ternary mixture of cyclohexylidene phenyl acetonitrile, cyclohexyl salicylate and 3-methyl-5-phenylpentan-1-ol.

Mixtures of cyclohexylidene phenyl acetonitrile and PETALIATM had crystallised when inspected after 1 week storage at 5 degree centigrade.

A ternary mixture of cyclohexylidene phenyl acetonitrile, phenyl ethyl alcohol and cyclohexanol was liquid upon inspection after 1 week, but after seeding and storage for a
5 further week, it had crystallised. A similar result was observed with binary mixtures of cyclohexylidene phenyl acetonitrile, and iso-propyl alcohol.

Claims:

1. A liquid fragrance composition consisting essentially of:-

cyclohexylidene phenyl acetonitrile; and

a component A, which is one or more ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and optionally

a component B, which is one or more of, ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methyphenyl ethyl alcohol), ROSAPHENTM (2methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and dimethyl benzyl carbinol; or a glycol ether.

2. A liquid fragrance composition according to claim 1 consisting essentially of:-

cyclohexylidene phenyl acetonitrile; and

a component A, which is one or more ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl

salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate).

3. A liquid fragrance composition according to claim 1 or claim 2 consisting essentially of:-

5 cyclohexylidene phenyl acetonitrile; and

a component A, which is a salicylate selected from the group consisting of cyclohexyl salicylate, iso-bornyl salicylate, and phenylethyl salicylate.

4. A liquid fragrance composition according to any of the preceding claims consisting essentially of:-

10 cyclohexylidene phenyl acetonitrile; and

a component A, which is an ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, isopropyl myristate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and

a component B, which is one or more of 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM), ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methoxyphenyl ethyl alcohol), ROSAPHENTM (2methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol,

cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol and dimethyl benzyl carbinol; or a glycol ether.

5. A liquid fragrance composition according to any of the preceding claims consisting essentially of:-

5 cyclohexylidene phenyl acetonitrile; and

a component A, which is an ingredient selected from the group consisting of cyclohexyl salicylate, MEFROSOLTM (3-methyl-5-phenyl-pentan-1-ol), HEDIONETM (methyl 2-(3-oxo-2-pentylcyclopentyl)acetate), diethyl phthalate, amyl salicylate, iso-amyl salicylate, benzyl salicylate, iso-bornyl salicylate, cis-3 hexenyl salicylate, ethyl salicylate, iso-butyl salicylate, methyl salicylate and phenylethyl salicylate and KARMAFLOR ((Z)-hept-4-en-2-yl salicylate); and

a component B, which is 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM) ROSE OXIDETM, SUPER MUGUETTM (6-ethyl-3-methyl-6-octen-1-ol), dimethyl benzyl carbinol, POMAROSETM (5,6,7-trimethyl-2,5-octadiene-4-one), PIVAROSETM (phenyl ethyl 2,2-dimethylpropanoate), PEOMOSATM (2-methyphenyl ethyl alcohol), ROSAPHENTM (2methyl-5-phenyl-pentan-1-ol) PELARGENETM (2-methyl-4-methylene-6-phenyl-tetrahydro-2H-pyran), diphenyl oxide, GERANODYLETM (1-hydroxy-2-(1-methyl-1-hydroxyethyl)-5-methylcyclohexane), 3-methyl-2-phenylbut-2-enenitrile, 3-ethyl-2-phenylpent-2-enenitrile or (Z)-2-phenylhex-2-enenitrile; an alcohol comprising more than 3 carbon atoms selected from the group consisting of benzyl alcohol, 1-hexanol, 1-heptanol, 1-octanol, cyclohexanol, cyclohexyl propanol, methyl phenyl carbinol, phenyl ethyl alcohol; and dimethyl benzyl carbinol; or a glycol ether.

6. A liquid fragrance composition according to any of the preceding claims wherein the glycol ether is selected from the group of consisting of propylene glycol, dipropylene glycol, 1-methoxy-2-propanol DOWANOLTM PM or 2-(2-methoxypropoxy)propan-1-ol DOWANOLTM DPM.

7. A liquid fragrance composition according to any of the preceding claims consisting essentially of cyclohexylidene phenyl acetonitrile; cyclohexyl salicylate or benzyl

salicylate or iso-bornyl salicylate or phenylethyl salicylate; and optionally 3-methyl-5-phenylpentan-1-ol (MEFROSOLTM).

8. A liquid fragrance composition according to any of the claims 1 to 6 consisting essentially of cyclohexylidene phenyl acetonitrile; cyclohexyl salicylate or benzyl salicylate or iso-bornyl salicylate or phenylethyl salicylate; and optionally benzyl alcohol or 1-hexanol, 1-heptanol or 1-octanol or cyclohexanol or cyclohexyl propanol or methyl phenyl carbinol or phenyl ethyl alcohol or dimethyl benzyl carbinol.
9. A liquid fragrance composition according to any of the claims 1 to 6 consisting essentially of cyclohexylidene phenyl acetonitrile; cyclohexyl salicylate or benzyl salicylate or iso-bornyl salicylate or phenylethyl salicylate; and optionally a glycol ether, still more particularly, wherein the glycol ether is propylene glycol or dipropylene glycol or 1-methoxy-2-propanol DOWANOLTM PM or 2-(2-methoxypropoxy)propan-1-ol DOWANOLTM DPM.
10. A liquid fragrance composition according to any of the preceding claims wherein the amount of cyclohexylidene phenyl acetonitrile about 40 to about 95 % by weight of the liquid fragrance composition.
11. A liquid fragrance composition according to any of the preceding claims wherein the component A is present is between about 5 and about 60 wt % of the liquid fragrance composition.
12. A liquid fragrance composition according to any of the preceding claims wherein component B is present between about 5 to about 25 % by weight of the liquid fragrance composition.
13. A liquid fragrance composition according to any of the preceding claims which consists of cyclohexylidene phenyl acetonitrile (about 40 to about 95 weight %); and cyclohexyl salicylate (about 20 to about 60 weight %).
14. A liquid fragrance composition according to any of the preceding claims which does not phase separate over a temperature range of – 20°C to 50 °C, more

particularly – 10°C to 50 °C, and still more particularly – 5°C to 50 ° C for a period of one week to one month.

15. A liquid fragrance composition according to any of the preceding claims in the form of a stock solution.
- 5 16. The use of a liquid fragrance composition as defined any of the preceding claims as a fragrance ingredient to perfume personal and household care products.
17. A method of rendering cyclohexylidene phenyl acetonitrile in liquid form for a period of up to 1 month at a temperature – 20°C to 50 °C, comprising the step of mixing it with a component A and optionally a component B as defined in any of
10 the preceding claims, to form a liquid mixture consisting of cyclohexylidene phenyl acetonitrile and a component A and optionally a component B.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2015/000866

A. CLASSIFICATION OF SUBJECT MATTER

INV. A61K8/40 A61Q13/00 C11D3/50 C11D9/44
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A61K A61Q C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6 069 125 A (PESARO MARIO [CH]) 30 May 2000 (2000-05-30) column 1, lines 24-26 -----	1-17
A	EP 1 890 999 A1 (GIVAUDAN SA [CH]) 27 February 2008 (2008-02-27) page 1, paragraph 0001 -----	1-17
X	EP 1 968 531 A2 (GIVAUDAN SA [CH]; NATSCH ANDREAS [CH]; WASESCHA MICHAEL [CH]) 17 September 2008 (2008-09-17) page 1 page 7; example Fragrance composition 2 page 4, paragraph 0013 pages 10-11 -----	1-17
A	EP 2 708 592 A1 (PROCTER & GAMBLE [US]) 19 March 2014 (2014-03-19) the whole document -----	1-17



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

15 September 2015

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2015/000866

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6069125	A	30-05-2000	DE 59607690 D1 18-10-2001
			EP 0858498 A1 19-08-1998
			ES 2162101 T3 16-12-2001
			JP 3802068 B2 26-07-2006
			JP 2000514104 A 24-10-2000
			US 6069125 A 30-05-2000
			WO 9716512 A1 09-05-1997
EP 1890999	A1	27-02-2008	AT 531689 T 15-11-2011
			BR PI0612299 A2 03-11-2010
			CN 101198585 A 11-06-2008
			EP 1890999 A1 27-02-2008
			ES 2375086 T3 24-02-2012
			JP 4938010 B2 23-05-2012
			JP 2008546652 A 25-12-2008
			KR 20080017033 A 25-02-2008
			US 2008200554 A1 21-08-2008
			WO 2006133592 A1 21-12-2006
EP 1968531	A2	17-09-2008	BR PI0615927 A2 31-05-2011
			CN 101262843 A 10-09-2008
			EP 1968531 A2 17-09-2008
			JP 2009509929 A 12-03-2009
			US 2010063011 A1 11-03-2010
			US 2012184513 A1 19-07-2012
			WO 2007030961 A2 22-03-2007
EP 2708592	A1	19-03-2014	CN 104662143 A 27-05-2015
			EP 2708592 A1 19-03-2014
			US 2014075686 A1 20-03-2014
			US 2015067972 A1 12-03-2015
			WO 2014043422 A1 20-03-2014