THE USE OF SALICYCLIC ACID ESTERS AS PERFUMES AND PERFUME COMPOSITIONS CONTAINING THEM

This invention relates to the use of salicylic acid esters as perfuming agents and to perfume compositions containing them.

Numerous esters of salicylic acid are known from the literature. Some of them, including for example the methyl , butyl, amyl, hexyl benzyl and 3-hexenyl esters of salicylic acid, are used in the perfume industry (S. Arctander, Perfume and Flavor Chemicals, 1969; P.Z. Bedoukian, Perfum. Flavor 6 (5) 60-61 (1981).

A new group of salicylic esters distinguished by surprising and valuable perfume properties, particularly by very high persistence, has now been found.

This group consists of 2-hexyl salicylate, 2-ethybutyl salicylate and 2-methylpentyl salicylate.

The esters are of particular interest by virtue of their pronounced odor profile.

The salicylic acid esters are produced in known manner

by esterifying salicylic acid with a secondary or primary branched-chain, saturated aliphatic alcohol corresponding to the above formula in the presence of acidic catalysts, the water given off during the reaction being removed, or by reacting salicylic acid chloride with the alkali alcoholate of the particular alcohol or by transesterifying methyl salicylate with the particular alcohol in the presence of alkaline catalysts.

Of the three compounds mentioned above 2-ethylbutyl salicylate is known from the literature (US-A-2,347,547) although there is no reference to its properties as a perfume. 2-hexyl and 2-methylpentyl salicylate are not known from the literature.

The odor characteristic of the salicylic acid esters in question is generally flowery-sweet and balsamy with a typical salicylate note, flowery, honey-like, herbal notes crucially determining the odor profile in individual cases.

The esters referred to are distinguished by extremely high stability of their odor profile. They do not produce any unpleasant secondary odors, even after prolonged storage of the products perfumed with them. By virtue of their emanating odor and their persistance coupled with their high stability to aggresive media, the esters are particularly suitable for use under practical conditions.

The esters may be used as such or may be combined with other perfumes and/or standard perfume ingredients to form new interesting perfume compositions. In such perfume compositions the esters may be used in a quantity of from 1 to 50% by weight, based on the perfume composition as a whole. Compositions such as these may be used for perfuming cosmetics, such as toilet waters, creams, lotions, aerosols, toilet soaps, in extract perfumery and also for improving the odor of industrial products, such as cleaners, disinfectants, fabric treatment preparations and the like. By virtue of their unusual power of emanation and persistence, the

esters are particularly suitable for perfuming fabric detergents, fabric softeners and cosmetics. The perfumes or compositions in question may be added to the various products in quantities of from 0.05 to 2% by weight, based on the product as a whole.

EXAMPLES

General procedure for an esterification process

1 mole of salicylic acid methyl ester, 2 moles of the particular alcohol and 18 g (0.1 mole) of a 30% sodium methylate solution are initially introduced into a reaction vessel. The methanol released is distilled off through a Claisen bridge at 120°C. The sump temperature rises to approximately 170°C.

On completion of the transesterification reaction, the residue is taken up in water and extracted with ether. The ether extract is washed until neutral, dried over sodium sulfate and concentrated.

The crude product gives the desired ester after distillation through a packed column.

1. 2-hexyl salicylate $B.p._{1.08}$ 108°C
n_D^{20} = 1.5038

Odor: sweet faintly balsamy, honey note

2. 2-ethylbutyl salicylate $B.p._{0.03}$ 103°C
n_D^{20} = 1.5080

Odor: clean, fresh-flowery, herbal-green note

3. 2-methylpentyl salicylate $B.p._{0.01}$ 90°C
n_D^{20} = 1.5059

Odor: green-flowery note.

4. BALSAMY ORIENTAL PERFUME BASE

25	Coumarin
25	Heliotropin
50	Moschus ketone
40	Ethyl vanillin

3	Evernyl(R) (RBD)
17	Labdanum Resin
20	Styrax Honduras
2	Aldehyde C 14 so-c., pure
50	Amyl cinnamic aldehyde (alpha)
30	Eugenol pure
50	Hydroxy citronellal pure
10	Isoraldein 70 (R) (Givaudan)
5	Sandalore (R) (Givaudan)
30	Benzyl acetate
20	Citronellol pure
50	Linalyl acetate
100	Tert.-butyl cyclohexyl acetate
50	Bergamot synthetic
25	Elemi oil
100	Orange oil sweet
50	Patchouli oil Singapore
25	Boisambrene forte (R) (Henkel)
50	Irotyl (R) (Henkel)
25	Hedione (R) (Firmenich)
30	Mandarin oil
25	Geranium oil Bourbon
10	Ambroxan (R) 10% (Henkel)
83	2-ethylbutyl salicylate
1,000	

5. Perfuming of soap

2-ethybutyl salicylate was incorporated in neat soap in a concentration of 1.5%. The forearm of a test subject was washed with the soap for 15 seconds and the odor of the lather assessed. The lather was then rinsed off, the forearm dried and the remaining odor assessed over a period of several hours.

The known perfume, benzyl salicylate, was used for comparison. Power of emanation and persistence were

assessed on a scale of 1 to 6 in which:
6 = very strong emanation and persistence
5 = strong emanation and persistence
4 = good emanation and persistence
3 = still noticeable emanation and persistence
2 = very weak emanation and persistence
1 = no emanation or persistence

Table 1

	Odor of lather	Odor of skin
2-ethylbutyl salicylate	5	5
Benzyl salicylate (Comparison perfume)	3	3

6. Perfuming a fabric softener

0.3% of 2-ethylbutyl salicylate was incorporated as perfume in the formulation of a standard, commercially available fabric softener based on cation-active quaternary ammonium compounds, emulsifiers, viscosity regulators, solvents and diluents.

A formulation perfumed with 0.3% of benzyl salicylate was used for comparison.

Three different cloths of cotton (CN), polyester (PE) and cotton/polyester blend (M) were rinsed with 100 ml of this fabric softener in a washing machine. After spin-drying, the cloths were assessed for odor both in moist form and after drying (overnight on a washing line) (cf Example 5). In addition, the cloths were stored under dry conditions (in polyethylene bags) and reassessed after various periods.

Table 2

	Fabric	Moist	Dried overnight	+24 h	+ 1 week	+ 2 weeks
2-ethylbutyl-salicylate	CN	6	5	4	3	2
	PE	6	4	4	3	2
	M	5	4	3	3	1
Benzyl salicylate	CN	4	3	2	3	1
	PE	4	3	2	2	1
	M	3	3	2	2	1

7. Perfuming a detergent

0.15 % of 2-ethylbutyl salicylate was incorporated as perfume in the formulation of a standard commercially available heavy-duty detergent based on anionic and nonionic surfactants, builders, complexing agents, perborate, redeposition inhibitors, soil suspending agents, brighteners and fillers. A second sample of the same detergent was perfumed with 0.15% of benzyl salicylate for comparison.

Normally soiled washing was washed with the detergents in a drum-type washing machine using the pre-wash and main-wash cycles.

On completion of the pre-wash and main-wash cycles, a) the wash liquor was assessed for odor after rinsing and spin-drying, b) the damp washing was assessed for odor in the same way as described in Example 5.

Table 3

	Odor of the wash liquor		Damp washing
	pre-wash	main-wash	
2-ethylbutyl salicylate	5	6	3
Benzyl salicylate (comparison perfume)	2	2	2

CLAIMS:

1. The use of 2-hexyl salicylate, 2-ethylbutyl salicylate, or 2-methylpentyl salicylate as perfumes.

2. Perfume compositions containing from 1 to 50% by weight of one or more of the compounds specified in claim 1, and other perfumes and/or conventional perfume ingredients.

3. A perfume composition as claimed in claim 2 substantially as herein described with reference to Example 4.

4. A composition perfumed by the addition of one or more salicylic acid esters as defined in claim 1 or by the addition of a perfume composition as claimed in claim 2.

5. A composition as claimed in claim 4 containing 0.05 to 2% by weight of said ester or said perfume composition.

6. A composition as claimed in claim 4 substantially as herein described with reference to Example 5, 6 or 7.

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