

1

2,889,254

SOAPS AND OTHER PERFUMED COMPOSITIONS CONTAINING DIHYDROTERPINYL ACETATE

Alphonse T. Fiore, Teaneck, Garry C. Kitchens, Packanack Lake, and Harry C. Saunders, Nutley, N.J., assignors to The Givaudan Corporation, New York, N.Y., a corporation of New Jersey

No Drawing. Application July 12, 1956
Serial No. 597,359

3 Claims. (Cl. 167—94)

This invention relates to soaps, such as toilet soap, and other perfumed compositions containing dihydroterpinyl acetate.

An object of this invention is to provide a toilet soap having the following odor qualities: good stability, freshness and naturalness (as opposed to a "chemical" odor).

Another object is to provide a toilet soap having an odor which is clean and pleasant such as is obtained from the ester components of important natural soap odorants, e.g., lavender oil, petitgrain oil, and bergamot oil, and yet not having the roughness and undesirable pine-like character associated with synthetic esters.

Another object of this invention is to furnish perfumed compositions containing a low-cost readily available synthetic ingredient which resembles the oils of bergamot and petitgrain.

Other objects of our invention will become apparent from the following description.

We have attained the foregoing and other objects by incorporating dihydroterpinyl acetate into toilet soap and other perfumed compositions. We have found that amounts of dihydroterpinyl acetate from about 0.02 to 1.5% by weight of the total soap give satisfactory results and that amounts from 0.1 to 0.6% on the same basis give excellent results.

In using dihydroterpinyl acetate as a substitute or extender for oil of petitgrain it has been found that the acetate can be used up to and including 40% by weight of the petitgrain oil-dihydroterpinyl acetate mixture.

Artificial bergamot oils can be made by using from 5 to 75% by weight of dihydroterpinyl acetate on the basis of the total mixture. We prefer to use amounts from 40 to 60% on the same basis.

Artificial oil of petitgrain containing from 5 to 65% by weight of dihydroterpinyl acetate on the basis of the total mixture can be made. However, we prefer to use amounts from 30 to 45% on the same basis.

In using our novel perfume ingredient the usual practices of the perfumer may be employed. For this reason specific instructions concerning the method of formulating toilet soap and other perfumed compositions containing dihydroterpinyl acetate are unnecessary here.

Dihydroterpinyl acetate has already been described in the literature. To the best of our present knowledge it was first described in 1910 by M. A. Béhal and M. A. Haller in *Compt. rend.*, 150, 1762-65.

However, in spite of the fact that dihydroterpinyl acetate has already been known for over 45 years and the fact that terpinyl acetate has been known for a long time for its olfactory properties, no one until now recognized the unique olfactory properties of dihydroterpinyl acetate. It may well be that the reason for this omission stems from the fact that the closely-related linalyl acetate is weakened in odor qualities when it is hydrogenated. It was therefore surprising to us that we found that the odor of dihydroterpinyl acetate is not only as strong as that of terpinyl acetate but the

2

former possesses the aforementioned desirable qualities not possessed by terpinyl acetate.

In order further to illustrate our invention we give the following examples, it being understood that the dihydroterpinyl acetate employed was prepared by the direct acetylation of dihydroterpineol prepared from commercial terpineols, or by the hydrogenation of commercial terpinyl acetate.

The physical constants of dihydroterpinyl acetate follow:

Specific gravity, 25° C./25° C. ----- 0.9328-0.9337
Refractive index, 20° C. ----- 1.4493-1.4495
Purity ----- percent -- 95-100

EXAMPLE I

Soap cakes were made as follows:

Twenty grams of standard No. 1 soap pellets and 0.1 gram of perfuming agent were milled together in a Coors No. 522-4 porcelain mortar until they were reduced to a fine homogeneous powder. Two grams of distilled water were added and the mixture was milled again to a plastic mass. The soap thus prepared was charged into a cylindrical tableting die which when pressed by a hand arbor-press produced a soap cake 3.8 cm. in diameter and 2.0 cm. thick. In separate cakes made as just described was incorporated the same specified portions of dihydroterpinyl acetate, terpinyl acetate and linalyl acetate. The cakes were then exposed to air and daylight indoors for a period of 6 months and examined about once a month.

The cake containing the dihydroterpinyl acetate maintained a strong, fresh odor during the entire period. The cake containing the terpinyl acetate weakened after two months and faded completely after three months. The cake containing the linalyl acetate turned rancid or sour smelling after two months.

EXAMPLE II

A soap perfume was formulated using dihydroterpinyl acetate as an ingredient. The formula is as follows:

Soap perfume No. 1

45	Dihydroterpinyl acetate -----	120
	Citronellol -----	40
	Phenylethyl alcohol -----	100
	Terpineol -----	120
	Benzyl acetate -----	40
50	Amylcinnamic aldehyde -----	20
	Geranium Bourbon oil -----	20
	Cassia oil redistilled -----	6
	Lavandin oil, 22 to 24% ester -----	3
	Musk xylol -----	2
55	Ionone pure -----	48
	Benzophenone -----	8
	Cedrenol -----	4
	Rue oil -----	6
	Patchouly oil -----	1
60	Thyme oil white -----	1
	Soluble resin galbanum (10% diethyl phthalate) --	5
	Soluble resin Styrax (10% in diethyl phthalate) ---	8

552

This perfume was incorporated in milled toilet soap at the rate of 1% in the same manner as described in Example I. It covered the odor of the soap well and gave a pleasant refreshing odor to the soap.

EXAMPLE III

Another soap perfume was formulated containing a

3

larger proportion of dihydroterpinyl acetate. Its formula is as follows:

Soap perfume No. 2

Dihydroterpinyl acetate	200
Citronellol	160
α -Methyl p-isopropylhydrocinnamic aldehyde	8
Cedryl acetate	5
Coumarin crystals N.F.	20
1,1,4,4-tetramethyl-6-acetyl 7-ethyl-1,2,3,4,-tetrahydronaphthalene (patent pending)	2
Vanillin (10% in diethyl phthalate)	7
Amylcinnamic aldehyde	20
o-Phenylanisole	12
Benzyl isoamyl ether	10
Sandalwood (East Indian) oil	1
Spike lavender oil	4
Soluble resin oak moss	1
Spearmint oil	3
Soluble resin olibanum	5
Bay leaves N.F. oil	11
Methyl benzoate	3
	472

This perfume was incorporated in soap at the rate of 1% in a manner as described in Example I and found to give a good cover to the soap odor and a pleasant clean odor.

EXAMPLE IV

Compositions containing oil of petitgrain and 10, 20, 30, 40, 50, 60 and 70% dihydroterpinyl acetate by weight of the total mixture were prepared. Similar series containing oil of petitgrain and other synthetic extenders such as terpinyl acetate and nopyl acetate (6,6-dimethylbicyclo-[1,1,3]-hept-2-ene-2 ethyl acetate) were prepared.

The petitgrain mixtures containing the other synthetic extenders possessed a distracting piney note which was not evident in the petitgrain mixture containing dihydroterpinyl acetate.

Those petitgrain mixtures, containing dihydroterpinyl acetate had more realistic petitgrain oil odor than the mixture containing the other petitgrain extenders at like concentrations.

EXAMPLE V

An artificial bergamot was formulated using 70% of dihydroterpinyl acetate as the main ester component. Following is its formula:

Bergamot type

Dihydroterpinyl acetate	600
Oil sweet orange cold pressed California	30
Methyl benzoate	20
N-decanal (10% in diethyl phthalate)	3
Camphene	16
Geranyl formate	60
Coumarin crystals N.F.	2
p-Isopropylacetophenone	26
Terpineol	40
2-methylundecanal-1 (10% in diethyl phthalate)	1
Peppermint rectified oil	5

4

Soluble resin galbanum (10% in diethyl phthalate)	6
Estragon oil (10% in diethyl phthalate)	14
Bay leaves oil	2
Lemon oil California	3
γ -Undecalactone (10% in diethyl phthalate)	1
Soluble resin labdanum (10% in diethyl phthalate)	2
Soluble resin olibanum	5
Benzaldehyde (1% in diethyl phthalate)	20
	856

EXAMPLE VI

An artificial oil of petitgrain was formulated. The following is the formula:

Dihydroterpinyl acetate	200
Dipentene	60
Camphene	40
Myrcene	24
Citronellol	10
Geranyl acetate	20
Methyl anthranilate	4
3-cyclohexylpropanone-2	16
Furfural	1
N-decanal (10% in diethyl phthalate)	2
Amyl benzoate	8
Bay leaves oil	1.5
Thyme oil white	1.5
Rue oil	2.5
p-Cresyl acetate	1.5
Fennel oil sweet	1
	393

While we have described our invention in detail in its preferred embodiment, it will be obvious to those skilled in the art, after understanding our invention, that various changes and modifications may be made therein without departing from the spirit or scope thereof. We aim in the appended claims to cover all such modifications and changes.

We claim:

1. Toilet soap containing a minor amount of dihydroterpinyl acetate sufficient to impart to said soap odor stability, freshness and naturalness.
2. Toilet soap, containing from about 0.02% to about 1.5% by weight of dihydroterpinyl acetate, said soap being characterized by odor stability, freshness and naturalness imparted thereto by said ester.
3. A perfume composition comprising oil of petitgrain and as an extender therefor, dihydroterpinyl acetate, the latter being present in an amount up to about 40% by weight of the total weight of said oil of petitgrain and said extender.

References Cited in the file of this patent

- Behal: Comptes Rendus, tome 150, June 1910, pp. 1762-4.
- Simonsen: *The Terpenes*, Cambridge Univ. Press, vol. 1, 2nd ed., revised 1953, pp. 253, 254, 262.
- Wertheim: *Textbook of Organic Chem.*, the Blakiston Co., Philadelphia, 2nd ed., 1946, pp 50, 52.