

UNITED STATES PATENT OFFICE

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PERFUME MATERIAL

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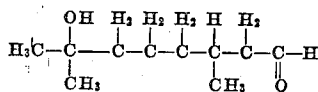
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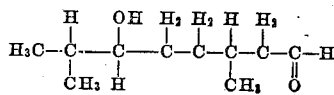
This invention deals with improvements in the perfumery art. More particularly, it relates to perfume compositions containing hydroxycitronellal and hydroxycitronellol. Some examples of such compositions are cosmetics, toilet and laundry soaps, and perfumes.

The object of this invention is to provide perfume ingredients containing hydroxycitronellal in a relatively stable form. A further object is to obtain a stabilized hydroxycitronellal whose lily of the valley odor has not been materially modified from that of the unstabilized hydroxycitronellal. Another object is to provide a stabilized hydroxycitronellal capable of effectively masking the undesirable "soapy" odor in soap and at the same time imparting a pleasant lily of the valley scent thereto. A still further object of this invention is to furnish new products for use in the perfumery arts. Other objects will be apparent to those skilled in the art from the description.

Hydroxycitronellal is a liquid having a very pronounced odor and is widely used in perfumery. Its probable structural formula is:



or



The alternate formulae are given because it is not known whether hydroxycitronellal has one of its hydroxyl groups in the No. 2 or No. 3 position, or whether it is a mixture of both such products whose formulae are above given. Unfortunately, much of the odor value of hydroxycitronellal is soon lost in use. This is a general failing of aldehydes and has been attributed generally to the formation of the corresponding acids by reason of oxidation.

We have discovered that the desirable properties of hydroxycitronellal can be practically utilized in perfumes, soaps, etc. by adding hydroxycitronellol thereto. The latter has been prepared by hydrogenating hydroxycitronellal and is described in the co-pending application of M. S. Carpenter, S. N. 337,834, filed May 29, 1940. It is a colorless and somewhat viscous oil having a very faint odor and characterized by the following physical data:

Boiling point ----- 140° C. at 3 mm. mercury
Specific gravity at 25°
C./25° C. ----- 0.930
Refractive index n_D^{20} ----- 1.460

That the hydroxycitronellol-hydroxycitronellal system is not a mere additive system is clear from

available data. There is a temperature increase upon admixture of the two materials. Moreover, density and refractive index figures are higher than one would expect to find if he were dealing with a mixture of the two substances. Viscosity of the hydroxycitronellol-hydroxycitronellal solution is appreciably lower than the calculated viscosity. It is probable that a chemical reaction occurs when hydroxycitronellol-hydroxycitronellal are brought into contact, even at room temperatures.

The stabilizing effect of hydroxycitronellol on hydroxycitronellal was tested as follows:

50 grams of the sample to be tested were placed in a Petri dish. The latter was allowed to stand uncovered at room temperature, i. e. 20°-25° C. The acid number of each sample was taken at the start of the test and after 4, 8 and 12 days. The following tables illustrate the changes in acid number of the samples with passage of time and show the effect of hydroxycitronellol.

Table I

Sample	Composition		Acid values			
	Hydroxycitronellal	Hydroxycitronellol	Initial	4 days	8 days	12 days
A.....	100	0	7.4	41.5	66.5	101.5
B.....	85	15	5.2	27.6	42.5	65.0
C.....	75	25	4.6	19.8	29.6	45.3
D.....	60	40	3.6	8.4	11.9	22.3
E.....	25	75	2.6	3.2	5.0	5.8
F.....	15	85	2.1	2.3	2.5	3.8
G.....	0	100	1.4	1.5	1.6	1.7

Table II

Sample	Calculated acid value of hydroxycitronellal*			
	Initial	4 days	8 days	12 days
A.....	7.4	41.5	66.5	101.5
B.....	5.9	32.2	49.7	76.5
C.....	5.65	25.9	39.0	59.7
D.....	5.3	15.3	22.2	44.0
E.....	5.0	8.35	15.2	18.0
F.....	6.05	6.9	7.6	15.7

*These data were obtained by subtracting the product of the corresponding acid value of hydroxycitronellol (G) and the figure representing the fraction of the sample consisting of hydroxycitronellol from the acid value of the sample (Table I) and dividing this figure by the number representing the fraction of the sample consisting of hydroxycitronellal.

These results show clearly the inhibiting effect of hydroxycitronellol on the oxidation of hydroxycitronellal. Whereas one gram of the latter has an acid number of 101.5 after 12 days exposure under the test conditions, it would have a calculated acid number of only 44.0 after the same period and under the same conditions if 1

gram of hydroxycitronellol were added to it initially, i. e. at the start of the test.

Soaps which contain hydroxycitronellal as a perfume ingredient have certain properties improved by the incorporation of hydroxycitronellol. The odor retention of such soaps as well as the color stability and tendency to resist rancidity are enhanced. Although other amounts of hydroxycitronellol impart desirable characteristics, we prefer to use between 25 and 75% of hydroxycitronellol based on the combined weight of the hydroxycitronellol-hydroxycitronellal. In practice, hydroxycitronellal is used in amounts ranging from ½% to 2%, based on the weight of the soap.

As has already been indicated, an advantage obtained by using hydroxycitronellol is that it not only acts as a stabilizer for hydroxycitronellal but it also does not materially affect the hydroxycitronellal odor, namely lily of the valley. The importance of these effects are apparent to those skilled in the art.

It is understood that the hydroxycitronellol-hydroxycitronellal solutions herein disclosed may be used as ingredients in perfumes having other perfume constituents wherever it is desired to use the lily of the valley odor of the stabilized hydroxycitronellal as herein disclosed.

It is understood that the invention is to be construed as broadly as permissible in view of the prior art, limited solely by the appended claims.

What we claim as new is:

1. A perfume composition containing hydroxycitronellal and hydroxycitronellol to stabilize the hydroxycitronellal against air oxidation.

2. A perfume ingredient consisting of hydroxycitronellal and hydroxycitronellol to stabilize the same against air oxidation, said ingredient being characterized by its lily of the valley odor.

3. A perfume composition having hydroxycitronellal and hydroxycitronellol to stabilize the hydroxycitronellal against air oxidation, the hydroxycitronellol being present in an amount ranging from 25% to 75% of the combined weight of hydroxycitronellol and hydroxycitronellal.

4. A perfume ingredient consisting of 75 to 25 parts by weight of hydroxycitronellal and 25 to 75 parts by weight of hydroxycitronellol to stabilize the hydroxycitronellal against air oxidation, said ingredient being characterized by its lily of the valley odor.

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