United States Patent [19]

Hall et al.

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[54] **NOVEL FRAGRANCE MATERIALS**

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[52] **U.S. Cl.** **252/522**

[51] **Int. Cl.** **C11b 9/00**

[58] **Field of Search** 252/522

[56] **References Cited**

UNITED STATES PATENTS

3,530,171 9/1970 Heckert 260/489

OTHER PUBLICATIONS

Chem. Abstr., Vol. 70, 1969, 32946 Z.

Grimm et al., — Z. Chem. 8, 424–425 (1968).

Primary Examiner—Frederick E. Waddell

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[57] **ABSTRACT**

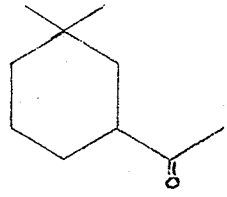
Perfume and fragrance compositions containing the ketone 2,5,5 trimethylacetylcycloheptane which imparts thereto long-lasting tobacco, musky-sweaty, tea-like, woody and orris notes.

4 Claims, No Drawings

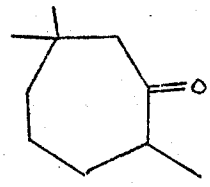
NOVEL FRAGRANCE MATERIALS

BACKGROUND OF THE INVENTION

There is a continuing search for materials having desirable fragrance properties. Such materials are sought either to replace costly natural materials or to provide new fragrances or perfume types which have not heretofore been available. Especially desirable qualities for substances having interesting fragrances are stability and persistence in a wide variety of perfumed articles and perfume compositions, ease of manufacture, and intensity of aroma. Thus, British Pat. No. 1,159,188 published on July 23, 1969 describes 3,3 dimethyl acetyl cyclohexane having the structure:



as a perfume ingredient having a "strong, minty herbaceous odor". (See Example V thereof). Enlarging the ring to seven carbon atoms, adding one methyl group, and varying the positions of the functional groups on the rings has herein been found to give rise to unobvious, unexpected and advantageous results in the field of perfumery as hereinafter more fully described. It should be noted that the process described in Example V of British Pat. No. 1,159,188 actually produces a mixture which also contains a compound having the structure:

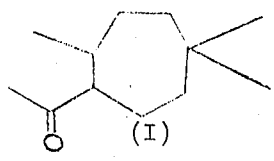


See Hall and Lala, "Cyclization of Dimethyl-1,6-Octadienes" J.Org.Chem.37,920 (1972).

THE INVENTION

The invention comprises the novel compositions containing 2,5,5 trimethylacetyl cycloheptane as well as the novel processes and steps of processes according to which such 2,5,5 trimethylacetyl cycloheptane is manufactured, the specific embodiments of which are described hereinafter by way of example and in accordance with which it is now preferred to practice the invention.

Briefly, the present invention provides the ketone 2,5,5 trimethylacetyl cycloheptane having the formula:



in perfume and fragrance-modifying materials. The ketone is obtained by hydrogenation of 4 αacetyl-2-carene as more fully described in Example I, infra.

It has been found that the ketone of this invention possesses tobacco, musky-sweaty, tea-like, woody and orris notes with good intensity and persistence. This fragrance quality particularly adapts the ketone for incorporation into perfume compositions and fragrance-modifying compositions having a desirable musky aroma. It will be appreciated by those skilled in the art from the present disclosure that the fragrance character of the finished perfume compositions can be tailored to specific uses, as more fully described hereinafter.

The preparation of 2,5,5 trimethylacetyl cycloheptane and its IR curve is set forth in Grimm et al. Z.Chem.8,424–5 (1968).

Purified 2,5,5 trimethylacetyl cycloheptane (herein sometimes called "Ketone I") is an olfactory agent and can be incorporated into a wide variety of compositions which will be enhanced by its tobacco, musky-sweaty, tea-like, woody and orris notes. The Ketone I can be added to perfume compositions in its pure form or it can be added to mixtures of materials in fragrance-imparting compositions to provide a desired fragrance character to a finished perfume material. The perfume and fragrance compositions obtained according to this invention are suitable in a wide variety of perfumed articles and can also be used to enhance, modify or reinforce natural fragrance materials. It will thus be appreciated that the Ketone I of this invention is useful as an olfactory agent and fragrance.

The term "perfume composition" is used herein to mean a mixture of compounds, including, for example, natural oils, synthetic oils, alcohols, aldehydes, ketones, esters, lactones, and frequently hydrocarbons which are admixed so that the combined odors of the individual components produce a pleasant or desired fragrance. Such perfume compositions usually contain (a) the main note or the "bouquet" or foundation-stone of the composition; (b) modifiers which round-off and accompany the main note; (c) fixatives which include odorous substances which lend a particular note to the perfume throughout all stages of evaporation, and substances which retard evaporation; and (d) top-notes which are usually low-boiling fresh-smelling materials. Such perfume compositions of this invention can be used in conjunction with carriers, vehicles, solvents, dispersants, emulsifiers, surface-active agents, aerosol propellants, and the like.

In perfume compositions the individual components contribute their particular olfactory characteristics, but the overall effect of the perfume composition will be the sum of the effect of each ingredient. Thus, the Ketone I of this invention can be used to alter the aroma characteristics of a perfume composition, for example, by highlighting or moderating the olfactory reaction contributed by another ingredient of the composition.

The amount of Ketone I of this invention which will be effective in perfume compositions depends on many factors, including the other ingredients, their amounts and the effects which are desired. It has been found that perfume compositions containing as much as 50% or as little as 3.0% by weight of mixtures or compounds of this invention, or even less can be used to impart a tobacco-musky odor to soaps, cosmetics and other products. The amount employed will depend on con-

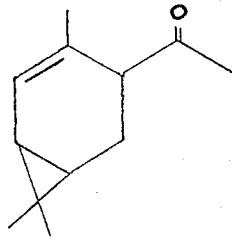
sideration of cost, nature of the end product, the effect desired in the finished product, and the particular fragrance sought.

The Ketone I disclosed herein can be used alone, in a fragrance-modifying composition, or in a perfume composition as an olfactory component in detergents and soaps; space deodorants; perfumes; colognes; bath preparations such as bath oil, bath salts; hair preparations such as lacquers, brilliantines, pomades, and shampoos; cosmetic preparations such as creams, deodorants, hand lotions, sun screens; powders such as talcs, dusting powders, face powder and the like. When the Ketone I of this invention is used in perfumed articles such as the foregoing, it can be used in amounts of 0.1% or lower. Generally, it is preferred not to use more than about 10% in the finished perfumed article, since the use of too much will tend to unbalance the total aroma and will needlessly raise the cost of the article

The following examples serve to illustrate embodiments of the invention as it is now preferred to practice it. It will be understood that these examples are illustrative and the invention is to be considered restricted thereto only as indicated in the appended claims.

EXAMPLE I

The following ingredients are introduced into an autoclave:

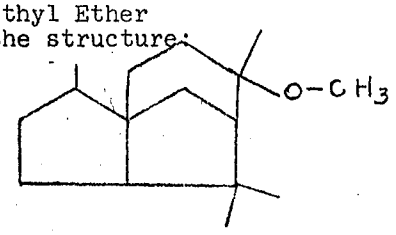
INGREDIENT	AMOUNT
4∝Acetyl-2-Carene (Richter, Chem.Ber. 100,1892-97 (1967); Alder et al. E.German Patent 39,693) Structure: 	450 g.
Acetic Acid	200.0 ml
Palladium (5%) on carbon catalyst	5.0 g

The autoclave is purged and pressure-checked and the temperature is brought to 95°C. Hydrogen is admitted with agitation over a period of one hour (50 psig. pressure) until the theoretical amount has been introduced into the autoclave. The reaction mixture so obtained is then filtered, washed and distilled at 85°–87°C at 2–3 mm Hg to obtain Ketone I.

The pure 2,5,5 trimethylacetyl cycloheptane has tobacco, musky-sweaty, tea-like, woody and orris notes.

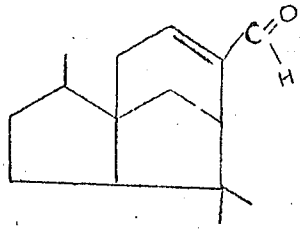
EXAMPLE II

The following mixture is prepared:

INGREDIENTS	PARTS
2,5,5 Trimethylacetyl Cycloheptane Prepared by the process of Example I	50
6,7-Dihydro-1,1,2,3,3-Pentamethyl-4(5H) Indanone Produced according to Prep. A of Swiss Patent 523,962	5
Cedrol Methyl Ether having the structure: 	15
Vertofix Coeur (Reaction product of Acetic Anhydride and Polyphosphoric acid with American Cedarwood Oil (Runeberg, Acta.Chem.Sound-15,592 (1961)	15
1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-Hexamethyl Cyclopenta-gamma-2-benzopyran	5

Grisalva
(Produced by the 50% sulfuric acid treatment of 3-Ethyl-1[2,2,6-Trimethyl Cyclohexene-5-yl-1] hexen-3-ol-6)

Cedrenal
having the structure:



Mixture containing primarily Methyl-2,6,10-Trimethyl-2,5,9-dodecatrien-1-yl-Ketone, produced according to the process of Example I of Canadian Patent 804,592

Sandalwood E.I.

8,9 Epoxy Cedrane

Addition of the 2,5,5 Trimethylacetyl Cycloheptane to this woody aroma composition imparts to it a persistent, strong tobacco, musky sweaty note which is highly desirable.

EXAMPLE III

Preparation of Soap Composition

One hundred grams of soap chips are mixed with one gram of the perfume composition of Example II until a substantially homogeneous composition is obtained. The perfumed soap composition manifests an excellent tobacco musky woody odor character.

EXAMPLE IV

Preparation of a Detergent Composition

A total of 100 grams of a detergent powder is mixed with 0.15 grams of the perfume composition of Example II until a substantially homogeneous composition is obtained. This composition has an excellent tobacco musky woody odor.

EXAMPLE V

Preparation of a Cosmetic Powder Composition

A cosmetic powder is prepared by mixing in a ball mill 100 grams of talcum powder with 0.25 gram of the product obtained from the process of Example I. It has an excellent tobacco musky sweaty odor.

EXAMPLE VI

Perfumed Liquid Detergent

Concentrated liquid detergents with a rich tobacco musky odor are prepared containing 0.10%, 0.15% and 0.20% of 2,5,5 Trimethylacetyl Cycloheptane. They are prepared by adding and homogeneously mixing the appropriate quantity of 2,5,5 Trimethyl acetyl Cycloheptane in the liquid detergent. The detergents all possess a tobacco-musky fragrance, the intensity increasing with greater concentration of 2,5,5 Trimethylacetyl Cycloheptane of this invention.

EXAMPLE VII

The compound produced by the process of Example I: 2,5,5 Trimethylacetyl Cycloheptane is incorporated in a cologne at a concentration of 2.5% in 85% aqueous ethanol; and into a handkerchief perfume at a concentration of 20% (in 95% aqueous ethanol). A distinct and definite strong tobacco, musky, sweaty fragrance is imparted to the cologne and the the handkerchief perfume.

EXAMPLE VIII

The composition of Example II is incorporated in a cologne at a concentration of 2.5% in 85% aqueous ethanol; and into a handkerchief perfume at a concentration of 20% (in 95% aqueous ethanol). The use of the 2,5,5 Trimethylacetyl Cycloheptane in the composition of Example II affords a distinct and definite strong tobacco, musky, sweaty note to the handkerchief perfume and cologne.

What is claimed:

1. A perfume composition comprising from 3.0% up to 50% by weight of said composition of 2,5,5 trimethylacetylcycloheptane, and a suitable perfume carrier.

2. A perfumed article comprising from 0.1% up to about 10% by weight of said perfumed article of 2,5,5 trimethylacetylcycloheptane, and a suitable perfume carrier.

3. A perfumed article of claim **2** wherein the carrier is selected from the group consisting of:

a cosmetic powder; and

a mixture of ethanol and water.

4. A cologne comprising about 25% of 2,5,5-trimethylacetylcycloheptane in a solution of 85% ethyl alcohol and 15% water.

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