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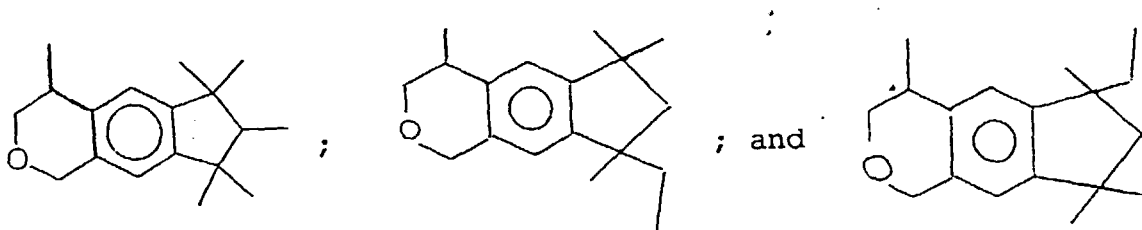
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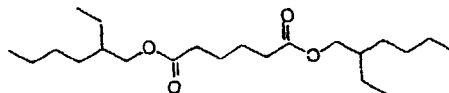
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(54) **Composition containing di(2-ethylhexyl) adipate and tricyclic isochroman compounds and use thereof in enzyme-active detergents and fabric softeners**

(57) Described is a mixture of (i) tricyclic isochroman compounds having the structures:

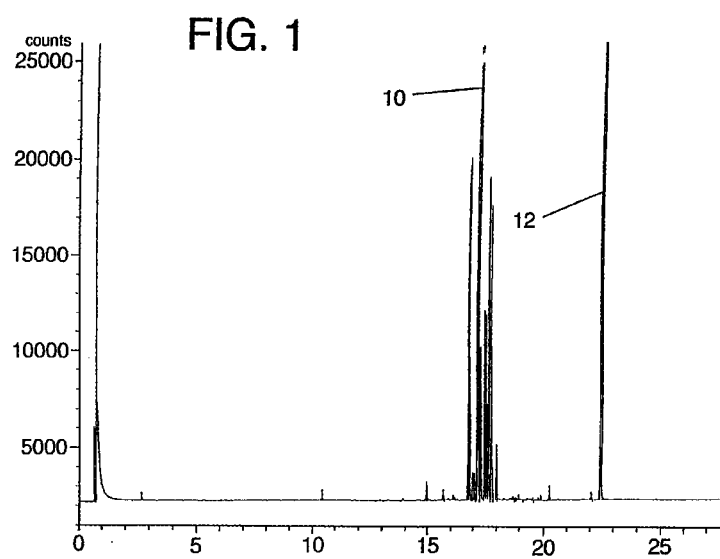


and (ii) di(2-ethyl hexyl) adipate having the structure:



with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being from about 1:1 up to about 3:1 as well as a method for perfuming fabrics using such a composition which comprises washing the fabrics in the presence of a lipase-containing detergent and, optionally, subsequently treating the fabric with a fabric softener.

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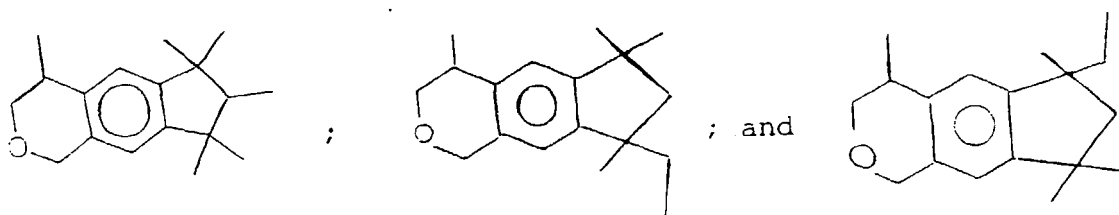
Description

BACKGROUND OF THE INVENTION

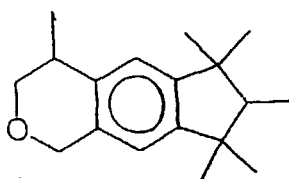
[0001] Our invention relates to the field of perfumery and, more particularly, to improved fragranced detergents and fabric softeners.

[0002] The use of enzymes in fabric detergents in order to improve their efficiency has been known for a number of years. Among such enzymes, lipases are particularly preferred as a result of the capacity to hydrolyze the fat materials on the dirty linen and thus to facilitate its cleaning. However, it is known that malodor problems can occasionally occur after certain application conditions and in order to solve these malodor problems, techniques exist for carefully choosing the perfume ingredients incorporated in the detergents and which, following a washing, are deposited on the fabrics, for example, European Patent No. 430,315. Accordingly, appropriate perfuming of such detergents appears to be extremely important.

[0003] A substantive fragrance ingredient that is desired to be incorporated into such fabric detergents for fragrancing same and such fabric softeners for fragrancing same is the tricyclic isochroman known as GALAXOLIDE® (trade-mark of International Flavors & Fragrances Inc. of New York, New York) which is a mixture of compounds having the structures:



with the majority of the isomers being the compound having the structure:

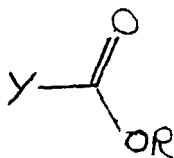


However, in very high concentrations (e.g., greater than 50%), GALAXOLIDE® has extremely high viscosity and at lower temperatures exists in the crystalline state making it difficult for incorporation into fragrances. In the prior art, the GALAXOLIDE® has been combined with other materials in order to form "eutectic" compositions containing GALAXOLIDE® and other materials, for example, benzophenone.

[0004] However, a need exists for creating a low viscosity, high concentration form of GALAXOLIDE® in fragrances which are used in fabric detergents and fabric softeners, particularly where the detergent and softener systems involve the use of enzymes such as lipase.

[0005] There is a need for such detergents and fabric softeners to be able to impart to the fabrics a long lasting odor as stated, supra, such the user perceives this odor for a considerable time after the textiles have been washed and subsequently dried. To this end, it has been known to use in the fabric detergents and softeners perfuming ingredients which have a good tenacity on the fabric, i.e., ingredients whose odor once imparted to the textiles upon the washing can be perceived by the consumer for several days. However, there are many perfuming substances known for their extremely pleasant odors and, namely, a quality of "freshness" often associated with the notion of cleanliness, which substances are unfortunately not very tenacious or even not tenacious at all on fabrics such as their perfuming effect can only be perceived very briefly, at the most, for a few hours following the washing and drying operations. In general, U.S. Letters Patent No. 5,649,979 issued on July 22, 1997 indicates that the fragrance effect of such substances is prolonged and thus the "freshness" of the fabrics has been prolonged. Thus, U.S. Letters Patent No. 5,649,979 indicates that a "novel" solution to the problem has been achieved where a process for perfuming textiles washed with detergents containing lipases has been discovered. U.S. Letters Patent No. 5,649,979 establishes that by adding particular ingredients to the fabric detergent and/or fabric softener that is subsequently applied, one could distinctly improve the odor of the fabrics treated with these products and prolong the fragrance of the fabrics after drying.

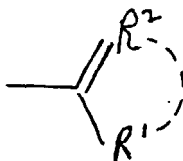
[0006] U.S. Letters Patent No. 5,649,979 provides a process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener with the process being characterized in that the detergent and/or fabric softener contains a compound of the formula:



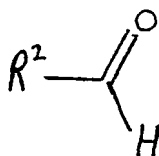
wherein:

(a) **R** represents a radical derived from a fragrant alcohol of formula **ROH** and **Y** represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a $-(\text{CH}_2)_n\text{COOR}$ group wherein **R** is defined as above and n is an integer from 0 to 6; or

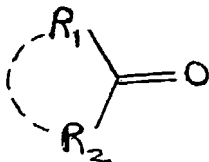
(b) **Y** represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical and **R** represents a group of the formula:



wherein either **R**¹ represents hydrogen and **R**² represents an alkylidene radical derived from a fragrant aldehyde of the formula:



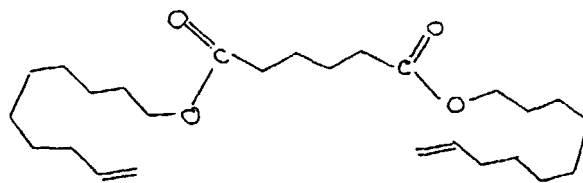
or **R**² represents an alkylidene radical and **R**¹ an alkyl radical, **R**¹ and **R**² being then derived from a fragrant ketone of the formula:



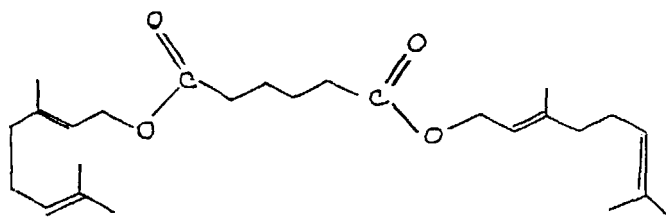
and, optionally, being part of a ring such as indicated by the dashed line which contains 5 to 18 carbon atoms and can be substituted.

Examples relevant to our invention as set forth in U.S. Letters Patent No. 5,649,979 are:

the compound having the structure:

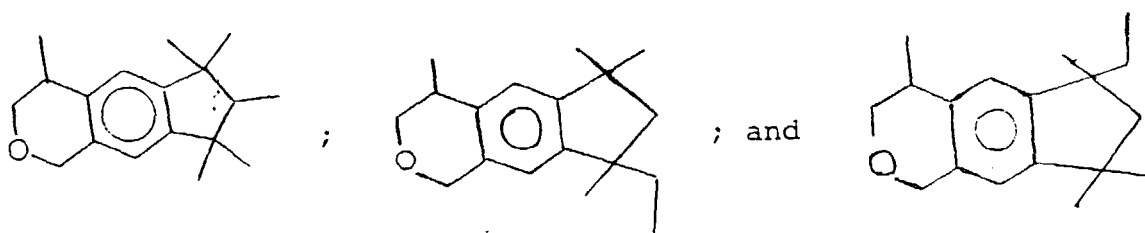


(at column 12, line 3) and the compound having the structure:



(at column 12, line 57), both compounds being adipic acid esters.

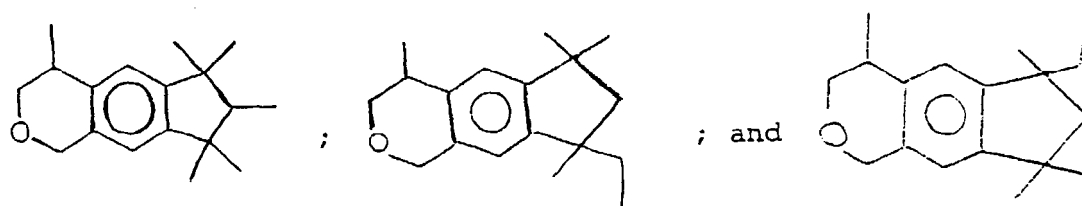
[0007] However, nothing in U.S. Letters Patent No. 5,649,979 and nothing in the prior art indicates the creation of the synergistic effect of formation of the mixture of the two fragrant materials in situ, 2-ethyl hexanol and the mixture of tricyclic isochromans having the structures:



(GALAXOLIDE®) as a result of admixing in proportions of from about 1:1 up to about 3:1 (weight:weight) of di(2-ethyl hexyl) adipate and GALAXOLIDE® and nothing in the prior art indicates that as a result of the discovery of such a mixture the concentration of pure GALAXOLIDE® can be greatly increased, particularly when used with fabric detergent and fabric softener systems which use enzymes.

THE INVENTION

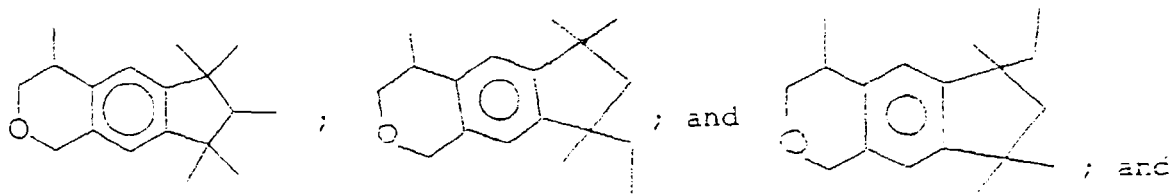
[0008] Our invention concerns a process for the creation of a liquid fragrance composition that is easily used in forming still further liquid fragrance compositions, containing GALAXOLIDE®, a mixture of compounds having the structures:



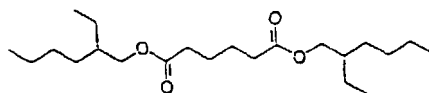
comprising the steps of admixing a sufficient quantity of di(2-ethyl hexyl) adipate with substantially pure GALAXOLIDE[®], thereby creating a composition in the liquid phase having a viscosity in the range of from about 180 centipoises (cps) up to about 250 cps at 25°C.

[0009] More specifically, our invention is directed to a mixture consisting essentially of:

(i) tricyclic isochroman compounds having the structures:



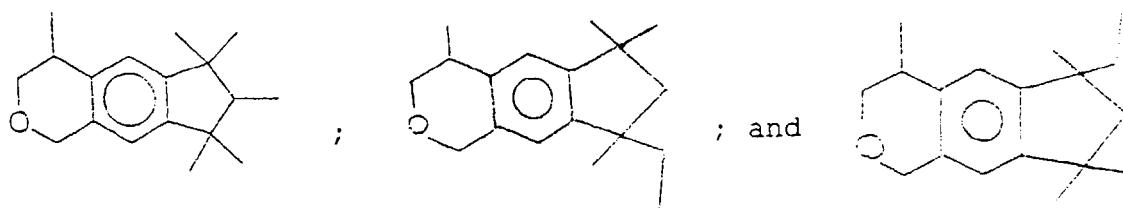
(ii) di(2-ethyl hexyl) adipate having the structure:



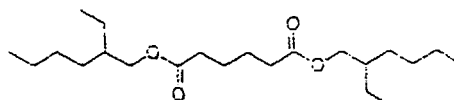
with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being from about 1:1 up to about 3:1.

More preferably, the foregoing mixture of tricyclic isochroman and di(2-ethyl hexyl) adipate is 2:1 and the viscosity thereof is about 210 cps at 25°C.

[0010] Our invention is further directed to a laundry detergent or fabric softener comprising the above-identified perfuming composition as an active perfume ingredient. Our invention is further directed to a method for perfuming fabrics which comprises washing the fabrics in the presence of a lipase-containing detergent and, optionally, subsequently treating the fabric with a fabric softener wherein the detergent or fabric softener comprises as an active perfume ingredient the above-identified range of mixtures of tricyclic isochroman compounds having the structures:



and di(2-ethyl hexyl) adipate having the structure:



with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being from about 1:1 up to about 3:1.

[0011] Relevant to the uniqueness of the mixture of GALAXOLIDE[®] and the di(2-ethyl hexyl) adipate of our invention is the fact that the calculated $\log_{10}$ [n-octanol/water partition coefficient] (hereinafter referred to as "C $\log_{10}P$ ") is in the range of from about 4.4 up to about 7.0 with the low part of the C $\log_{10}P$ range being for the mixture have the ratio of GALAXOLIDE[®]:di(2-ethyl hexyl) adipate of 3:1, and the high part of the range (7.0) being for the ratio of GALAXOLIDE[®]:di(2-ethyl hexyl) adipate of 1:1. Accordingly, for the di(2-ethyl hexyl) adipate - GALAXOLIDE[®] compositions of

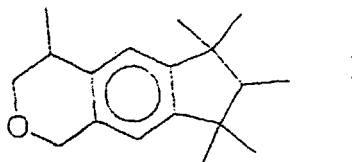
matter of our invention, the following inequality applies:

$$4 \cdot 4 \leq \log_{10} P \leq 7 \cdot 0.$$

5 The $\log_{10} P$ of the most preferred composition of our invention, the composition wherein the ratio of GALAXOLIDE[®]:di(2-ethyl hexyl) adipate is 2:1, is 6.61.

[0012] The standard for the optimum GALAXOLIDE[®] aroma is for the main peak of the GALAXOLIDE[®] (that is for the presence of the compound having the structure:

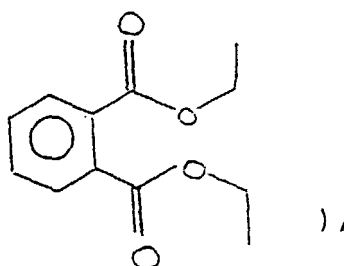
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to be between 52% and 56% by weight of all components obtained in a GLC (liquid chromatography) sample. Thus, when using a diethyl phthalate solvent (with diethyl phthalate having the structure:

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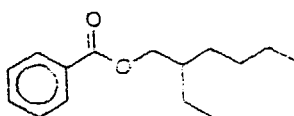


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the equation of **G** vs. **W** is: $G = 0 \cdot 7143 W + 6 \cdot 29$. On the other hand, when using in place of the di(2-ethyl hexyl) adipate, octyl benzoate having the structure:

35



40

the equation of **G** vs. **W** is: $G = 0 \cdot 7 W + 3$.

[0013] When using di(2-ethyl hexyl) adipate (the mixture of our invention), the equation is: $G = 0 \cdot 7059 W + 4 \cdot 71$.

45 [0014] Thus, the mixture of GALAXOLIDE[®] and di(2-ethyl hexyl) adipate in combination provides a new aroma containing notes of both 2-ethyl hexanol and GALAXOLIDE[®], but also enables the GALAXOLIDE[®] to be used in much greater proportions than has been used before, for example, with diethyl phthalate.

[0015] In addition, the process of our invention is advantageous whenever other fragrance compounds are also used which show a weak tenacity on fabrics and which are compatible from an olfactory standpoint with GALAXOLIDE[®] and 2-ethyl hexanol.

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[0016] The GALAXOLIDE[®] - di(2-ethyl hexyl) adipate of our invention can be added to detergents and fabric softeners either as such or in admixture with other perfuming ingredients, solvents or adjuvants of current use in perfumery. The concentrations in which they can be added to the detergents and fabric softeners according to the inventions have the values usual in the art for this type of product. The skilled person in the art is quite able to select such values as a function of the nature of the product to be perfumed and of the desired olfactive effect. By way of example, concentrations of the order of from about 0.005 up to about 7% by weight of the mixture of GALAXOLIDE[®] and di(2-ethyl hexyl) adipate relative to the weight of detergent and/or fabric softener composition are useful.

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[0017] The fabric detergents and softeners, according to our invention, can take the form of powders or granular

solids, bars, pastes as well as aqueous or non-aqueous liquids and contain the usual ingredients for this type of product. Thus the detergents can typically contain, in addition to the lipase enzyme (as specifically disclosed in European Patent No. 430,315) active anionic, cationic, nonionic or zwitterionic surfactants as well as filling agents, bleaching agents, confining agents and other ingredients of current use in the detergent bases intended for washing cloth such as linen. Many examples of such compositions are found in the art and in literature, for example, European Patent No. 397,245. By way of example, a base detergent of this type to which there is added the lipase in the desired concentrations can have the following composition (origin: Henkel KGaA of Düsseldorf, Germany):

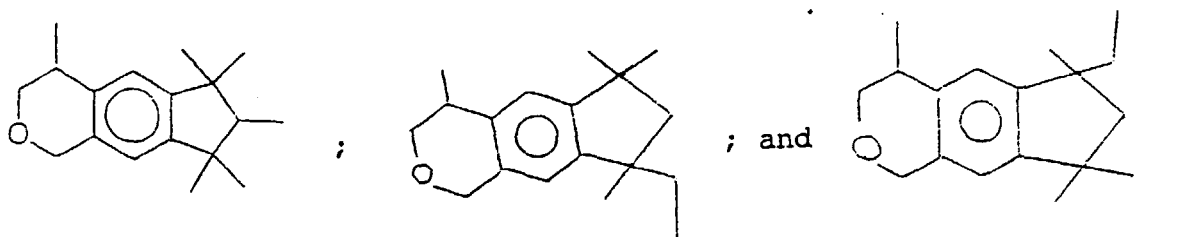
Ingredients	Parts by Weight
Linear sodium alkyl benzene sulfonate (average length of the alkyl chain: C _{11.5})	8.0
Ethoxylated tallow alcohol	2.9
Sodium triphosphate	43.8
Sodium silicate	7.5
Magnesium silicate	1.9
Carboxymethylcellulose	1.2
Sodium ethylenediaminetetraacetate	0.2
Sodium sulfate	21.2
Water	13.3
Total	100.0

[0018] Similar considerations apply to the fabric softener bases useful in the practice in our invention which typically contain cationic softening ingredients as cited in European Patent No. 397, 245.

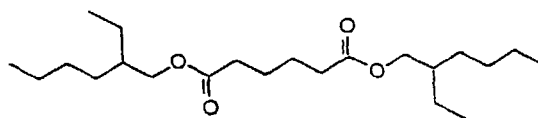
BRIEF DESCRIPTION OF THE DRAWINGS

[0019]

Figure 1 is the GLC profile for a mixture of two parts GALAXOLIDE® (a mixture of compounds having the structures:



and one part di(2-ethyl hexyl) adipate having the structure:

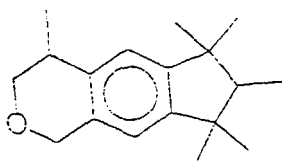


(conditions: 250 mm capillary silica column programmed from 50 up to 250°C at 10°C per minute).

Figure 2 is another GLC profile for the 2:1 mixture of GALAXOLIDE® di(2-ethyl hexyl) adipate (conditions: 200 mm

CARBOWAX® 20M column programmed from 50 up to 250°C at 10°C per minute).

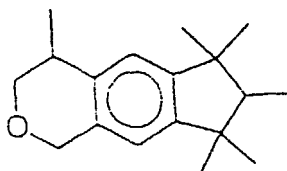
Figure 3 is a graph showing on the Y axis the GLC percent main peak for GALAXOLIDE® (the main peak being for the compound having the structure:



vs. weight percent GALAXOLIDE® undiluted in the mixture on the X axis.

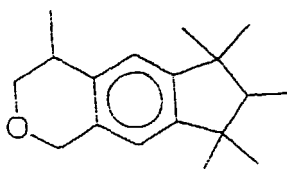
DETAILED DESCRIPTION OF THE DRAWINGS

[0020] Referring to Figure 1, the peak indicated by reference numeral 10 is the peak for the compound having the structure:

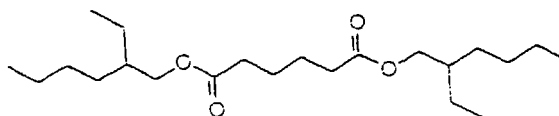


The peak indicated by reference numeral 12 is the peak for the di(2-ethyl hexyl) adipate.

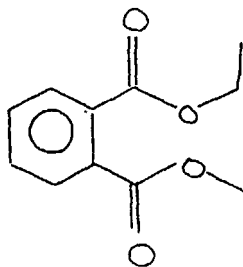
[0021] Referring to Figure 2, the peak indicated by reference numeral 20 is the peak for the compound having the structure:



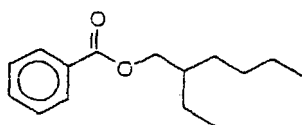
The peak indicated by reference numeral 22 is the peak for the di(2-ethyl hexyl) adipate



[0022] Referring to Figure 3, the Y axis for the GLC percent for the main peak of GALAXOLIDE® is indicated by reference numeral 36 and the weight percent GALAXOLIDE® (undiluted) on the X axis is indicated by reference numeral 35. The graph indicated by reference numeral 30 is the graph for the mixtures of GALAXOLIDE® and diethyl phthalate having the structure:

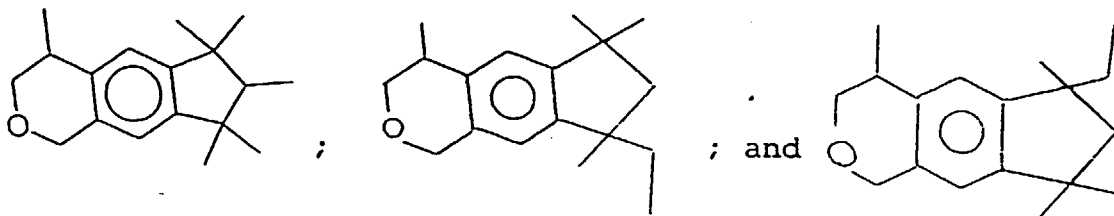


The graph indicated by reference numeral 32 is the graph for mixtures of GALAXOLIDE® and di(2-ethyl hexyl) adipate. The graph indicated by reference numeral 34 is the graph for mixtures of GALAXOLIDE® and 2-ethyl hexyl benzoate having the structure:



[0023] The use of the di(2-ethyl hexyl) adipate with the tricyclic isochroman:

(iv) enables the creation of novel mixtures of the fragrance material, 2-ethyl hexanol and the tricyclic isochroman mixture defined according to the three structures:



when using an enzyme detergent via control release mechanisms for the fragrance composition.

(i) enables the concentration of the initial tricyclic isochroman to be substantially increased (and still be useful in conjunction with the mixing of same with other fragrances);

(ii) significantly eliminates any adverse viscosity problem caused by the increasing concentration of the tricyclic isochroman;

(iii) facilitates the use of high concentrations of tricyclic isochroman in connection with fabric softeners and detergents; and

EXAMPLE I

TEST ON FABRICS

[0024] A number of tests were carried out on fabrics under a variety of conditions, with the fabrics having been treated according to the following method:

[0025] A standard cotton swatch of 32 grams is placed in a LINITEST® type vessel containing 1.3 grams of a standard powder detergent base comprising 1% by weight of lipase (LIPOLASE® 100T manufactured by Novo Nordisk S.A. of Denmark) and 260 ml water. The cotton swatch is washed for 30 minutes at 40°C. It is then taken out of the vessel

and rinsed with 3 x 200 ml portions of water. It is then plunged for 5 minutes in 200 ml water containing 0.6 grams of a fabric softener base which contains a certain percentage by weight comprised between 0.5 and 1% of a mixture of GALAXOLIDE® and di(2-ethyl hexyl) adipate or, when applicable, of the corresponding alcohol which is 2-ethyl hexanol-1. The cotton swatch is then spin-dried without having been used and dried on a clothes line.

[0026] This method is equivalent to a washing in a real machine for washing 5 kg of linen in approximately 20 liters of water with a rinsing of 4 x 20 liters of water, the fabric softener being applied in the last rinsing water. The fabric softener base used has the following compositions:

Ingredients	Parts by Weight
ARQUAD® 2HT (75%) (trademark of Akzo Corporation of the Netherlands)	5.00
Formalin (40%)	0.20
Demineralized water	94.80
Total	100.00

[0027] In two separate tests, the cotton swatches were treated according to this general method, using as an additive to the fabric softener, respectively, (i) a 2:1 mixture of GALAXOLIDE®:di(2-ethyl hexyl) adipate, 1% in test **A**; and (ii) a 2:1 weight:weight mixture of GALAXOLIDE®:2-ethyl hexanol-1 in test **B**.

[0028] The two swatches were submitted for a blind panel test evaluation 24 hours after having been taken out of the LINITEST® apparatus. Each member of the panel is then requested to smell a strip which had been dipped into 2-ethyl hexanol-1 in order to cause them to be familiar with the odor of this particular component. Each member of the panel was then asked to smell the two cotton swatches as set forth above and state whether they detect the nuances of (i) 2-ethyl hexanol-1 and (ii) GALAXOLIDE®.

[0029] With the swatches of tests **A** and **B**, all members of the panel detected all nuances of GALAXOLIDE®.

[0030] Seventy percent of the panel members detected all of the nuances of 2-ethyl hexanol-1 in test **A**. Twenty-five percent of the panel members detected all of the nuances of 2-ethyl hexanol-1 in test **B**.

[0031] A further evaluation of the two swatches was carried out by the same panel under identical conditions 24 hours later, that is, 48 hours after taking the swatches out of the LINITEST® apparatus. None of the panel members could identify the aroma of 2-ethyl hexanol-1 on the swatch of test **B**, whereas 50% of the panel members identified all of the nuances of 2-ethyl hexanol-1 on the swatch coming out of test **A**.

[0032] Similar results are obtained when the above-mentioned mixture of GALAXOLIDE® and di(2-ethyl hexyl) adipate were present in the fabric softener at a rate of 0.5% by weight.

EXAMPLE II

FABRICS TREATED IN A WASHING MACHINE

[0033] A base perfume composition is prepared by admixing the following ingredients:

Ingredients	Percent by Weight
Vetiver Venezuela	30
Phenylethyl alcohol	25
Benzylpropionate	20
10-Undecenal	8
Hexylcinnamic aldehyde	80
Methyl anthranilate	20
Verdyl acetate	30
Trans, trans- δ -damascone	12

(continued)

Ingredients	Percent by Weight
Eugenol	8
Isoeugenol	20
Orange essential oil	25
Benzylsalicylate	40
Phenylsalicylate	25
α -Terpeniol	20

[0034] Two compositions are prepared with the aforementioned base as follows:

Composition A:

500 parts by weight of fragrance composition and 150 parts by weight of the mixture of GALAXOLIDE®:di(2-ethyl hexyl) adipate, in a weight ratio of 2:1; and

Composition B:

500 parts by weight of the fragrance as set forth, supra, and 150 parts by weight of a 2:1 mixture of GALAXOLIDE® (100%) and 2-ethyl hexanol-1 (2:1).

[0035] The resulting compositions are then each added at the rate of 0.7% by weight to a non-perfumed fabric softener base to prepare two samples: **A** and **B** of a perfumed softener.

[0036] In two identical washing machines, two standard batches of 1,400 grams each of cotton textiles were treated. Each machine was charged with 130 grams of a standard, unperfumed detergent base containing 1% of lipase (LIPOLASE® 100T of the Novo Nordisk S.A. of Denmark).

[0037] To one machine, 100 grams of the perfumed softener **A** was added. To the second machine, 100 grams of the perfumed softener **B** was added.

[0038] After washing, each batch of textiles was sampled by a blind panel of 25 individuals. Each member of the blind panel identified the aroma of the batch treated with perfumed softener samples **A** and **B**.

[0039] As a result of this test, 20 panelists indicated a distinct difference between the quality, intensity and substantivity of the aroma created in a textile batch when using sample **A** compared to using sample **B** as follows (on a scale of 1-100):

Sample	Intensity	Substantivity	Quality
A	8	9	9
B	3	4	8

[0040] When the evaluation was repeated after 48 hours of air drying, 50% of the panelists detected a distinction and that distinction is as follows:

Sample	Intensity	Substantivity	Quality
A	6	8	9
B	1	2	7

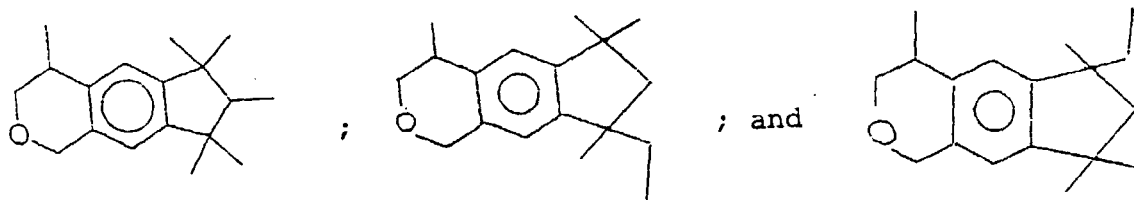
[0041] The features disclosed in the foregoing description, in the following claims and/or in the accompanying drawings may, both separately and in any combination thereof, be material for realising the invention in diverse forms thereof.

[0042] In the present specification "comprise" means "includes or consists of" and "comprising" means "including

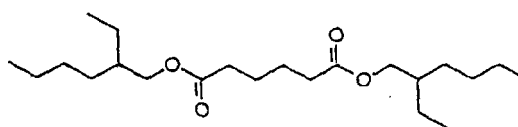
or consisting of".

Claims

1. A mixture consisting essentially of (i) tricyclic isochroman compounds having the structures:

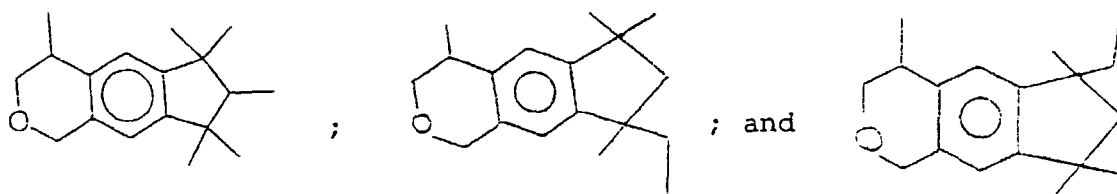


and (ii) di(2-ethyl hexyl) adipate having the structure:

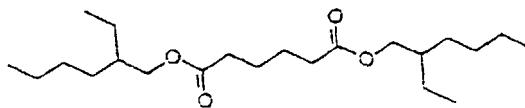


with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being from about 1:1 up to about 3:1.

2. The mixture of Claim 1 wherein the weight ratio of tricyclic isochroman:di(2-ethyl hexyl) adipate is 2:1 and the viscosity thereof is about 210 centipoises (cps) at 25°C.
3. A laundry detergent or fabric softener composition comprising a perfuming composition comprising as an active perfuming ingredient, the composition of Claim 1.
4. A laundry detergent or fabric softener composition comprising a perfuming composition comprising as an active perfuming ingredient, the composition of Claim 2.
5. A method for perfuming fabric which comprises washing said fabric in the presence of a lipase-containing detergent and, optionally, subsequently treating said fabric with a fabric softener wherein said detergent and said fabric softener each comprise as an active perfuming ingredient the mixture of Claim 1.
6. A method according to Claim 5 wherein said fabrics, after the washing cycle, are treated with a fragrance composition containing a mixture of (i) tricyclic isochromans having the structures:

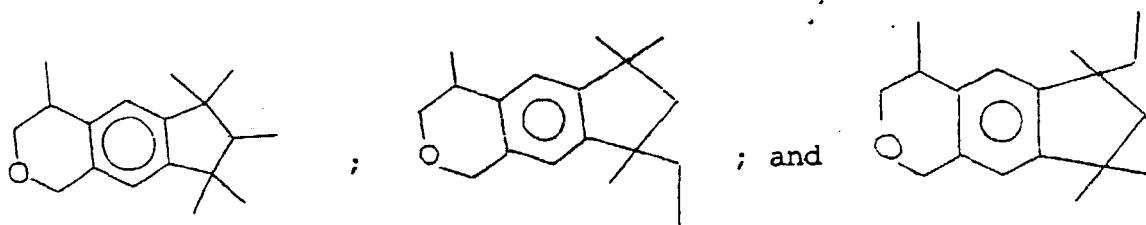


and (ii) di(2-ethyl hexyl) adipate having the structure:

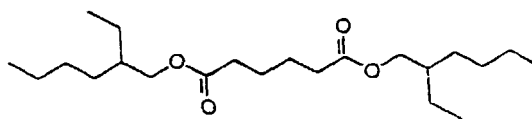


with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being from about 1:1 up to about 3:1 and, in addition, a fragrance composition containing components other than tricyclic isochromans, 2-ethyl hexanol-1 or di(2-ethyl hexyl) adipate.

7. A method for perfuming fabric which comprises washing said fabric in the presence of a lipase-containing detergent and, optionally, subsequently treating said fabric with a fabric softener wherein said detergent and said fabric softener each comprise as an active perfuming ingredient the mixture of Claim 2.
8. A method according to Claim 5 wherein said fabrics, after the washing cycle, are treated with a fabric softener composition containing a mixture of (i) tricyclic isochroman compounds having the structures:

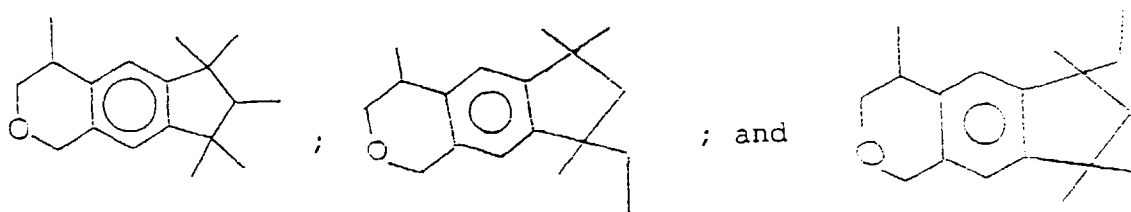


and (ii) di(2-ethyl hexyl) adipate having the structure:



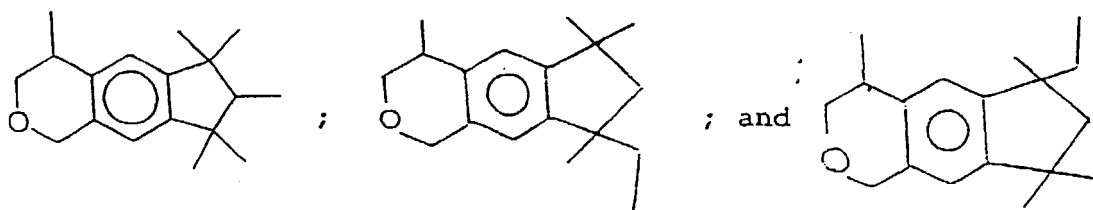
with the range of weight ratios of tricyclic isochroman:di(2-ethyl hexyl) adipate being 2:1 and the viscosity of the tricyclic isochroman:di(2-ethyl hexyl) adipate being about 210 cps at 25°C.

9. A fragrance composition consisting essentially of 2-ethyl hexanol-1 and the tricyclic isochroman composition containing the compounds having the structures:

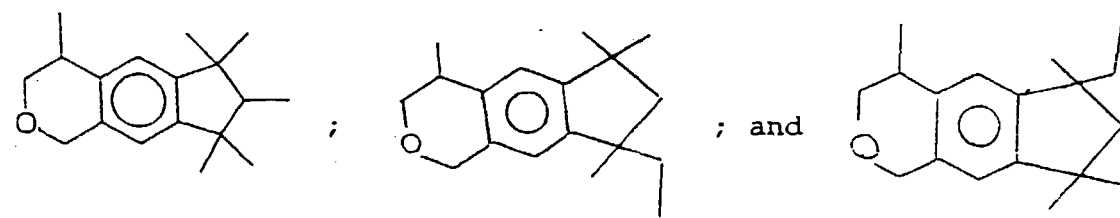


wherein the weight ratio of tricyclic isochroman:2-ethyl hexanol-1 is in the range of from about 1:1 up to about 3:1.

10. A process for creation of a liquid fragrance composition Containing a mixture of compounds having the structures:



and 2-ethyl hexanol-1 comprising the steps of admixing a sufficient quantity of di(2-ethyl hexyl) adipate with a substantially pure mixture of the compounds having the structures:



- 25
11. A mixture or composition according to any one of Claims 1 to 4 or Claim 9 having a $C \log_{10} P$ range defined according to the inequality $4.4 \leq \log_{10} P \leq 7.0$.
- 30
12. A method for perfuming fabric which comprises washing said fabric in the presence of a detergent and, optionally, subsequently treating said fabric with a fabric softener wherein said detergent and said fabric softener each comprise as an active perfuming ingredient the fragrance composition of Claim 9.
13. A process according to Claim 10 further comprising the step of admixing a lipase.
- 35
14. A process according to Claim 10 or Claim 13 wherein the liquid fragrance composition has a viscosity in the range of from about 180 cps up to about 250 cps at 25°C.
- 40
15. A process according to Claim 10, 13 or 14 wherein the liquid fragrance composition has a $C \log_{10} P$ range defined according to the inequality $4.4 \leq \log_{10} P \leq 7.0$.

FIG. 1

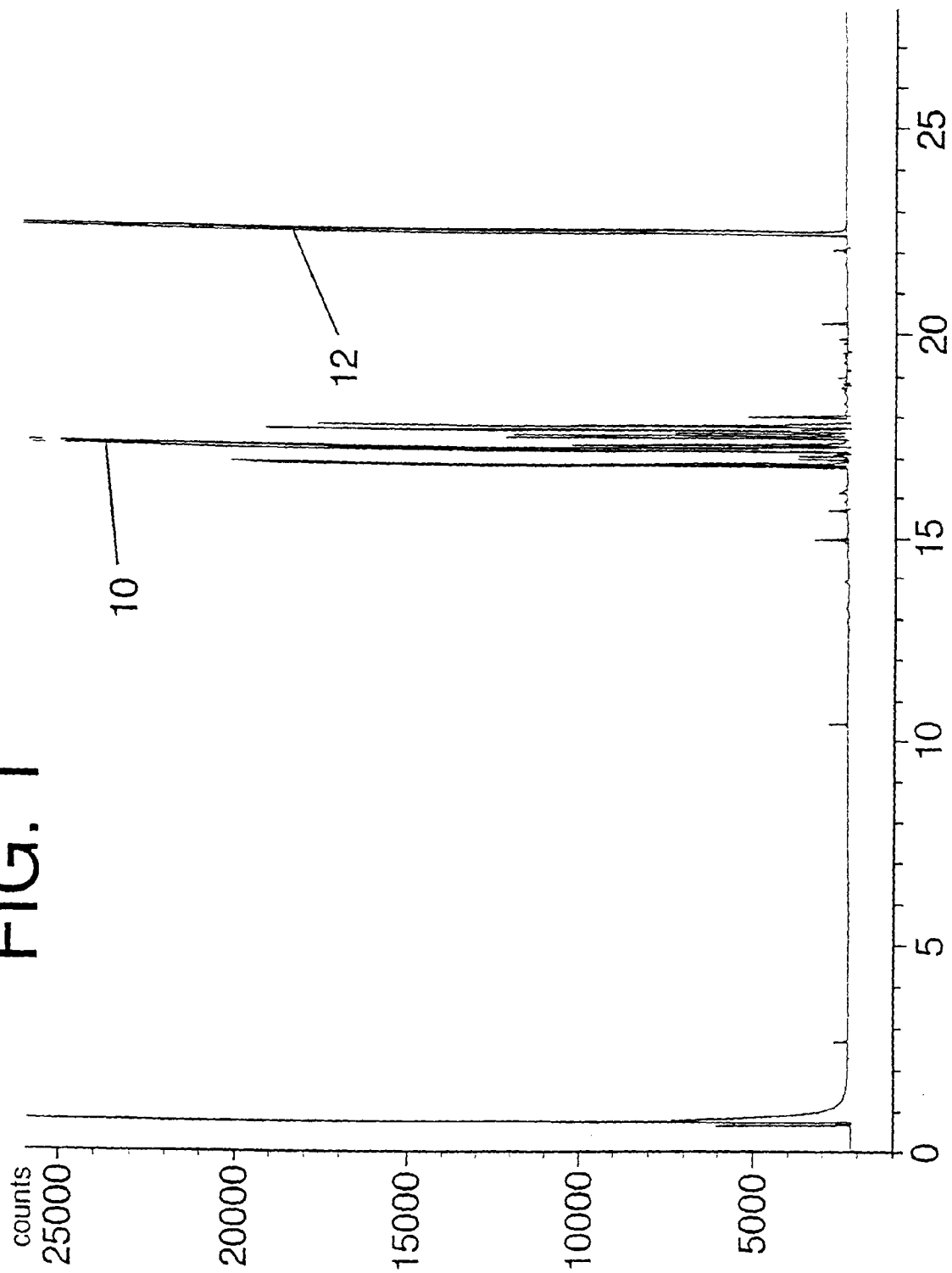


FIG. 2

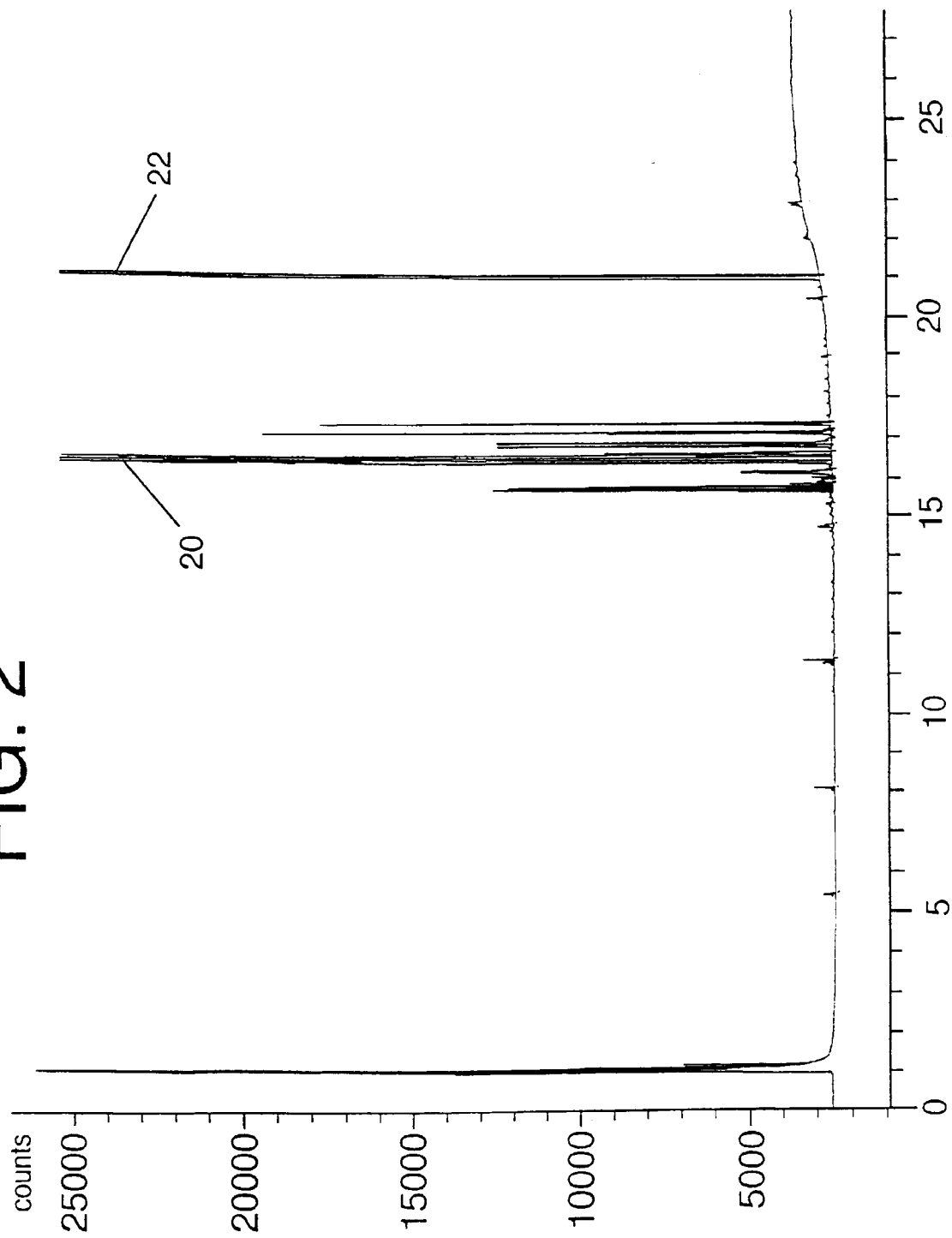


FIG. 3

