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(71) Applicant *(for all designated States except US)*: **TAKASAGO INTERNATIONAL CORPORATION** [JP/JP]; 5-37-1, Kamata, Ohta-ku, Tokyo, 144-8721 (JP).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **DEROSA, Frank** [US/US]; 927 Cain Court, Belle Mead, NJ 08502 (US). **LANKIN, Michael, E.** [US/US]; 118 Cregar Road, High Bridge, NJ 08829 (US). **LUPO, Andrew, T.** [US/US]; 8 Powell Road, Emerson, NJ 07630 (US).

(74) Agents: **ROBINSON, Joseph, R.** et al.; Darby & Darby P.c., P.o. Box 770, Church Street Station, New York, NY 10008-0770 (US).

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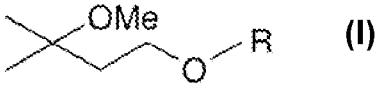
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(54) Title: SUBSTITUTED BUTANOL DERIVATIVES AND THEIR USE AS FRAGRANCE AND FLAVOR MATERIALS



(I)

(57) Abstract: The present invention is related to substituted butanol derivatives of the formula; wherein R is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl an uosubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted Cu= branched chain alkenyl, an unsubstituted or substituted C_{3-6} cycioalkyl, an unsubstituted or substituted C_{1-6} alkoxy, nitiile, halo, amino, an unsubstituted or substituted C_{1-6} alkyiamino, w. unsubstituted or substituted C_{1-6} dialkyfaraino, carboxy-C_{1-6} alkyiamino, carboxy-C_{1-6} di alkyl amino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyi, an unsubstituted or substituted C_{1-6} aikylcarbonyi, an unsubstituted or substituted C_{1-6} aikylcarboxy, ao unsubsiituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy. carboxamido, an unsubstituted or substituted C1-6 alkylcarboxarøido or an unsubstituted or substituted C1-6 dia'lkylcarboxamido. Such compounds are useful in flavor or flavor compositions.

SUBSTITUTED BUTANOL DERIVATIVES AND THEIR USE AS FRAGRANCE AND FLAVOR MATERIALS

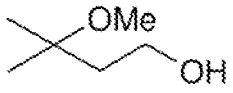
FIELD OF THE INVENTION

[001] The present invention relates to compounds useful as fragrance or flavor components in fragrance or flavor compositions.

BACKGROUND OF THE INVENTION

[002] The fragrance industry is constantly reliant on the development of new chemicals with favorable organoleptic properties to provide perfumers and other persons the capability of creating new, unique fragrances for applications such as personal care products, air care products, perfumes, colognes and the like.

[003] 3-methoxy-3-methyl-1-butanol, also known as Solfit™, is known in the perfume industry and has been applied in many consumer products (WO 9512379; JP 2001226246; JP 2005290236; JP 001104462). The structure of 3-methoxy-3-methyl-1-butanol is shown below:

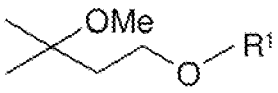


[004] Some esters derived from this alcohol have been employed as solvents for consumer products (EP 462605A2). Ether analogues of this alcohol have been used as intermediates to useful homoallylic alcohol entities (US 4,990,697).

SUMMARY OF THE INVENTION

[005] The present invention is directed to the synthesis and application of 3-methoxy-3-methyl-1-butanol derivatives having unique and desired odor or organoleptic characteristics. The compounds of the present invention can be employed alone or incorporated as perfumery ingredients to enhance already existing fragrance compositions, solvents, media and the like.

[006] In one embodiment, the present invention provides fragrance compounds of the formula (I),



(I)

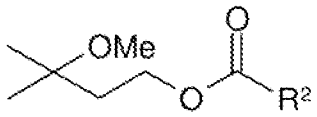
wherein R^1 is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{3-6} cycloalkyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, an unsubstituted or substituted phenyl, an unsubstituted or substituted benzyl, an unsubstituted or substituted naphthyl, an unsubstituted or substituted aryl, an unsubstituted or substituted piperazino, an unsubstituted or substituted morpholinyl, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{6-12} arylamino, an unsubstituted or substituted C_{1-6} dialkylamino, an unsubstituted or substituted C_{6-12} diarylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted

C_{1-6} alkylcarbonyl, thio, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido, an unsubstituted or substituted C_{1-6} dialkylcarboxamido, an unsubstituted or substituted phenoxy, an unsubstituted or substituted benzyloxy, phenylcarbonyl, benzylcarbonyl, an unsubstituted or substituted nitrophenyl, C_{1-6} trialkylsilyl or nitro, an unsubstituted or substituted C_{1-12} straight chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain aryl sulfonate or an unsubstituted or substituted C_{1-12} branched chain aryl sulfonate.

[007] In a preferred embodiment, R^1 as shown in formula I above, is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{3-6} cycloalkyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{1-6} dialkylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted C_{1-6} alkylcarbonyl, an unsubstituted or substituted C_{1-6} alkylcarboxy, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido or an unsubstituted or substituted C_{1-6} dialkylcarboxamido.

[008] In a more preferred embodiment, R^1 is a C_{1-6} straight chain alkyl, C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{1-6} alkenyl, an unsubstituted or substituted C_{1-6} acyl, or aryl.

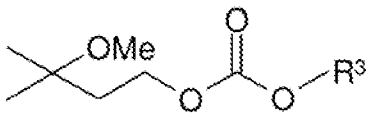
[009] In another embodiment, the invention provides fragrance compounds of the formula (II),



(II)

wherein R^2 is the same as described for R^1 above in paragraph 6. In an alternative embodiment, R^2 is as R^1 is described above in paragraphs 7 or 8. Additionally, R^2 may be hydrogen. Usually, the alkyl 3-methoxy-3-methyl-1-butanol esters represented by formula II have a strong fruity note associated with them.

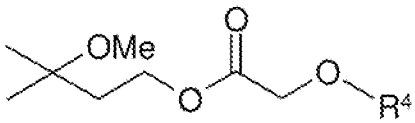
[010] In another embodiment, the invention provides fragrance compounds of the formula (III),



(III)

wherein R^3 is the same as described for R^2 above. Generally, the carbonate compounds represented by formula III have weak, varying odor characteristics but maintain a light and soft quality.

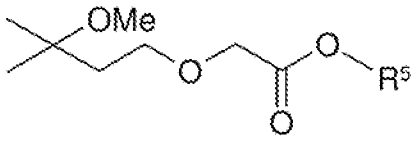
[011] In another embodiment, the invention provides fragrance compounds of the formula (IV),



(IV)

wherein R^4 is the same as described for R^2 above. For example, when the alcohol, 3-methoxy-3-methyl-1-butanol, is reacted with an alkoxy acetic acid, a compound of formula (IV) will result from the condensation reaction. Glycolates represented by formula IV generally have a consistent light, powdery musk property.

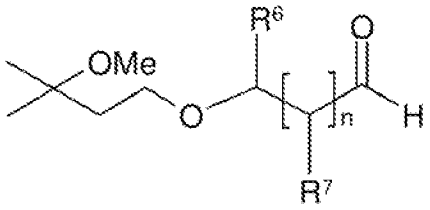
[012] In another embodiment, the invention provides fragrance compounds of the formula (V),



(V)

wherein R^5 is the same as described for R^2 above. Compounds encompassed by formula (V) often possess variations of plastic and green olfactory notes.

[013] In another embodiment, the invention provides fragrance compounds of the formula (VI),



(VI)

wherein R^6 and/or R^7 is a hydrogen or an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl group and n = 0-1. Preferably, R^6 and/or R^7 are hydrogen or a methyl group.

[014] Preferred fragrance compounds are set forth in the table below, in which R_n refers to the R group of the respective formula.

TABLE 1

Compound No.	Formula	R_n	Olfactory Description
1	I	$-CH_2CH(OCH_3)_2$	weak, marine-like, clean
2	I	$-CH_2CH=CH_2$	citrus, herbal, pine, sweet orange, candy
3	I	$-CH_2C(CH_3)=CH_2$	citrus, green, lemon, slight orange
4	I	$-CH_2CH=C(CH_3)_2$	green, chocolate, sweet, bitter
5	I	$-CH(CH_3)CH=CH_2$	strong, mint, citrus, rose
6	I	$-CH_2CH_2CH_3$	woody, floral, slightly bitter banana
7	I	$-CH_2CH(CH_3)_2$	woody, green pear, slight chocolate
8	I	$-CH_2CH_2CH(CH_3)_2$	chocolate, liqueur, sour,

Compound No.	Formula	R_8	Olfactory Description
			slightly pungent
9	I	$-CH_2CH_2C(OCH_3)(CH_3)_2$	mold, mildew
10	II	-H	woody, camphoraceous, dry, chemical
11	II	$-CH_2CH_3$	fruity, banana, floral, bubble gum
12	II	$-CH(CH_3)_2$	fruity, green, pear, apple, sour
13	II	$-C(CH_3)=CHCH_3$	sugary, sweet sap, green
14	III	$-CH_3$	weak, light, green, floral
15	III	$-CH_2CH=CH_2$	weak, soft chocolate, airy
16	III	$-CH_2CH_2CH_3$	weak, soft musk, light vanilla
17	IV	$-CH_3$	weak, fruity, slight coconut, powder musk
18	IV	$-CH_2CH_3$	powder musk, fresh, soft, light, clean
19	IV	$-CH(CH_3)_2$	very weak, powder musk
20	V	$-CH_3$	metallic green, plastic
21	V	$-CH_2CH_3$	vegetable green, spicy, plastic
22	V	$-CH(CH_3)_2$	green, licorice, slightly mint, fresh
23	VI	-H (R^6) (n=0)	fresh, melon, clean, floral, muguet, green
24	VI	$-CH_3$ (R^6) (n=0)	fresh, watery melon, clean, floral, muguet
25	VI	-H (R^6), -H (R^7) (n=1)	waxy, oily, muguet, light floral
26	VI	$-CH_3$ (R^6) –H (R^7) (n=1)	waxy, oily, fatty

[015] In one embodiment the fragrance compound is selected from 3-methoxy-3-methylbutyl 2-ethoxyacetate, 2-(3-methoxy-3-methylbutoxy)ethanal, 1-methoxy-1,1-dimethyl-3-(3-methylbut-2-enyloxy)propane, 1-methoxy-1,1-dimethyl-3-prop-2-enyloxypropane, and 1-methoxy-1,1-dimethyl-3-(1-methylprop-2-enyloxy)propane.

[016] In one aspect, the present invention provides a method to modify, enhance or improve olfactory and/or organoleptic property (e.g odor or flavor property) of a fragrance or flavor composition by adding to said composition an olfactory or organoleptic effective quantity of the compound of formulas (**I-VI**). In one

embodiment, a fragrance compound is added to a fragrance carrier, fragrance base or both to provide a fragrance composition. In an alternative embodiment, a flavor compound is added to a flavor carrier to provide a flavor composition. It is understood here also that the invention may be described as the use of any composition containing formulas (**I-VI**) in fragrance and/or flavor compositions.

DETAILED DESCRIPTION OF THE INVENTION

Fragrance Compositions

[017] The present invention is directed to the use of the above compounds as fragrances in a fragrance composition. The compounds can be incorporated alone, as a mixture of two or more of said compounds, or as an enhancer to an existing fragrance composition (discussed below). These compounds add a favorable olfactory effect to the desired product. The compounds are typically present in an amount of from about 0.001 to about 30.0 by weight of the total fragrance composition. Typically a more preferred embodiment would contain between 0.01% and 20% by weight and a most preferred embodiment would contain between 0.01% and 10% by weight. None of thes examples shown are meant to be limiting or restrictive on the use of the material as stated.

[018] One embodiment of the present inventions provides a method to modify, enhance or improve the odor properties of a fragrance composition by adding to said composition an olfactory effective quantity of the compound of formulas (**I-VI**). The invention may also be described as the use of any composition containing compound (**I-VI**) which can be advantageously employed in the fragrance industry as active ingredients.

[019] Such compositions may contain or consist of at least one ingredient selected from a group consisting of a fragrance carrier and a fragrance base. Such compositions may also consist of at least one fragrance adjuvant.

[020] Said fragrance carriers may be a liquid or a solid and typically do not significantly alter the olfactory properties of the fragrance ingredients. Some non-limiting examples of fragrance carriers include an emulsifying system, encapsulating materials, natural or modified starches, polymers, gums, pectins, gelatinous or porous cellular materials, waxes, and solvents which are typically employed in fragrance applications.

[021] Said fragrance base refers to any composition comprising at least one fragrance co-ingredient. In general, these co-ingredients belong to chemical classes such as, but not limited to: alcohols, aldehydes, ketones, esters, ethers, acetals, oximes, acetates, nitriles, terpenes, saturated and unsaturated hydrocarbons and essential oils of natural or synthetic origins.

[022] Table 2 provides an example of a formulated fragrance in which compounds of the present invention can be added.

Table 2. Fragrance Formulation (ingredients are listed in parts per formulation based on a total of 1000 parts by weight, and also shown as WT% by formula amount) %

MATERIAL	Parts / 1000	Wt %
Acetyl Tetralin	20	2.0
Ambretone	5	0.5
Benzy Acetate	100	10.0
Bergamot Synth	25	2.5
Citral Synth @ 10%	10	1.0
Citronellol, Laevo	40	4.0
Citronellyl Acetate, Laevo	5	0.5
Citronellyl Nitrile, Laevo	7	0.7
Cyclacet	45	4.5
Trepanol	15	1.5
DH Myrcenol	125	12.5
Dynascone @10%	5	0.5
Eugenol	5	0.5
Geranium Oil	10	1.0
Hedione	27	2.7
Heliotropine	8	0.8
Hindinol	5	0.5
Ionone, Beta	25	2.5
Iso Bornyl Methyl Ether	25	2.5
Iso E Super	25	2.5

Iso Propxy Ethyl Salicylate	10	1.0
Linalool Syn	85	8.5
Linalyl Acetate	15	1.5
Melonal @10%	5	0.5
Methyl Ionone, Gamma	55	5.5
Norlimbanol Dextro @10%	6	0.6
Orange Oil Brazilian	25	2.5
Phenyl Ethyl Alcohol	30	3.0
Rose Oxide	3	0.3
Styrallyl Acetate	8	0.8
Tamarine Base 41.310G	3	0.3
Terpineol	20	2.0
Thesaron	7	0.7
Triplal Extra	3	0.3
Undecalactone, Gamma	20	2.0
Vanillin	5	0.5
Vertenex	85	8.5
Ylang Oil Extra	3	0.3
2-(3-methoxy-3-methylbutoxy)ethanal [Compound 23; TABLE 1]	80	8.0

[023] As used herein, olfactory effective quantity will be defined as the amount of said compound in a fragrance composition in which the individual component will contribute its characteristic olfactory properties, for example an olfactory property found to be more hedonistically appealing. A person of ordinary skill in the art may optimize the olfactory effect of the fragrance composition based on the incorporation of a fragrance compound of the present invention. The fragrance compounds may be used individually, or a part of mixture such that the sum of the effects of all fragrance ingredients present in the mixture yields a higher hedonistic rating. Therefore, the compounds embodied in the present invention can be employed to modify the characteristics of existing fragrance composition via their own olfactory properties or through additively effecting the contributions of other ingredient(s) present within the said composition. The quantity will vary widely depending on the other ingredients present, their relative amounts, the desired effect and the nature of the product.

Flavor Composition

[024] Compounds of formulas (**I-VI**) can be employed alone or incorporated into mixtures to enhance already existing flavor compositions. These compounds add a favorable organoleptic property and effect to the desired product. The compounds are typically present in an amount of from about 0.01% to about 20.0% by weight of the total flavor composition. Typically a more preferred embodiment would contain between 0.01% and 10% by weight and a most preferred embodiment would contain between 0.01% and 5% by weight. None of thes examples shown are meant to be limiting or restrictive on the use of the material as stated.

[025] As used herein, organoleptic effective quantity will be defined as the amount of said compound in a flavor composition in which the individual component will contribute its characteristic flavor properties. However, the organoleptic effect of the flavor composition will be the sum of the effects of all flavor ingredients present. Therefore, the compounds embodied in the present invention can be employed to modify the characteristics of the flavor composition via their own organoleptic properties or through additively effecting the contributions of other ingredient(s) present within the said composition. The quantity will vary widely depending on the presence of other ingredients present, their relative amounts, the desired effect and the nature of the product.

[026] The flavor carrier may be a liquid or a solid and typically do not significantly alter the olfactory or organoleptic properties of the flavor ingredients, respectively. Some non-limiting examples of flavor carriers include an emulsifying system, encapsulating materials, natural or modified starches, polymers, pectins, proteins, polysaccharides, gums and solvents which are typically employed in flavor applications.

[027] As used herein, the term "flavor carrier" may also encompass the food or beverage to which the fragrance compound (i.e. compounds encompassed by formulas I-VI) are added. Examples of such foods or beverages include, but are not limited to carbonated fruit beverages, carbonated cola drinks, wine coolers, cordials, flavored

water, powders for drinks (e.g., powdered sports or "hydrating" drinks), hard candy, soft candy, taffy, chocolates, sugarless candies, chewing gum, bubble gum, condiments, spices and seasonings, dry cereal, oatmeal, granola bars, soups, alcoholic beverages, energy beverages, juices, teas, coffees, salsa, gel beads, film strips for halitosis, gelatin candies, pectin candies, starch candies, lozenges, cough drops, throat lozenges, throat sprays, toothpastes and mouth rinses.

Intended Use

[028] Compounds of formulas (**I-VI**) can be employed alone or incorporated into mixtures to enhance already existing fragrance compositions, solvents, media and the like. The use of such compounds is applicable to a wide variety of products in the perfume industry for consumer use such as, but not limited to: sprays, candles, air fresheners, perfumes, colognes, gels, soft solids, solids, devices for introducing said compounds into a space (*e.g.*, a plug-in electrical device or a battery operated device), a liquid wicking system, personal care products such as soaps, talcum powder, antiperspirants, personal wash bar, personal wash liquid, personal wipe, deodorants, shampoos, conditioners, styling sprays, mousses, hair wipes, hair sprays, hair pomades, shower gels and shaving lotions; cosmetics such as oils, lotions and ointments; as well as detergents (*e.g.*, synthetic detergent), fabric care products (*e.g.*, fabric washing liquids and powders, fabric softeners, fabric conditioners), wipes, dishwashing liquids and powders, and household cleaning agents (*e.g.*, hard surface cleaning liquids and powders and aqueous and non-aqueous sprays). The sprays can be aqueous or non-aqueous. The candles and gels can be opaque, translucent, or transparent, and may contain optional ingredients to enhance their appearance. The plug-in and battery-operated devices can include devices that vaporize the fragrance by heat, evaporation, or nebulization.

[029] The use of such compounds is also applicable to a wide variety of products in the flavor industry such as, but not limited to: foodstuffs such as baked goods, dairy products, desserts, etc.; beverages such as juices, sodas, flavored waters, etc.; confectionaries such as sweets, hard candy, gums, gelatinous materials, etc. The flavor compositions can also be added to pharmaceutical applications, such as

lozenges, strips to deliver medicines or personal care products (e.g. fresh breath strips), cough syrup or other liquid or bucally administered medicines.

Synthesis Details

[030] Compounds of formula I may be isolated from the reaction of 3-methoxy-3-methyl-1-butoxide with an alkyl halide. Similarly, reaction of this alkoxide with an allyl halide results in a compound of formula (I) as well. Table 1 lists the olfactory properties of various novel compounds synthesized in accordance to formula (I). In particular, the unsaturated 3-methoxy-3-methyl-1-butanol ether derivatives described in the present invention all contain citrus and/or green notes incorporated into their odor compositions. Saturation of these olefinic substituents via hydrogenation results in a woody, fruitier olfactory character.

[031] Compounds encompassed by formula II can be synthesized via simple condensation reactions between the alcohol, 3-methoxy-3-methyl-1-butanol, and the respective carboxylic acid. Alternatively, compounds of formula (II) can be synthesized by direct reaction of the alcohol with the respective acid chloride. Table 1 lists the odor characteristics of various novel compounds synthesized in accordance to formula (II).

[032] The syntheses of all compounds related to formula (II) proceed with high yields. As represented in Table 1, the majority of the alkyl 3-methoxy-3-methyl-1-butanol esters have a strong fruity note associated with them.

[033] Compounds of formula III may be isolated from the reaction of 3-methoxy-3-methyl-1-butanol (Solfit™) with an alkyl chloroformate. The synthesis proceeds in a straight forward, facile manner. Table 1 lists the olfactory properties of various novel compounds synthesized in accordance to formula (III). The fragrance compounds encompassed by formula III generally maintain a light and soft quality.

[034] The syntheses of compounds (glycolates) encompassed by formula (IV) proceeded smoothly and in high yield. Simple esterification of various alkoxy

carboxylic acids with 3-methoxy-3-methyl-1-butanol in the presence of a catalytic amount of acid resulted in the desired products. Glycolates of this nature have a consistent light, powdery musk property. The musk qualities associated with these chemical entities have been reported in other glycolate-type compounds (See, e.g., U.S. Published Application No. 2006/0052277, which is hereby incorporated by reference).

[035] The present inventors have found that glycolates encompassed by formula (V) can be isolated in high yields when the reaction specifically employs 2-(3-methoxy-3-methylbutoxy)acetic acid with a respective alcohol to undergo esterification in similar fashion to the syntheses of compounds of formula (IV). Alternatively, reactions involving an alkyl halo-acetate with 3-methoxy-3-methyl-1-butoxide result in a low yield of the desired glycolate due to a mixture of undesired side products.

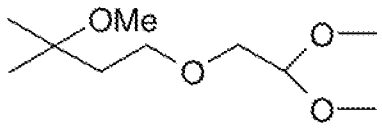
[036] The synthesis of the compound of formula (VI) in which n = 0 and R^6 = hydrogen proceeded smoothly via its dimethyl acetal precursor. Cleavage of the acetal group under acidic conditions afforded the respective acetaldehyde. This compound can also be synthesized via ozonolysis of allyl 3-methoxy-3-methyl-1-butanol ether. Such an approach was taken for the compound of formula (VI) in which n = 0 and R^6 = methyl. Both compounds possess odor characteristics which can be described as clean, melon-like and fresh with very strong diffusive properties.

[037] The synthesis of compounds of formula (VI) in which n = 1 and R^6 = hydrogen or a methyl group was based on a revised adaptation of previously reported syntheses between alcohols and α-β-unsaturated aldehydes (US 2694733; Feldman, D.P.; Stonkus, V.V.; Shimanskaya, M.V.; Avots, A.A. *Russ. J. Gen. Chem.* **1995**, *65*, 250-253). In the present case, acrolein and crotonaldehyde have been chosen as the α-β-unsaturated aldehydes. In each of the previously reported syntheses for reactions of this type, specific conditions involving the buffer capacity of the system were stressed. The syntheses reported here proceeded with extremely low yields (<5%) when employing such conditions. The present inventors found that the presence of a catalytic amount of acid greatly promotes the formation of the desired aldehydes. As

listed in Table 1, such aldehydes of this nature have a much more waxy, fatty-type aroma than the acetaldehyde analogues (n=0).

EXAMPLE 1

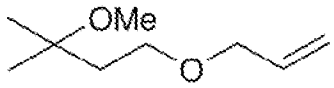
[038] This example illustrates the synthesis of 3-(2,2-dimethoxyethoxy)-1-methoxy-1,1-dimethylpropane.



[039] A suspension of sodium hydride (11.8 g, 0.47 mol) in anhydrous THF (400 mL) was warmed to approximately 40°C under an inert atmosphere. A portion of 3-methoxy-3-methyl-1-butanol (50.0 g, 0.42 mol) was then added dropwise via syringe over a period of 20 minutes during which time the temperature of the mixture was slowly raised to 70°C at 5 degree intervals. After one hour, bromoacetaldehyde dimethyl acetal (54.0 mL, 0.46 mol) was added dropwise via syringe over a period of 30 minutes and the mixture was stirred vigorously at 70°C for 16 hours. After this time, the mixture was cooled to room temperature, treated with H_2O (200 mL) and extracted with diethyl ether (3 x 200 mL). The organic phases were collected and washed with saturated $NaHCO_3$ (aq.) (2 x 200 mL) followed by H_2O (2 x 100 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting light yellow liquid was fractionally distilled (87°C, 3.00 torr) to yield the desired colorless, pure ether (57.0 g, 65.5 %). Odor: weak, marine-like, clean. GC/MS(EI): *m/z*(%) – 206(1), 191(1), 159(1), 143(3), 127(4), 111(5), 97(3), 89(2), 85(13), 75(100), 73(37), 69(8), 58(4), 55(3), 47(8), 45(15), 43(7). ^{1}H NMR ($CDCl_3$): δ 1.16 (s, 6H), 1.81 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.38 (s, 6H), 3.47 (d, *J* = 5.04 Hz, 2H), 3.55 (t, *J* = 7.33 Hz, 2H), 4.48 (t, *J* = 5.04 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 25.4, 39.3, 49.2, 53.9, 68.0, 70.8, 73.7, 102.9.

EXAMPLE 2

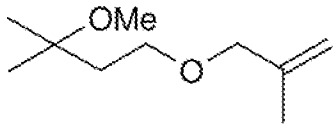
[040] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-prop-2-enyloxypropane.



[041] This compound was synthesized employing a procedure analogous to Example 1 using 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) and allyl chloride (16.4 mL, 0.20 mol). The isolated crude material was fractionally distilled (22°C, 0.80 torr) resulting in a colorless, pure liquid (22.5 g, 70.8 %). Odor: citrus, herbal, pine, sweet orange, candy. GC/MS(EI): *m/z*(%) – 158(1), 143(2), 126(1), 111(3), 97(1), 87(9), 85(7), 73(100), 71(14), 57(9), 55(9), 45(7), 43(13), 41(25). ^{1}H NMR ($CDCl_3$): δ 1.16 (s, 6H), 1.80 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.50 (t, *J* = 7.79 Hz, 2H), 3.96 (d, *J* = 5.96 Hz, 2H), 5.15 (dd, *J* = 11.9 Hz, 1H), 5.25 (dd, *J* = 18.8 Hz, 1H), 5.90 (m, 1H). ^{13}C NMR ($CDCl_3$): δ 25.4, 39.3, 49.2, 66.7, 71.9, 73.8, 116.8, 135.1.

EXAMPLE 3

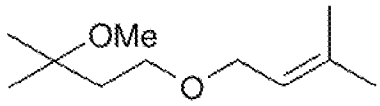
[042] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-(2-methylprop-2-enyloxy)propane.



[043] This compound was synthesized employing a procedure analogous to Example 1 using 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) and methallyl chloride (20.7 mL, 0.21 mol). The isolated crude material was fractionally distilled (103°C, 36.0 torr) resulting in a colorless, pure liquid (28.8 g, 79.3 %). Odor: citrus, green, lemon, slight orange. GC/MS(EI): *m/z*(%) – 172(1), 157(1), 140(1), 125(9), 111(2), 101(2), 95(7), 87(9), 85(10), 73(100), 71(11), 69(14), 55(36), 45(9), 43(12), 41(10). ^{1}H NMR ($CDCl_3$): δ 1.17 (s, 6H), 1.72 (s, 3H), 1.81 (t, *J* = 6.87 Hz, 2H), 3.18 (s, 3H), 3.47 (t, *J* = 7.33 Hz, 2H), 3.85 (s, 2H), 4.87 (s, 1H), 4.94 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 19.5, 25.5, 39.4, 49.2, 66.5, 73.8, 75.0, 111.8, 142.6.

EXAMPLE 4

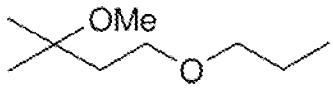
[044] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-(3-methylbut-2-enyloxy)propane.



[045] This compound was synthesized employing a procedure analogous to Example 1 using 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) and prenyl chloride (23.9 mL, 0.21 mol). The isolated crude material was fractionally distilled (66°C, 3.00 torr) resulting in a colorless, pure liquid (30.4 g, 77.0 %). Odor: green, chocolate, sweet, bitter. GC/MS(EI): *m/z*(%) – 186(1), 171(1), 154(1), 139(58), 103(4), 85(44), 78(4), 73(100), 69(86), 55(11), 45(12), 43(12), 41(26). ^{1}H NMR ($CDCl_3$): δ 1.15 (s, 6H), 1.66 (s, 3H), 1.73 (s, 3H), 1.79 (t, J = 7.33 Hz, 2H), 3.17 (s, 3H), 3.48 (t, J = 7.33 Hz, 2H), 3.93 (d, J = 6.87 Hz, 2H), 5.34 (t, J = 7.33 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 18.1, 25.5, 25.9, 39.2, 49.2, 66.5, 67.5, 73.8, 121.2, 136.8.

EXAMPLE 5

[046] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-propoxypropane.

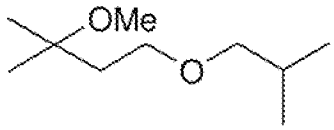


[047] A portion of the unsaturated ether, 1-methoxy-1,1-dimethyl-3-prop-2-enyloxypropane, (20.0 g, 0.13 mol) was dissolved in absolute EtOH (120 mL) and to this was added 5% (*w/w*) catalyst (5% Pd-C). The suspension was stirred at ambient temperature and treated with H_2 (250 psi) for 16 hours. Upon completion, the suspension was filtered through a glass frit (M) filter packed with filter paper and celite and rinsed with ethyl acetate. The solvent was removed under reduced pressure via rotary evaporation affording a light yellow liquid which was fractionally distilled

(58°C, 9.70 torr) to yield the desired colorless, pure ether (11.9 g, 59.1 %). Odor: woody, floral, slightly bitter banana. GC/MS(EI): *m/z*(%) – 160(1), 145(2), 128(8), 113(12), 87(8), 73(100), 71(20), 55(7), 43(24), 41(11). ^{1}H NMR ($CDCl_3$): δ 0.90 (t, *J* = 7.33 Hz, 3H), 1.16 (s, 6H), 1.58 (m, 2H), 1.78 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.35 (t, *J* = 6.42 Hz, 2H), 3.47 (t, *J* = 7.79 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 10.7, 23.0, 25.5, 39.2, 49.2, 67.0, 72.8, 73.9.

EXAMPLE 6

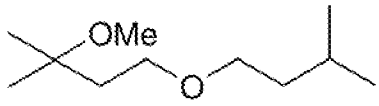
[048] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-(2-methylpropoxy)propane.



[049] This compound was synthesized employing a procedure analogous to Example 5 using 1-methoxy-1,1-dimethyl-3-(2-methylprop-2-enyloxy)propane (20.0 g, 0.12 mol). The isolated crude material was fractionally distilled (53°C, 5.00 torr) resulting in a colorless, pure liquid (16.0 g, 80.0 %). Odor: woody, green pear, slight chocolate. GC/MS(EI): *m/z*(%) – 174(1), 159(1), 142(5), 127(3), 99(2), 87(9), 85(5), 73(100), 71(17), 57(26), 43(11), 41(14). ^{1}H NMR ($CDCl_3$): δ 0.88 (d, *J* = 6.87 Hz, 6H), 1.16 (s, 6H), 1.78 (t, *J* = 7.33 Hz, 2H), 1.83 (m, 1H), 3.15 (d, *J* = 6.42 Hz, 2H), 3.18 (s, 3H), 3.47 (*J* = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 19.5, 25.5, 28.5, 39.2, 49.2, 67.2, 73.9, 78.1.

EXAMPLE 7

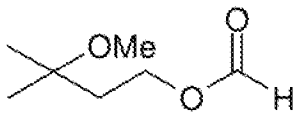
[050] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-(3-methylbutoxy)propane.



[051] This compound was synthesized employing a procedure analogous to Example 5 using 1-methoxy-1,1-dimethyl-3-(3-methylbut-2-enyloxy)propane (20.0 g, 0.11 mol). The isolated crude material was distilled via Kugelrohr apparatus (25°C, 0.15 torr) resulting in a colorless, pure liquid (9.93 g, 49.2 %). Odor: chocolate, liqueur, sour, slightly pungent. GC/MS(EI): *m/z*(%) – 188(1), 173(1), 156(4), 141(11), 99(7), 87(11), 73(100), 71(38), 55(11), 43(30), 41(14). ^{1}H NMR ($CDCl_3$): δ 0.89 (d, *J* = 6.87 Hz, 6H), 1.16 (s, 6H), 1.45 (q, *J* = 6.87 Hz, 2H), 1.67 (m, 1H), 1.78 (t, *J* = 7.33 Hz, 2H), 3.18 (s, 3H), 3.41 (t, *J* = 6.87 Hz, 2H), 3.47 (t, *J* = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 22.7, 25.2, 25.5, 38.7, 39.2, 49.2, 67.1, 69.5, 73.9.

EXAMPLE 8

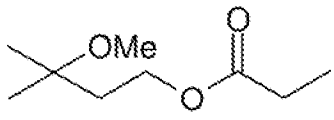
[052] This example illustrates the synthesis of 3-methoxy-3-methylbutyl formate.



[053] A portion of 3-methoxy-3-methyl-1-butanol (30.0 g, 0.25 mol) was dissolved in neat formic acid (60.9 g, 1.27 mol) and was stirred at ambient temperature for approximately 6 hours. Upon completion (monitored via GC), H_2O (100 mL) was added and the solution was extracted with diethyl ether (3 x 75 mL). The organic phases were collected and washed with saturated $NaHCO_3$ (aq.) (3 x 100 mL) followed by H_2O (2 x 50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting light yellow liquid was fractionally distilled (57°C, 9.00 torr) to yield the desired colorless, pure product (20.9 g, 56.3 %). Odor: woody, camphoraceous, dry, chemical. GC/MS(EI): *m/z*(%) – 146(1), 131(1), 85(43), 73(100), 69(14), 55(15), 43(12), 41(13). ^{1}H NMR ($CDCl_3$): δ 1.18 (s, 6H), 1.85 (t, *J* = 7.33 Hz, 2H), 3.18 (s, 3H), 4.27 (t, *J* = 7.33 Hz, 2H), 8.03 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 25.2, 38.3, 49.3, 60.7, 73.5, 161.3.

EXAMPLE 9

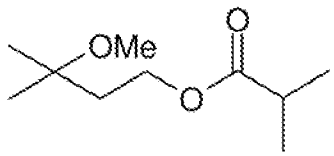
[054] This example illustrates the synthesis of 3-methoxy-3-methylbutyl propanoate.



[055] A portion of 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) was dissolved in anhydrous THF (200 mL) and purged with nitrogen. To this solution was added the propionyl chloride (20.2 mL, 0.23 mol) dropwise over a 15-minute period. The solution was stirred at ambient temperatures for 2 hours. Upon completion (monitored via GC), H_2O (100 mL) was added and the solution was extracted with diethyl ether (3 x 75 mL). The organic phases were collected and washed with saturated $NaHCO_3$ (aq.) (3 x 100 mL) followed by H_2O (2 x 50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting yellow liquid was fractionally distilled (110°C, 36.0 torr) to yield the desired colorless, pure product (29.4 g, 80.1 %). Odor: fruity, banana, floral, bubble gum. GC/MS(EI): *m/z*(%) – 173(1), 159(1), 143(1), 101(1), 85(57), 73(100), 69(27), 57(24), 55(16), 43(10), 41(10). ^{1}H NMR ($CDCl_3$): δ 1.12 (t, J = 7.33 Hz, 3H), 1.17 (s, 6H), 1.81 (t, J = 7.33 Hz, 2H), 2.30 (t, J = 7.33 Hz, 2H), 3.18 (s, 3H), 4.15 (t, J = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 9.2, 25.3, 27.7, 38.3, 49.3, 61.0, 73.6, 174.6.

EXAMPLE 10

[056] This example illustrates the synthesis of 3-methoxy-3-methylbutyl 2-methylpropanoate.

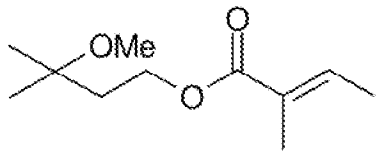


[057] This compound was synthesized employing a procedure analogous to Example 9 using 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) and isobutyryl chloride (24.4 mL, 0.23 mol). The isolated crude material was fractionally distilled (58°C, 3.00 torr) resulting in a colorless, pure liquid (29.6 g, 74.2 %). Odor: fruity, green, pear, apple, sour. GC/MS(EI): *m/z*(%) – 188(1), 173(1), 157(1), 115(1), 101(1), 85(55), 73(100), 71(12), 69(25), 55(13), 43(26), 41(15). ^{1}H NMR ($CDCl_3$): δ 1.14 (d,

J = 7.33 Hz, 6H), 1.18 (s, 6H), 1.81 (t, *J* = 7.33 Hz, 2H), 2.51 (m, 1H), 3.18 (s, 3H), 4.15 (t, *J* = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 19.0, 25.3, 34.1, 38.3, 49.3, 61.0, 73.6, 177.3.

EXAMPLE 11

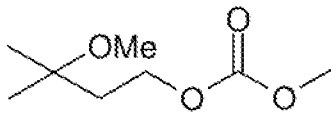
[058] This example illustrates the synthesis of 3-methoxy-3-methylbutyl (2*E*)-2-methylbut-2-enoate.



[059] Portions of tiglic acid (12.6 g, 0.13 mol) and p-toluene sulfonic acid (1.21 g, 6.35 mmol) were dissolved in 3-methoxy-3-methyl-1-butanol (45.0 g, 0.38 mol) and stirred vigorously while heated at 40°C for 27 hours. After this time, the solution was cooled to room temperature and diethyl ether was added (100 mL). The solution was washed with saturated $NaHCO_3$ (3 x 75 mL) followed by H_2O (100 mL). The aqueous fractions were back-extracted with diethyl ether (50 mL) and the organic layers were dried with $MgSO_4$. The solvent removed via rotary evaporation and the resulting clear liquid was fractionally distilled (76°C, 1.23 torr) to afford the desired colorless, pure ester (5.25 g, 20.8 %). Odor: sugary, sweet sap, green. GC/MS(EI): *m/z*(%) – 200(1), 185(1), 169(1), 127(1), 101(6), 85(50), 73(100), 69(16), 55(26), 43(7), 41(8). ^{1}H NMR ($CDCl_3$): δ 1.18 (s, 6H), 1.76 (d, *J* = 7.79 Hz, 3H), 1.81 (s, 3H), 1.85 (t, *J* = 7.33 Hz, 2H), 3.19 (s, 3H), 4.21 (t, *J* = 7.33 Hz, 2H), 6.83 (dq, *J* = 8.71 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 12.1, 14.4, 25.4, 38.3, 49.3, 61.1, 73.6, 128.8, 137.0, 168.3.

EXAMPLE 12

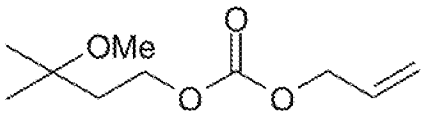
[060] This example illustrates the synthesis of 3-methoxy-3-methylbutyl methoxyformate.



[061] A portion of 3-methoxy-3-methyl-1-butanol (30.0 g, 0.25 mol) was dissolved in anhydrous THF (100 mL) and purged with nitrogen. To this solution was added methyl chloroformate (21.5 mL, 0.28 mol) dropwise. The solution was cooled to 0°C and stirred vigorously. To this solution was added pyridine (22.6 mL, 0.28 mol) *slowly*, which resulted in a very exothermic reaction and the immediate formation of a white precipitate. Upon completion (monitored via GC) (<1 hour), H_2O (100 mL) was added and the solution was extracted with diethyl ether (3 x 75 mL). The organic phases were collected and washed with 10% HCl (aq.) (3 x 75 mL) followed by brine solution (2 x 50 mL) and H_2O (2 x 50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting yellow liquid was fractionally distilled (84°C, 9.00 torr) to yield the desired colorless, pure product (35.7 g, 80.0 %). Odor: weak, light, green, floral. GC/MS(EI): *m/z*(%) – 176(1), 161(1), 101(2), 85(43), 73(100), 69(22), 59(5), 55(12), 45(6), 43(8), 41(8). ^{1}H NMR ($CDCl_3$); δ 1.17 (s, 6H), 1.86 (t, J = 7.33 Hz, 2H), 3.17 (s, 3H), 3.75 (s, 3H), 4.22 (t, J = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 25.3, 38.3, 49.3, 54.8, 64.8, 73.4, 155.9.

EXAMPLE 13

[062] This example illustrates the synthesis of 3-methoxy-3-methylbutyl prop-2-enyloxyformate.

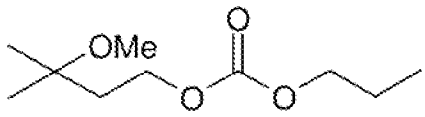


[063] This compound was synthesized employing a procedure analogous to Example 12 using 3-methoxy-3-methyl-1-butanol (25.0 g, 0.21 mol) and allyl chloroformate (25.5 mL, 0.23 mol). The isolated crude material was fractionally distilled (86°C, 3.00 torr) resulting in a colorless, pure liquid (37.3 g, 87.0 %). Odor: weak, soft chocolate, airy. GC/MS(EI): *m/z*(%) – 202(1), 187(1), 146(1), 108(1), 101(4), 85(45), 73(100), 69(39), 55(11), 43(8), 41(17). ^{1}H NMR ($CDCl_3$); δ 1.18 (s, 6H), 1.87 (t, J =

7.79 Hz, 2H), 3.18 (s, 3H), 4.23 (t, J = 7.33 Hz, 2H), 4.60 (d, J = 5.50 Hz, 2H), 5.25 (dd, J = 10.54 Hz, 1H), 5.34 (dd, J = 18.33 Hz, 1H), 5.91 (m, 1H). ^{13}C NMR ($CDCl_3$): δ 25.3, 38.3, 49.3, 64.8, 68.4, 73.4, 118.9, 131.8, 155.1.

EXAMPLE 14

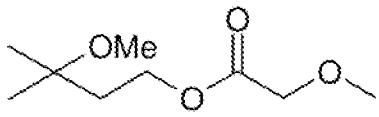
[064] This example illustrates the synthesis of 3-methoxy-3-methylbutyl propoxyformate.



[065] A portion of the unsaturated carbonate, 3-methoxy-3-methylbutyl prop-2-enyloxyformate, (20.0 g, 99.0 mmol) was dissolved in absolute EtOH (120 mL) and to this was added 1% (*w/w*) catalyst (5% Pd-C). The suspension was stirred at ambient temperature and treated with H_2 (250 psi) for 2 hours. Upon completion (monitored via GC), the suspension was filtered through a glass frit (M) filter packed with filter paper and celite and rinsed with ethyl acetate. The solvent was removed under reduced pressure via rotary evaporation affording a light yellow liquid which was fractionally distilled (86°C, 3.20 torr) to yield the desired colorless, pure product (18.5 g, 91.5 %). Odor: weak, soft musk, light vanilla. GC/MS(EI): *m/z*(%) – 204(1), 189(1), 101(2), 85(34), 73(100), 69(18), 55(9), 43(12), 41(12). ^{1}H NMR ($CDCl_3$): δ 0.95 (t, J = 7.79 Hz, 3H), 1.18 (s, 6H), 1.67 (m, 2H), 1.87 (t, J = 7.79 Hz, 2H), 3.18 (s, 3H), 4.08 (t, J = 6.87 Hz, 2H), 4.22 (t, J = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 10.3, 22.1, 25.3, 38.3, 49.3, 64.6, 69.6, 73.6, 155.6.

EXAMPLE 15

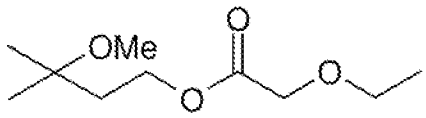
[066] This example illustrates the synthesis of 3-methoxy-3-methylbutyl 2-methoxyacetate.



[067] A portion of methoxyacetic acid (20 g, 0.22 mol) was dissolved in CH_2Cl_2 (160 mL) and dimethylformamide (40 mL). The solution was treated with oxalyl chloride (27.1 mL, 0.31 mol) in CH_2Cl_2 (100 mL) dropwise and stirred at room temperature for 2 hours. The temperature was then increased to 40°C and the solution was stirred for an additional 40 minutes. The solution was then cooled to room temperature, purged with nitrogen gas for 5 minutes and then cooled to 0°C. The cooled solution was treated with a mixture of 3-methoxy-3-methyl-1-butanol (26.2 g, 0.22 mol) and pyridine (19.8 mL, 0.22 mol) in CH_2Cl_2 (50 mL) dropwise and was stirred at that temperature for 2 hours. Upon completion, 10% HCl (aq.) was added and the organic layer was washed (3 x 100 mL) followed by H_2O (100 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting yellow liquid was fractionally distilled (71°C, 1.26 torr) to yield the desired colorless, pure product (25.8 g, 61.1 %). Odor: weak, fruity, slight coconut, powder musk. GC/MS(EI): *m/z*(%) – 190(1), 175(1), 128(1), 85(37), 73(100), 55(9), 45(24), 41(11). ^{1}H NMR ($CDCl_3$): δ 1.17 (s, 6H), 1.84 (t, J = 7.79 Hz, 2H), 3.17 (s, 3H), 3.43 (s, 3H), 4.00 (s, 2H), 4.25 (t, J = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 25.3, 38.3, 49.3, 59.4, 61.6, 70.0, 73.5, 170.4.

EXAMPLE 16

[068] This example illustrates the synthesis of 3-methoxy-3-methylbutyl 2-ethoxyacetate.

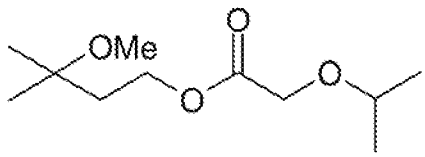


[069] Portions of ethoxyacetic acid (50 g, 0.47 mol) and p-toluenesulfonic acid (4.50 g, 23.5 mmol) were dissolved in 3-methoxy-3-methyl-1-butanol (167 g, 1.41 mol) and stirred vigorously while heated at 40°C for 5 hours. After this time, the solution was cooled to room temperature and diethyl ether was added (100 mL). The solution was

washed with saturated $NaHCO_3$ (3 x 75 mL) followed by H_2O (100 mL). The aqueous fractions were back-extracted with diethyl ether (50 mL) and the organic layers were dried with $MgSO_4$. The solvent removed via rotary evaporation and the resulting clear liquid was fractionally distilled (70°C, 1.04 torr) to afford the desired colorless, pure product (68.4 g, 71.2 %). Odor: powder musk, fresh, soft, light, clean. GC/MS(EI): *m/z*(%) – 204(1), 189(1), 128(4), 85(32), 73(100), 59(9), 55(7), 45(7). ^{1}H NMR ($CDCl_3$); δ 1.17 (s, 6H), 1.24 (t, *J* = 6.87 Hz, 3H), 1.84 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.57 (q, *J* = 7.33 Hz, 2H), 4.04 (s, 2H), 4.25 (t, *J* = 7.79 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 15.1, 25.3, 38.3, 49.3, 61.6, 67.3, 68.2, 73.5, 170.7.

EXAMPLE 17

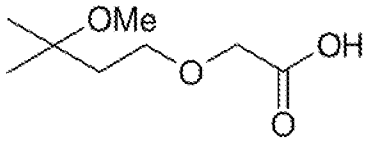
[070] This example illustrates the synthesis of 3-methoxy-3-methylbutyl 2-(methylethoxy)acetate.



[071] This compound was synthesized employing a procedure analogous to Example 16 using 3-methoxy-3-methyl-1-butanol (56.0 g, 0.48 mol) and methylethoxyacetic acid (14.0 g, 0.12 mol). The isolated crude material was fractionally distilled (72°C, 0.96 torr) resulting in a colorless, pure liquid (10.3 g, 39.9 %). Odor: very weak powder musk. GC/MS(EI): *m/z*(%) – 218(1), 203(1), 187(1), 160(1), 145(1), 128(8), 101(4), 85(40), 73(100), 69(52), 55(8), 45(10), 43(24), 41(12). ^{1}H NMR ($CDCl_3$); δ 1.16 (s, 6H), 1.19 (d, *J* = 5.96 Hz, 6H), 1.83 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.65 (m, 1H), 4.04 (s, 2H), 4.23 (t, *J* = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 21.8, 25.2, 38.5, 49.2, 61.5, 65.9, 72.6, 73.5, 170.9.

EXAMPLE 18

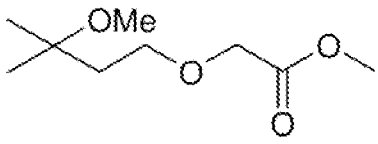
[072] This example illustrates the synthesis of 2-(3-methoxy-3-methylbutoxy)acetic acid.



[073] A suspension of sodium hydride (4.70 g, 0.19 mol) in anhydrous THF (100 mL) was warmed to approximately 40°C under an inert atmosphere. A portion of 3-methoxy-3-methyl-1-butanol (20.0 g, 0.17 mol) was then added dropwise via syringe over a period of 20 minutes during which time the temperature of the mixture was slowly raised to 70°C at 5 degree intervals. After one hour, sodium bromoacetate (28.2 g, 0.18 mol) was added in small portions over a period of 20 minutes and the mixture was stirred vigorously at 70°C for 4 hours. Upon completion (monitored via GC), the mixture was cooled to room temperature, treated with diethyl ether (100 mL) and washed with 10% HCl (aq.) (3 x 75 mL) followed by brine solution (100 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The crude light yellow liquid was not purified further for use in subsequent reactions. Yield: (29.2 g, 98.0 %). Odor: harsh, chemical. GC/MS(EI): *m/z*(%) – 176(1), 161(1), 129(2), 85(30), 73(100), 69(14), 55(9), 45(7). ^{1}H NMR ($CDCl_3$): δ 1.21 (s, 6H), 1.83 (t, J = 5.96 Hz, 2H), 3.21 (s, 3H), 3.66 (t, J = 5.96 Hz, 2H), 3.75 (s, 1H), 4.06 (s, 2H). ^{13}C NMR ($CDCl_3$): δ 25.2, 39.3, 49.4, 68.3, 68.6, 75.0, 173.1.

EXAMPLE 19

[074] This example illustrates the synthesis of methyl 2-(3-methoxy-3-methylbutoxy)acetate.

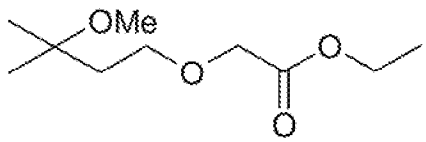


[075] A portion of 2-(3-methoxy-3-methylbutoxy)acetic acid (Example 18) (28.0 g, 0.16 mol) was diluted with methanol (50 mL) and treated with p-toluenesulfonic acid (1.51 g, 7.95 mmol) and stirred vigorously at 40°C for 3 hours. After this time, the solution was cooled to room temperature and diethyl ether was added (100 mL). The solution was washed with saturated $NaHCO_3$ (3 x 75 mL) followed by H_2O (100 mL).

The aqueous fractions were back-extracted with diethyl ether (50 mL) and the organic layers were dried with $MgSO_4$. The solvent removed via rotary evaporation and the resulting light yellow liquid was fractionally distilled (60°C, 1.00 torr) to afford the desired colorless, pure product (18.9 g, 62.6 %). Odor: metallic green, plastic. GC/MS(EI): *m/z*(%) – 190(1), 175(1), 143(3), 99(8), 85(67), 73(100), 69(27), 55(11), 45(25). ^{1}H NMR ($CDCl_3$): δ 1.16 (s, 6H), 1.84 (t, *J* = 7.33 Hz, 2H), 3.16 (s, 3H), 3.58 (t, *J* = 7.33 Hz, 2H), 3.73 (s, 3H), 4.06 (s, 2H). ^{13}C NMR ($CDCl_3$): δ 25.4, 39.1, 49.2, 51.9, 68.2, 68.4, 73.7, 171.0.

EXAMPLE 20

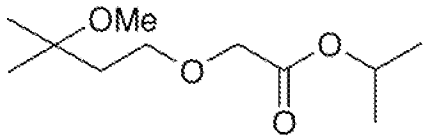
[076] This example illustrates the synthesis of ethyl 2-(3-methoxy-3-methylbutoxy)acetate.



[077] This compound was synthesized employing a procedure analogous to Example 19 using 2-(3-methoxy-3-methylbutoxy)acetic acid (14.0 g, 79.5 mmol) and ethanol (28 mL). The isolated crude material was fractionally distilled (65°C, 0.97 torr) resulting in a colorless, pure liquid (7.50 g, 46.9 %). Odor: vegetable green, spicy, plastic. GC/MS(EI): *m/z*(%) – 204(1), 189(1), 157(3), 99(10), 85(64), 73(100), 69(24), 55(9), 45(11). ^{1}H NMR ($CDCl_3$): δ 1.16 (s, 6H), 1.27 (t, *J* = 7.33 Hz, 3H), 1.84 (t, *J* = 7.33 Hz, 2H), 3.17 (s, 3H), 3.59 (t, *J* = 7.33 Hz, 2H), 4.04 (s, 2H), 4.20 (q, *J* = 7.33 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 14.3, 25.4, 39.1, 49.3, 60.9, 68.2, 68.6, 73.7, 170.6.

EXAMPLE 21

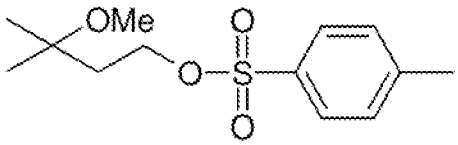
[078] This example illustrates the synthesis of methylethyl 2-(3-methoxy-3-methylbutoxy)acetate.



[079] This compound was synthesized employing a procedure analogous to Example 19 using 2-(3-methoxy-3-methylbutoxy)acetic acid (28.0 g, 0.16 mol) and 2-propanol (75 mL). The isolated crude material was fractionally distilled (70°C, 0.93 torr) resulting in a colorless, pure liquid (21.4 g, 61.8 %). Odor: green, licorice, slightly mint, fresh. GC/MS(EI): *m/z*(%) – 218(1), 203(1), 188(1), 171(1), 146(1), 129(5), 119(3), 99(12), 85(68), 73(100), 69(34), 55(9), 45(15), 43(21), 41(13). ^{1}H NMR ($CDCl_3$): δ 1.17 (s, 6H), 1.25 (d, J = 6.42 Hz, 6H), 1.85 (t, J = 7.33 Hz, 2H), 3.17 (s, 3H), 3.60 (t, J = 7.33 Hz, 2H), 4.01 (s, 2H), 5.08 (m, 1H). ^{13}C NMR ($CDCl_3$): δ 21.9, 25.4, 39.1, 49.3, 68.2, 68.5, 68.7, 73.7, 170.2.

EXAMPLE 22

[080] This example illustrates the synthesis of 3-methoxy-3-methylbutyl 4-methylbenzenesulfonate.

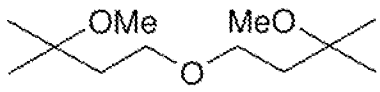


[081] A portion of p-toluenesulfonyl chloride (32.9 g, 0.17 mol) was dissolved in pyridine (50 mL) and cooled to 0°C. To this solution was added 3-methoxy-3-methyl-1-butanol (20.0 g, 0.17 mol) dropwise *slowly* via syringe. A white precipitate formed immediately and the mixture was stirred at 0°C for one hour followed by an additional 3 hours at room temperature. After this time, the mixture was treated with diethyl ether (100 mL) and washed with 10% HCl (aq.) (3 x 75 mL), saturated $NaHCO_3$ (aq.) (2 x 75 mL) and H_2O (2 x 50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting crude light yellow liquid was not purified further for use in subsequent reactions. Yield: (42.0 g, 91.1 %). Odor: odorless. GC/MS(EI): *m/z*(%) – 272(1), 257(1), 207(1), 173(1), 155(4), 91(21), 85(55), 73(100), 69(17), 65(10), 55(8), 45(5), 43(6). ^{1}H NMR ($CDCl_3$): δ 1.11 (s, 6H),

1.85 (t, J = 7.33 Hz, 2H), 2.43 (s, 3H), 3.08 (s, 3H), 4.11 (t, J = 7.33 Hz, 2H), 7.33 (d, J = 8.25 Hz, 2H), 7.77 (d, J = 8.25 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 21.7, 25.2, 38.8, 49.3, 67.4, 73.3, 128.0, 129.9, 133.2, 144.8.

EXAMPLE 23

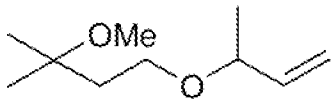
[082] This example illustrates the synthesis of 1-methoxy-3-(3-methoxy-3-methylbutoxy)-1,1-dimethylpropane.



[083] A suspension of sodium hydride (2.35 g, 93.1 mmol) in anhydrous THF (60 mL) was warmed to approximately 40°C under an inert atmosphere. A portion of 3-methoxy-3-methyl-1-butanol (10.0 g, 84.6 mmol) was then added dropwise via syringe over a period of 20 minutes during which time the temperature of the mixture was slowly raised to 70°C at 5 degree intervals. After one hour, the sulfonate product from Example 22 (23.0 g, 84.6 mmol) was added in small portions After this time, the mixture was cooled to room temperature, treated with diethyl ether (100 mL) and washed with saturated $NaHCO_3$ (aq.) (2 x 100 mL) followed by brine solution (2 x 50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting light yellow liquid was fractionally distilled (73°C, 0.95 torr) to yield the desired colorless, pure ether (13.2 g, 71.7 %). Odor: mold, mildew. GC/MS(EI): m/z(%) – 218(1), 171(1), 154(2), 139(21), 115(3), 99(10), 85(39), 73(100), 69(36), 55(9), 45(18), 43(14), 41(12). ^{1}H NMR ($CDCl_3$); δ 1.15 (s, 12H), 1.77 (t, J = 7.33 Hz, 4H), 3.17 (s, 6H), 3.47 (t, J = 7.33 Hz, 4H). ^{13}C NMR ($CDCl_3$): δ 25.5, 39.3, 49.2, 67.2, 73.8.

EXAMPLE 24

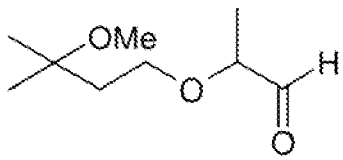
[084] This example illustrates the synthesis of 1-methoxy-1,1-dimethyl-3-(1-methylprop-2-enyloxy)propane.



[085] This compound was synthesized employing a procedure analogous to Example 23 using 3-methoxy-3-methylbutyl 4-methylbenzenesulfonate (37.9 g, 0.14 mol) and 3-butene-2-ol (12.4 mL, 0.14 mol). The isolated crude material was fractionally distilled (40°C, 2.45 torr) resulting in a colorless, pure liquid (14.0 g, 58.6 %). Odor: strong, mint, citrus, rose. GC/MS(EI): *m/z*(%) – 172(1), 157(1), 117(1), 95(3), 85(14), 73(100), 69(12), 55(33), 45(6), 41(8). ^{1}H NMR ($CDCl_3$): δ 1.15 (s, 6H), 1.22 (d, J = 5.96 Hz, 3H), 1.77 (m, 2H), 3.16 (s, 3H), 3.37 (dq, J = 9.16 Hz, 1H), 3.52 (dq, J = 8.71 Hz, 1H), 3.79 (t, J = 6.87 Hz, 1H), 5.10 (d, J = 12.37 Hz, 1H), 5.16 (d, J = 16.95 Hz, 1H), 5.72 (dt, J = 7.33 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 21.4, 25.5, 39.4, 49.2, 64.5, 73.9, 115.6, 140.6.

EXAMPLE 25

[086] This example illustrates the synthesis of 2-(3-methoxy-3-methylbutoxy)propanal.

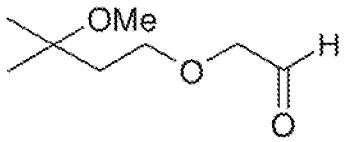


[087] A portion of 1-methoxy-1,1-dimethyl-3-(1-methylprop-2-enyloxy)propane (5.75 g, 33.3 mmol) was dissolved in CH_2Cl_2 (50 mL) and cooled to -78°C. The solution was purged with ozone for approximately one hour. Once the solution became blue, O_2 was bubbled through it for 30 minutes until the color disappeared and the reaction was quenched via addition of triphenylphosphine (10.5 g, 40.0 mmol). The solvent was removed via rotary evaporation and the resulting residue was suspend in a hexane:diethyl ether (1:1) mixture overnight in the refrigerator. The mixture was filtered and the precipitate was rinsed with hexane. The filtrate was placed on a rotary evaporator and the solvent was removed under reduced pressure. The isolated crude material was distilled via Kugelrohr apparatus (64°C, 0.40 torr)

resulting in a colorless liquid (4.57 g, 78.8 %). Odor: fresh, watery melon, clean, floral, muguet. GC/MS(EI): *m/z*(%) – 174(1), 159(1), 145(1), 127(3), 113(36), 101(5), 85(24), 73(100), 69(75), 59(41), 57(14), 55(11), 45(26), 43(19), 41(22). ^{1}H NMR ($CDCl_3$): δ 1.17 (s, 6H), 1.27 (d, J = 6.87 Hz, 3H), 1.84 (dt, J = 6.42 Hz, 2H), 3.18 (s, 3H), 3.61 (m, 2H), 3.76 (dq, J = 8.71 Hz, 1H), 9.64 (d, J = 1.83 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 15.3, 25.4, 39.6, 49.3, 66.5, 73.7, 80.5, 204.0.

EXAMPLE 26

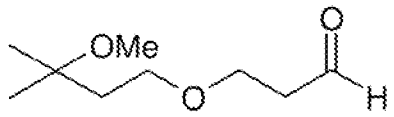
[088] This example illustrates the synthesis of 2-(3-methoxy-3-methylbutoxy)ethanal.



[089] A portion of the dimethyl acetal, 3-(2,2-dimethoxyethoxy)-1-methoxy-1,1-dimethylpropane (Example 1), (27.6 g, 0.13 mol) was dissolved in a large excess of neat formic acid (74.0 g, 1.61 mol) and stirred vigorously for 4 hours. Upon completion (monitored via GC), the solution was treated with H_2O (150 mL) and extracted with ethyl acetate (3 x 100 mL). The organic phases were collected and dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting light yellow liquid was *carefully* distilled via Kugelrohr apparatus first to remove any leftover formic acid (25°C, 1.00 torr) followed by isolation of the desired aldehyde (40°C, 0.10 torr) resulting in a colorless, pure liquid (13.0 g, 60.2 %). Odor: fresh, melon, clean, floral, muguet, green. GC/MS(EI): *m/z*(%) – 161(1), 145(2), 113(1), 99(6), 85(29), 73(100), 69(27), 55(9), 45(23), 43(14), 41(11). ^{1}H NMR ($CDCl_3$): δ 1.17 (s, 6H), 1.85 (t, J = 7.33 Hz, 2H), 3.17 (s, 3H), 3.61 (t, J = 7.33 Hz, 2H), 4.06 (s, 2H), 9.71 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 25.4, 39.3, 49.3, 68.4, 73.7, 76.5, 201.0.

EXAMPLE 27

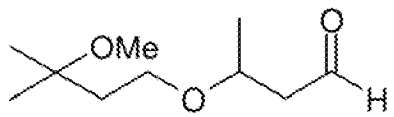
[090] This example illustrates the synthesis of 3-(3-methoxy-3-methylbutoxy)propanal.



[091] A portion of 3-methoxy-3-methyl-1-butanol (10.0 g, 84.6 mmol) was treated with acrolein (25.1 mL, 0.34 mol) and a small aliquot of concentrated HCl (7 drops). The solution was stirred vigorously at 40°C in subdued light for 3 days. After this time, the solution was cooled to room temperature and ethyl acetate (75 mL) was added. The solution was washed with saturated $NaHCO_3$ (aq.) (100 mL) followed by H_2O (50 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting light yellow liquid was distilled (58°C, 0.92 torr) to yield the desired colorless aldehyde (8.21 g, 55.9 %). Odor: waxy, oily, muguet, light floral. GC/MS(EI): *m/z*(%) – 174(1), 159(1), 127(5), 85(19), 73(100), 69(7), 57(9), 55(8), 45(12), 43(11). ^{1}H NMR ($CDCl_3$): δ 1.14 (s, 6H), 1.76 (t, J = 7.33 Hz, 2H), 2.64 (dt, J = 4.12 Hz, 2H), 3.16 (s, 3H), 3.51 (t, J = 7.33 Hz, 2H), 3.74 (t, J = 5.96 Hz, 2H), 9.77 (t, J = 2.29 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 25.4, 39.2, 44.0, 49.2, 64.6, 67.6, 73.8, 201.4.

EXAMPLE 28

[092] This example illustrates the synthesis of 3-(3-methoxy-3-methylbutoxy)butanal.

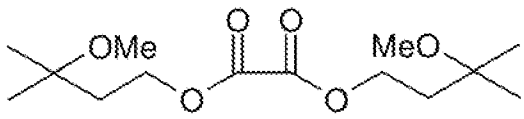


[093] This compound was synthesized employing a procedure analogous to Example 27 using 3-methoxy-3-methyl-1-butanol (10.0 g, 84.6 mmol) and crotonaldehyde (42.9 mL, 0.51 mol). The isolated crude material was fractionally distilled (48°C, 0.35 torr) resulting in a colorless liquid (3.24 g, 20.4 %). Odor: waxy, oily, fatty. GC/MS(EI): *m/z*(%) – 188(1), 173(1), 141(3), 101(5), 85(18), 73(100), 69(11), 55(7),

43(20), 41(15). ^{1}H NMR ($CDCl_3$): δ 1.14 (s, 6H), 1.23 (d, J = 6.42 Hz, 3H), 1.74 (t, J = 6.87 Hz, 2H), 2.45 (qd, J = 22.91 Hz, 1H), 2.60 (qd, J = 26.12 Hz, 1H), 3.17 (s, 3H), 3.43 (m, 1H), 3.60 (m, 1H), 3.93 (m, 1H), 9.77 (t, J = 2.29 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 19.9, 25.4, 39.6, 49.2, 50.6, 64.9, 71.0, 73.8, 201.7.

EXAMPLE 29

[094] This example illustrates the synthesis of bis(3-methoxy-3-methylbutyl ethane)-1,2-dioate.



[095] A portion of 3-methoxy-3-methyl-1-butanol (5.00 g, 42.3 mmol) was dissolved in CH_2Cl_2 (25 mL) and cooled to 0°C. An aliquot of oxalyl chloride (2.21 mL, 25.4 mmol) was *slowly* added dropwise via syringe to the vigorously stirring solution. After one hour, the solution was warmed to room temperature and washed with saturated $NaHCO_3$ (aq.) (2 x 50 mL) followed by brine solution (30 mL) and H_2O (30 mL). The organic phase was dried with $MgSO_4$ and the solvent was removed under reduced pressure. The resulting clear liquid was distilled via kugelrohr apparatus (160°C, 0.15 torr) to yield the desired colorless, viscous oxalate (4.50 g, 73.3 %). Odor: weak, chemical. GC/MS(EI): m/z(%) – 290(1), 275(1), 257(1), 227(1), 191(1), 159(1), 101(2), 85(37), 73(100), 69(21), 55(6), 43(7), 41(9). ^{1}H NMR ($CDCl_3$): δ 1.19 (s, 12H), 1.91 (t, J = 7.33 Hz, 4H), 3.18 (s, 6H), 4.37 (t, J = 7.79 Hz, 4H). ^{13}C NMR ($CDCl_3$): δ 25.3, 37.9, 49.4, 63.8, 73.5, 158.0.

[096] The below examples demonstrate the use of the fragrance formulation of Table 2 in various consumer products. These examples are illustrative only and are not intended to limit the scope of the invention in any way. Unless otherwise noted, all percentages set forth in the examples are by weight (wt). Q.S. means a sufficient quantity.

EXAMPLE 30: ALL PURPOSE CLEANER – CONCENTRATED

Ingredient	Supplier	Percentage	Purpose
Neodol® 91-8	Shell	6.00	Surfactant
Dowanol® DPnB	Dow	6.00	Solvent
Kathon® CG	Rohm & Haas	0.10	Preservative
Fragrance of Table 2	Takasago Int'l	1.25	Fragrance
D. I. Water	----------	86.65	Solvent
Dye	Pylam	Q.S.	Color

[097] This formulation is for dilution in a bucket at approximately one part concentrate to ten parts tap water.

[098] Procedure:

[099] 1. The Neodol® 91-8, Dowanol® DPnB, and fragrance were mixed in a suitable vessel until completely uniform and clear.

[0100] 2. Water was added slowly with constant agitation to the solution in # 1. Kathon® was added with mixing and stirring continued for 1 minute. The final formulation was clear.

[0101] 3. Dye was added match standard.

EXAMPLE 31: HARD SURFACE CLEANER – PUMP SPRAY

Ingredient	Supplier	Percentage	Purpose
Neodol® 91-8	Shell	1.50	Surfactant
Dowanol® DPnB	Dow	4.00	Solvent
Fragrance of Table 2	Takasago Int'l	0.40	Fragrance
Kathon® CG	Rohm & Haas	0.07	Preservative
D. I. Water	---------	94.03	Solvent

[0102] Procedure:

[0103] 1. Neodol® 91-8, Dowanol® DPnB, and fragrance were mixed in a suitlable vessel until completely uniform and clear.

[0104] 2. Water was added slowly to the solution in # 1 with constant agitation. The final formulation was clear. Kathon® CG was added with stirring.

[0105] The formulation was then filled into suitable plastic containers (PET preferred), with the proper trigger or pump closure.

EXAMPLE 32: LIQUID LAUNDRY DETERGENT

Ingredient	INCI or Nomenclature	Percentage
Water	Water	Q.S.
Glucopon® 625UP (1)	Alkyl Polyglucosides	12.50
Standapol® ES-40 (1)	Alkyl Ether Sulfates	25.60
Versene® 100 (38%) (2)	Tetrasodium EDTA	00.40
MEA (2)	Monoethanolamine	01.00
Sulfuric Acid (25% Aq.)	Acid	03.00
Sodium Chloride (25%)	Salt	01.20
Takasago Fragrance(3)	Fragrance of Table 2	00.75
		100.00

[0106] Suppliers

(1) Cognis Corporation
(2) The DOW Chemical Co.
(3) Takasago International Corporation

[0107] **Procedure:**

[0108] 1. Glucopon® was added to water heated to 65°C and mix at medium speed until clear.

[0109] 2. Standapol® was added and mixing continued until the mixture was clear and homogenous.

[0110] 3. The mixture was removed from the heat and remaining ingredients were added in order, with mixing at slow to medium speed each addition.

[0111] 4. pH was adjusted with sulfuric acid solution to pH of 8.0 to 8.5.

[0112] 5. Viscosity was adjusted with sodium chloride.

EXAMPLE 33: LIQUID FABRIC SOFTENER

Ingredient	Supplier	Percentage	Purpose
Deionized Water	------	83.50	Solvent
Rewoquat® WE-16E	Degussa	15.00	Softener
Sodium Chloride	------	0.50	Thickening
Fragrance of Table 2	Takasago	1.00	Fragrance

[0113] Procedure:

[0114] 1. Rewoquat®, fragrance and water were mixed in a suitable vessel until the mixture was translucent to opaque.

[0115] 2. Sodium chloride was added with mixing and mixed for 5 minutes.

EXAMPLE 34: HAND & BODY LOTION

SEQ.	INGREDIENTS		INCI or Nomenclature	PERCEN
Part 1				
1	Deionized water		Water	QS
1	Versene® 220	(1)	Tetrasodium EDTA	00.05
2	Carbopol® 934	(2)	Carbomer 934	00.30
3	Glycerin		Glycerin	01.00
3	Propylene Glycol		Propylene Glycol	01.00
Part 2				
4	Myrj® 52S (sprayed)	(4)	PEG-40 Stearate	01.80
4	Liponate® GC	(5)	Caprylic/Capric Triglyceride	13.00
4	Liponate® IPM	(5)	Isopropyl Myristate	08.50
4	Span® 65S (sprayed)	(4)	Sorbitan Tristearate	02.00
4	Pharmalan®, USP	(6)	Lanolin	00.50
4	White Protopet® 1S	(7)	Petrolatum	00.30
4	Propylparaben	(8)	Propylparaben	00.10
Part 3				
1	Deionized Water		Water	10.00
1	TEA		Triethanolamine	00.30
Part 4				
1	Deionized Water		Water	01.00
1	Unicide® U-13	(5)	Imidazolidinyl Urea	00.25
Part 5				
1	Germaben® II	(3)	Propylene Glycol (and) Diazo-lidinyl Urea (and) Methylparaben (and) Propylparaben	00.70
Part 6				
1	Takasago Fragrance Oil		Fragrance of Table 2	QS

[0116] Suppliers:

(1) Dow Chemical
(2) Noveon
(3) Sutton/ISP
(4) Uniqema
(5) Lipo Chemicals, Inc.
(6) Croda
(7) Crompton/Witco
(8) Tri-K

[0117] Procedure:

[0118] Part 1

[0119] 1. Seq. #1 was heated to 75°C and mixed together at medium speed using an overhead mixer until clear.

[0120] 2. Seq. #2 was added slowly to Seq. #1 with mixing. Mixing was continued until Seq. #2 is completely was hydrated. Hydration was checked by dipping a metal spatula into and out of the solution to observe if there are any gum particles that have not hydrated.

[0121] 3. Seq. #3 was added in order to the batch without heating.

[0122] Part 2

[0123] 4. Seq. #4 was premixed and heated until completely melted at approximately 65°C.

[0124] 5. **Part 1** was placed on a Homomixer at low to medium speed, and **Part 2** added to **Part 1** and mixed for 1 minute.

[0125] 6. The batch was placed back onto the overhead mixer at medium speed and premixed **Part 3** was added without heating to the batch for approximately 2 minutes.

[0126] 7. Mix was continued at low speed and the mixture was cooled to 35°C.

[0127] 8. **Part 4** was premixed at 35°C and added to the batch, while cooling down at low speed to 30°C.

[0128] 9. **Part 5** was added at 30°C with mixing at low speed.

[0129] Liquid fragrance was added slowly while mixing.

[0130] 10. Lotion was placed in jars and allowed to at room temperature for 24 hours.

EXAMPLE 35: CLEAR LIQUID HAND SOAP

SEQ.	INGREDIENTS		INCI	PERCENT
1	Deionized water		Water	66.50
1	Methyl Paraben	(1)	Methyl Paraben	00.25
2	Liponic® EG-1	(2)	Glycereth-26	01.00
2	Glycerin	(3)	Glycerin	01.00
2	Lipopeg® 6000DS	(2)	PEG-150 Distearate	00.50
3	Monamid® 716	(4)	Lauramide DEA	03.50
3	Standapol® ES-2	(3)	Sodium Laureth Sulfate	25.00
3	Velvetex® BK-35	(3)	Cocamidopropyl Betaine	15.00
4	Deionized Water		Water	01.00
4	Unicide® U-13	(2)	Imidazolidinyl Urea	00.25
5	Fragrance as defined in Table 2 above			00.50
6	Citric acid (25% Solution)			QS

[0131] Suppliers:

(1) TRI-K
(2) Lipo Chemicals, Inc.
(3) Cognis/Henkel
(4) Uniqema

[0132] Procedure:

[0133] 1. The methyl paraben was added slowly to the DI water heated to 65°C with mixing at medium/high speed using an overhead mixer until completely into solution and clear. (Seq.#1)

[0134] 2. Seq. #2 was added to Sequence #1 at low speed until completely clear.

[0135] 3. Seq. #3 was added to batch without heating, in order of addition, and cooled down to 35°C with low agitation.

[0136] 4. Seq. #4 was premixed until clear, and added to batch.

[0137] 5. Seq. #5 was added to the batch with low agitation and cooled down to 25°C.

[0138] 6. Seq. #6 was added to adjust batch to desired pH. The product was placed in jars, pouring very slowly onto the sides of the jars to eliminate any additional aeration. pH=adjust to: 6.64 +/-0.2; viscosity = 18,640 cps (+/- 10%) with Brookfield LV Sp. #4 @ 30 rpm

EXAMPLE 36: CLEAR SHAMPOO

SEQ.	INGREDIENTS		INCI	PERCENT
1	Deionized water		Water	Q.S.
2	Versene® 220	(1)	Teterasodium EDTA	00.05
3	Methocel® E4M (*prep)	(2)	Hydroxypropyl Methylcellulose	00.30
4	Standapol® T	(3)	TEA Lauryl Sulfate	18.00
4	Standapol®A	(3)	Ammonium Lauryl Sulfate	08.00
4	Monamid® 150-LMWC	(4)	Lauramide DEA	04.00
4	Palmitic Acid	(5)	Palmitic Acid	00.30
5	Glydant®2000	(6)	DMDM Hydantoin	00.15
5	Sodium Chloride, Granular	(7)	Sodium hloride	00.34
5	Citric Acid	(8)	Citric Acid	00.43
6	Takasago perfume oil	(9)	Fragrance of Table 2	Q.S.

[0139] Suppliers:

(1) AND
(2) DOW CHEMICAL CO.
(3) COGNIS/HENKEL
(4) UNIQEMA
(5) Takasago International Corp (TIC), USA
(6) LONZA
(7) FISHER SCIENTIFIC
(8) TIC, USA
(9) TIC, USA

[0140] Procedure:1. 1/3 of Seq.#1and Seq.#2 was heated to 85°C. Methocel powder was by mixing thoroughly using 1/5 to 1/3 of the required total amount of water as hot water (80-90°C). Mixing was continued with overhead mixer at medium speed until all of the particles were wetted down, and a consistent dispersion was obtained. The remainder of the water containing Seq. #2 was added as cold water while mixing. The solution was cooled down to less than 30°C. Mixing was continued after the proper temperature was achieved for approximately 20 minutes. After preparation was completed, the Methocel® solution was reheated to 60°C.

[0141] 2. Seq.#4 at 60 to 65°C was added slowly to the batch in order of addition with mixing continued at low speed.

[0142] 3. Seq.#5 was added slowly to batch and mixing continued at low speed until room temperature was reached. Citric acid was added to a pH of 5.5-6.0 and sodium chloride was added to achieve the desired viscosity. Fragrance Seq.#6 was weighed and added to the formulation while mixing.

4. Viscosity: = 2020cps. (±10%) taken at 20°C, (Brookfield LV Sp.#3 @ 12rpm), pH=5.5 (+/-0.5)

The below examples demonstrate the use of the materials claimed in flavor formulations in various consumer products. These examples are illustrative only and are not intended to limit the scope of the invention in any way. In these examples, all % are % (wt), unless otherwise noted and Q.S. means a sufficient quantity.

EXAMPLE 37: TOOTH PASTE

Toothpaste with flavor and the claimed compound(s) was prepared according to the formulation below.

Components	WT%
2-(3-methoxy-3-methylbutoxy)ethanal [Compound 23; TABLE 1]	0.50
Calcium hydrogen phosphate (dihydrate)	50.00
Glycerin	25.00

Sodium lauryl sulfate	1.40
Carboxymethyl cellulose sodium salt	1.50
Saccharin sodium salt	0.20
Sodium benzoate	0.10
Strawberry type flavor	0.70
Purified water balance	qs
	Total 100.00

EXAMPLE 38: PEACH FLAVOR

Peach flavor utilizing the claimed compound(s) was prepared according to the formulation below utilizing the claimed compounds.

Components	WT%
2-(3-methoxy-3-methylbutoxy)ethanal[Compound 23; TABLE 1]	0.20
Benzaldehyde	0.20
methyl cyclohexyl)ethanone	0.30
Ethyl acetate	2.0
Ethyl butyrate	0.8
Ethyl maltol	0.3
γ-Undecalactone	0.5
Linalool	0.5
Peach flavor base	5.0
Ethyl alcohol balance	q.s.
	Total 100.00

EXAMPLE 39: GREEN TEA FLAVOR

Green tea flavor was prepared according to the formulation below utilizing the claimed compound(s)

Components	WT%
Benzyl alcohol	1.0
2-(3-methoxy-3-methylbutoxy)ethanal [Compound 23; TABLE 1]	0.3
Cis-3-hexenol	0.3
Dimethyl sulfide	0.1
Geraniol	0.6
l-menthol	2.5
Linalool	0.9
Nerolidol	0.2
Terpineol	0.2
Green tea base	5.0
Ethyl alcohol balance	qs
	Total 100.00

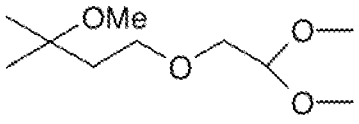
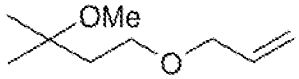
EXAMPLE 40: BLACK TEA FLAVOR

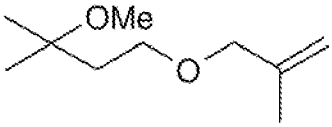
Black tea flavor was prepared according to the formula below utilizing the claimed compounds.

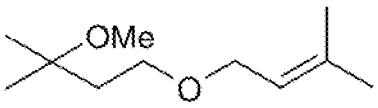
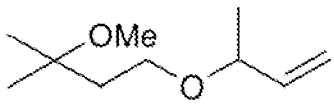
Components	WT%
α-Ionone	0.2
α-Ionone	0.2
Benzaldehyde	1.0
2-(3-methoxy-3-methylbutoxy)ethanal [Compound 23; TABLE 1]	1.0
Cis-3-hexenol	6.0
δ-Decalactone	2.5
δ-Dodecalactone	2.0
Damascenone	0.1
Linalool	3.5
Geraniol	6.0
Citral	1.0
Linalool oxide	1.6
Methyl salicylate	2.0
Phenylethyl alcohol	6.0
Hexyl aldehyde	1.0
Propylene glycol balance	qs
	Total 100.00

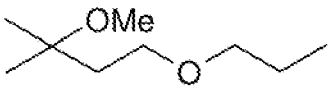
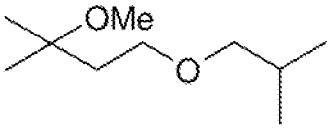
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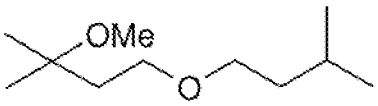
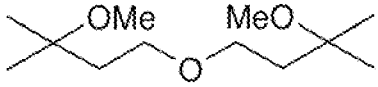
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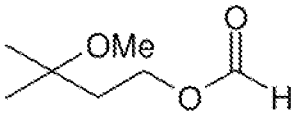
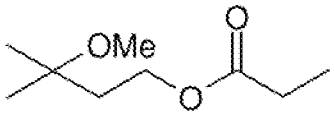
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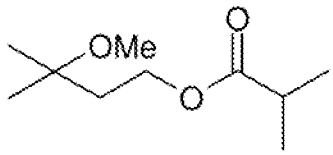
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, Compound 5: , Compound

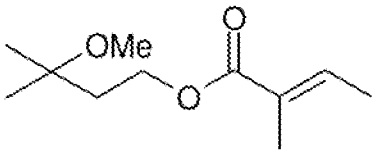
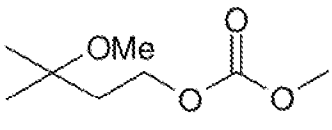
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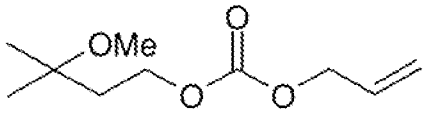
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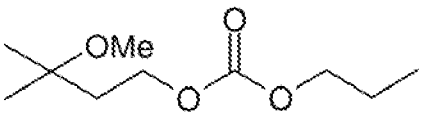
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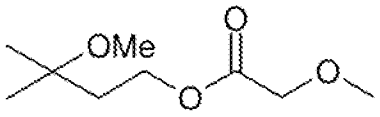
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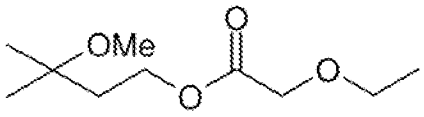
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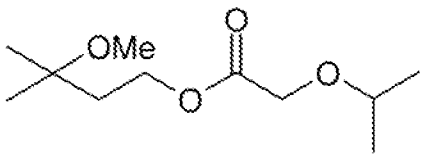
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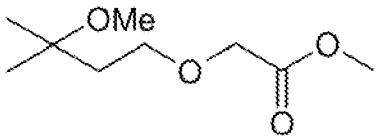


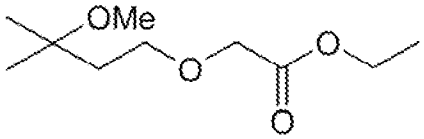
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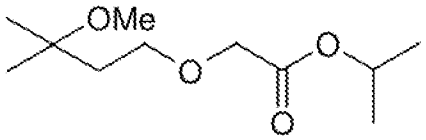
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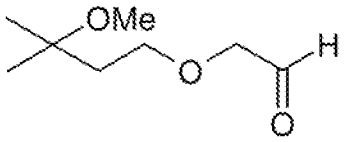
Compound 18: ,

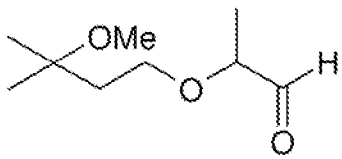
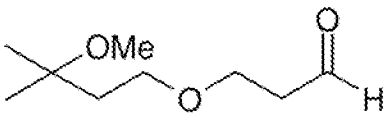
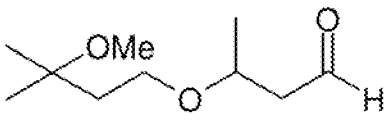
Compound 19: ,

Compound 20: ,

Compound 21: ,

Compound 22: ,

Compound 23: ,

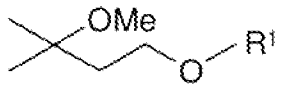
Compound 24: , Compound 25: , and Compound 26: .

2. A compound selected from the group consisting of 3-methoxy-3-methylbutyl 2-ethoxyacetate, 2-(3-methoxy-3-methylbutoxy)ethanal, 1-methoxy-1,1-dimethyl-3-(3-methylbut-2-enyloxy)propane, 1-methoxy-1,1-dimethyl-3-prop-2-enyloxypropane, and 1-methoxy-1,1-dimethyl-3-(1-methylprop-2-enyloxy)propane.

3. A fragrance composition comprising:

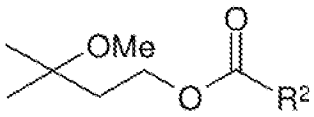
(i) a compound represented by

(a) the formula (I):



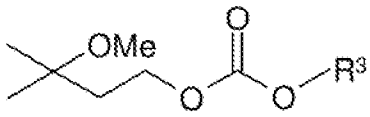
(I),

(b) the formula (II):



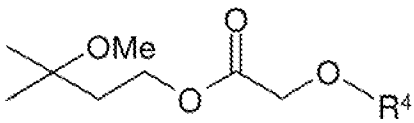
(II),

(c) the formula (III):



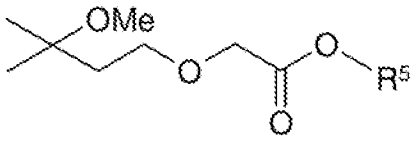
(III),

(d) the formula (IV):



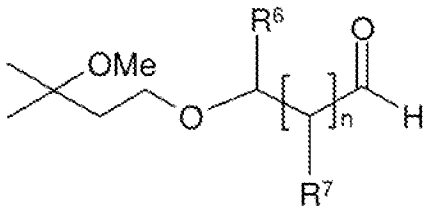
(IV),

(e) the formula (V):



(V), or

(f) the formula (VI):



(VI),

wherein R^1, R^2, R^3, R^4, or R^5 is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, an unsubstituted or substituted phenyl, an unsubstituted or substituted benzyl, an unsubstituted or substituted naphthyl, an unsubstituted or substituted aryl, an unsubstituted or substituted piperazino, an unsubstituted or substituted morpholinyl, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{6-12} arylamino, an unsubstituted or substituted C_{1-6} dialkylamino, an unsubstituted or substituted C_{6-12} diarylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted C_{1-6} alkylcarbonyl, thio, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido, an unsubstituted or substituted C_{1-6} dialkylcarboxamido, an unsubstituted or substituted phenoxy, an unsubstituted or substituted benzyloxy, phenylcarbonyl, benzylcarbonyl, an unsubstituted or substituted nitrophenyl, C_{1-6} trialkylsilyl or nitro, an unsubstituted or substituted C_{1-12} straight chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain aryl sulfonate or an unsaturated sulfonate; or

R^2, R^3, R^4, or R^5 is hydrogen; or

R^1, R^2, R^4, or R^5 is an unsubstituted or substituted C_{3-6} cycloalkyl; or

R^6 and R^7 are, independently, a hydrogen or an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl group and n = 0-1; and

(ii) a fragrance carrier or fragrance base.

4. The composition of claim 3, wherein R^1, R^2, R^3, R^4, or R^5 is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{1-6} dialkylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted C_{1-6} alkylcarbonyl, an unsubstituted or substituted C_{1-6} alkylcarboxy, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido or an unsubstituted or substituted C_{1-6} dialkylcarboxamido or

R^1, R^2, R^4, or R^5 is an unsubstituted or substituted C_{3-6} cycloalkyl.

5. The composition of claim 3, wherein R^1, R^2, R^3, R^4, or R^5 is a C_{1-6} straight chain alkyl, C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{1-6} alkenyl, an unsubstituted or substituted C_{1-6} acyl, or aryl.

6. The composition of claim 3, wherein the compound is present in an amount from about 0.001% to about 30% by weight.

7. The composition of claim 3, wherein the fragrance carrier is an emulsifying agent, an encapsulating material, natural or modified starches, polymers, gums, pectins, gelatinous or porous cellular materials, or waxes.

8. A method of improving, enhancing, or modifying the olfactory properties of a product comprising adding an olfactory effective amount of a composition of claim 3 to the product.

9. The method of claim 8, wherein the olfactory effective amount is from about 0.001% to about 30% by weight.

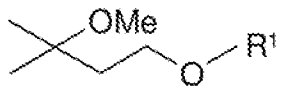
10. A consumer product comprising the composition of claim 3.

11. The consumer product of claim 10, where the consumer product is an air care product, candle, perfume, cologne, personal care product, cosmetic, detergent, fabric care product, or household cleaning agent.

12. A flavor composition comprising:

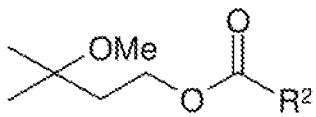
(i) a compound represented by

(a) formula (I):



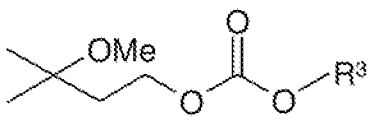
(I),

(b) formula (II):



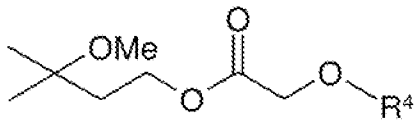
(II),

(c) formula (III):



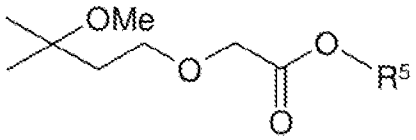
(III),

(d) formula (IV):



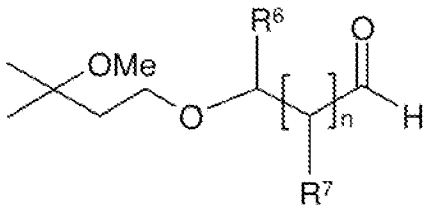
(IV),

(e) formula (V):



(V), or

(f) formula (VI):



(VI),

wherein R^1, R^2, R^3, R^4, or R^5 is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, an unsubstituted or substituted phenyl, an unsubstituted or substituted benzyl, an unsubstituted or substituted naphthyl, an unsubstituted or substituted aryl, an unsubstituted or substituted piperazino, an unsubstituted or substituted morpholinyl, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{6-12} arylamino, an unsubstituted or substituted C_{1-6} dialkylamino, an unsubstituted or substituted C_{6-12} diarylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted C_{1-6} alkylcarbonyl, thio, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido, an unsubstituted or substituted C_{1-6} dialkylcarboxamido, an unsubstituted or substituted phenoxy, an unsubstituted or substituted benzyloxy, phenylcarbonyl, benzylcarbonyl, an unsubstituted or substituted nitrophenyl, C_{1-6}

trialkylsilyl or nitro, an unsubstituted or substituted C_{1-12} straight chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} branched chain alkenyl sulfonate, an unsubstituted or substituted C_{1-12} straight chain aryl sulfonate or an unsaturated sulfonate; or

R^2, R^3, R^4, or R^5 is hydrogen; or

R^1, R^2, R^4, or R^5 is an unsubstituted or substituted C_{3-6} cycloalkyl; or

R^6 and R^7 are, independently, a hydrogen or an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl group and n = 0-1; and

(ii) a flavor carrier.

13. The composition of claim 12, wherein R^1, R^2, R^3, R^4, or R^5 is an unsubstituted or substituted C_{1-6} straight chain alkyl, an unsubstituted or substituted C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{3-6} straight chain alkenyl, an unsubstituted or substituted C_{3-6} branched chain alkenyl, an unsubstituted or substituted C_{1-6} alkoxy, nitrile, halo, amino, an unsubstituted or substituted C_{1-6} alkylamino, an unsubstituted or substituted C_{1-6} dialkylamino, carboxy-C_{1-6} alkylamino, carboxy-C_{1-6} dialkylamino, an unsubstituted or substituted acetoxy, carboxy, an unsubstituted or substituted carboxyethyl, an unsubstituted or substituted C_{1-6} alkylcarbonyl, an unsubstituted or substituted C_{1-6} alkylcarboxy, an unsubstituted or substituted C_{1-6} alkylthio, an unsubstituted or substituted C_{1-6} alkyloxy, carboxamido, an unsubstituted or substituted C_{1-6} alkylcarboxamido or an unsubstituted or substituted C_{1-6} dialkylcarboxamido or

R^1, R^2, R^4, or R^5 is an unsubstituted or substituted C_{3-6} cycloalkyl.

14. The composition of claim 12, wherein R^1, R^2, R^3, R^4, or R^5 is a C_{1-6} straight chain alkyl, C_{3-6} branched chain alkyl, an unsubstituted or substituted C_{1-6} alkenyl, an unsubstituted or substituted C_{1-6} acyl, or aryl.

15. The composition of claim 12, wherein the compound is present in an amount from 0.001% to about 30% by weight.

16. The composition of claim 12 wherein the flavor carrier is an emulsifying agent, an encapsulating material, natural or modified starches, polymers, gums, pectins, gelatinous or porous cellular materials, or waxes.

17. A method of improving, enhancing, or modifying the organoleptic properties of a product comprising adding an organoleptically effective amount of a composition of claim 12 to the product.

18. The method of claim 17, wherein the organoleptically effective amount is 0.001% to about 30% by weight.

19. A foodstuff, beverage, confectionary, or pharmaceutical application comprising a composition of claim 12.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 08/68781

A. CLASSIFICATION OF SUBJECT MATTER
IPC(8) - C07F 11/00 (2008.04)
USPC - 512/25; 568/300
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
USPC - 512/25; 568/300

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
USPC - 512/25, $; 568/300, $
Search Terms Below

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
PubWEST (USPT, PGPB, EPAB, JPAB); google.com
Search Terms Used: butanol, derivatives, fragrance, flavor, methoxy, enyloxy, ethanal, methylbutoxy, methylbutyl, ethoxy, acetate, ethoxyacetate

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6,207,857 B1 (Anderson et al.) 27 March 2001 (27.03.2001) entire document, especially Abstract; col. 1, ln. 45-67; col. 3, ln. 1-31; col. 7, ln. 1-7; col. 18, ln. 40-43; col. 19, ln. 22-25	1-19
Y	US 5,081,104 A (Orson et al.) 14 January 1992 (14.01.1992) entire document, especially Abstract; col. 4, ln. 20-35	1-19

☐ Further documents are listed in the continuation of Box C. ☐

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 September 2008 (16.09.2008)	22 SEP 2008
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201	Authorized officer: Lee W. Young PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774

Form PCT/ISA/210 (second sheet) (April 2007)