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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	15149C
First Named Inventor	David Arthur Sturgis
Title	DEODORANT COMPOSITIONS
Priority Mail Express® Label No.	FILING VIA EFS

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

1. ☐ **Fee Transmittal Form** (PTO/SB/17 or equivalent)
2. ☐ **Applicant asserts small entity status.** See 37 CFR 1.27
3. ☐ **Applicant certifies micro entity status.** See 37 CFR 1.29. Applicant must attach form PTO/SB/15A or B or equivalent.
4. ☑ **Specification** [*Total Pages* 23] Both the claims and abstract must start on a new page. *(See MPEP § 608.01(a) for information on the preferred arrangement)*
5. ☐ **Drawing(s)** (35 U.S.C. 113) [*Total Sheets* _____]
6. **Inventor's Oath or Declaration** [*Total Pages* 2] *(including substitute statements under 37 CFR 1.64 and assignments serving as an oath or declaration under 37 CFR 1.63(e))*
   a. ☐ Newly executed (original or copy)
   b. ☑ A copy from a prior application (37 CFR 1.63(d))
7. ☑ **Application Data Sheet** ** See note below.* See 37 CFR 1.76 (PTO/AIA/14 or equivalent)
8. **CD-ROM or CD-R** in duplicate, large table, or Computer Program (*Appendix*)
   ☐ Landscape Table on CD
9. **Nucleotide and/or Amino Acid Sequence Submission** (*if applicable, items a. – c. are required*)
   a. ☐ Computer Readable Form (CRF)
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ACCOMPANYING APPLICATION PAPERS

10. ☐ **Assignment Papers** (cover sheet & document(s))
    Name of Assignee _____
11. ☐ **37 CFR 3.73(c) Statement** (*when there is an assignee*) ☑ **Power of Attorney**
12. ☐ **English Translation Document** (*if applicable*)
13. ☑ **Information Disclosure Statement** (PTO/SB/08 or PTO-1449)
    ☐ Copies of citations attached
14. ☐ **Preliminary Amendment**
15. ☐ **Return Receipt Postcard** (*MPEP § 503*) (*Should be specifically itemized*)
16. ☐ **Certified Copy of Priority Document(s)** (*if foreign priority is claimed*)
17. ☐ **Nonpublication Request** Under 35 U.S.C. 122(b)(2)(B)(i). Applicant must attach form PTO/SB/35 or equivalent.
18. ☐ **Other:** _____

*__Note__: (1) Benefit claims under 37 CFR 1.78 and foreign priority claims under 1.55 **must** be included in an Application Data Sheet (ADS).
(2) For applications filed under 35 U.S.C. 111, the application must contain an ADS specifying the applicant if the applicant is an assignee, person to whom the inventor is under an obligation to assign, or person who otherwise shows sufficient proprietary interest in the matter. See 37 CFR 1.46(b).

19. CORRESPONDENCE ADDRESS

☐ The address associated with Customer Number: _____ **OR** ☐ Correspondence address below

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Signature	/Kathleen Y. Carter/	Date	December 17, 2019
Name (Print/Type)	Kathleen Y. Carter	Registration No. (Attorney/Agent)	60,583

This collection of information is required by 37 CFR 1.53(b). The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
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9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

ABSTRACT

A deodorant stick comprising: at least about 25% of a liquid triglyceride; at least one antimicrobial; a primary structurant with a melting point of at least about 50°C; and less than 8% of secondary structurants having a melting point of at least about 60°C; said stick being free of an aluminum salt; and said stick having a hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM D-1321 needle.

CLAIMS

What is claimed is:

1. A deodorant stick comprising:
    a. at least about 25% of a liquid triglyceride;
    b. at least one antimicrobial; and
    c. a primary structurant with a melting point of at least about 50°Celsius; and

    wherein said stick is free of an aluminum salt.

2. The deodorant stick of claim 1, wherein the antimicrobial is selected from the group consisting of baking soda, magnesium hydroxide, hexamidine, magnesium carbonate, zinc carbonate, thymol, magnesium hydroxide & magnesium carbonate hydroxide, sodium carbonate, magnesium carbonate hydroxide, partially carbonated magnesium hydroxide, calcium carbonate, polyvinyl formate, salycilic acid, niacinamide, cinnamon essential oil, cinnamon bark essential oil, cinnamic aldehyde and combinations thereof.

3. The deodorant stick of claim 1, wherein the antimicrobial is a powder with a water solubility of at most about 90 g/L at 25°C.

4. The deodorant stick of claim 1, wherein the deodorant stick is substantially free of baking soda.

5. The deodorant stick of claim 1, wherein the deodorant stick is substantially free of a synthetic fragrance.

6. The deodorant stick of any claim 1, wherein the deodorant stick is substantially free of silicones.

7. The deodorant stick of claim 1, wherein the deodorant stick comprises from 0% to 5% silicones.

8. The deodorant stick of claim 1, comprising at least about 35% of a liquid triglyceride.

9. The deodorant stick of claim 1, wherein the antimicrobial comprises baking soda.

10. The deodorant stick of claim 1, wherein the antimicrobial comprises magnesium hydroxide.

11. The deodorant stick of claim 1, wherein the stick comprises from about 8% to about 20% of the primary structurant.

12. The deodorant stick of claim 1, wherein the primary structurant has a melting point from about 50°C to 75°C.

13. The deodorant stick of claim 1, comprising less than 5% of secondary structurants having a melting point above 60°C.

14. The deodorant stick of claim 1, wherein said stick comprises less than 8% of secondary structurants having a melting point of at least about 60°Celsius.

15. The deodorant stick of claim 1, wherein said stick has a hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM-D 1321 needle.

DEODORANT COMPOSITIONS

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a continuation of U.S. Patent Application No. 16/021,965, filed June 28, 2018, which claims the benefit of U.S. Provisional Application No. 62/527,180, filed June 30, 2017, and of U.S. Provisional Application No. 62/647,104, filed March 23,2018.

FIELD OF THE INVENTION

The present disclosure relates to deodorant compositions and methods relating thereto.

BACKGROUND OF THE INVENTION

Many consumers are seeking more natural, aluminum-free deodorant offerings, often mostly free of silicones. Consumers also want a good glide, non-sticky, and non-greasy application. This is a challenge, because a mostly silicone-free formula will often use natural oils or natural oil-based triglycerides. But often natural oils, such as coconut oil, bring a large portion of wax with them, often making the resulting deodorant sticks too hard to meet a more preferable product application for both shaven and unshaven underarms. They are also often undesirably greasy due to the non-volatile nature.

Thus there remains a challenge to formulate an aluminum-free, mostly silicone-free deodorant stick that is not too hard.

SUMMARY OF THE INVENTION

A deodorant stick comprising: at least about 25% of a liquid triglyceride; at least one antimicrobial; a primary structurant with a melting point at least about 50°C; and less than 8% of secondary structurants having a melting point of at least about 60°C; said stick being free of an aluminum salt; and said stick having a hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM D-1321 needle.

DETAILED DESCRIPTION OF THE INVENTION

While the specification concludes with claims that particularly point out and distinctly claim the invention, it is believed the present invention will be better understood from the following description.

The present invention can comprise, consist of, or consist essentially of the essential elements and limitations of the invention described herein, as well any of the additional or optional ingredients, components, or limitations described herein.

All percentages, parts and ratios are based upon the total weight of the compositions of the present invention, unless otherwise specified. All such weights as they pertain to listed ingredients are based on the active level and, therefore do not include carriers or by-products that may be included in commercially available materials.

The components and/or steps, including those which may optionally be added, of the various embodiments of the present invention, are described in detail below.

All documents cited are, in relevant part, incorporated herein by reference; the citation of any document is not to be construed as an admission that it is prior art with respect to the present invention.

All ratios are weight ratios unless specifically stated otherwise.

All temperatures are in degrees Celsius, unless specifically stated otherwise.

Except as otherwise noted, all amounts including quantities, percentages, portions, and proportions, are understood to be modified by the word “about”, and amounts are not intended to indicate significant digits.

Except as otherwise noted, the articles “a”, “an”, and “the” mean “one or more”.

Herein, “comprising” means that other steps and other ingredients which do not affect the end result can be added. This term encompasses the terms “consisting of” and “consisting essentially of”. The compositions and methods/processes of the present invention can comprise, consist of, and consist essentially of the essential elements and limitations of the invention described herein, as well as any of the additional or optional ingredients, components, steps, or limitations described herein.

Herein, “effective” means an amount of a subject active high enough to provide a significant positive modification of the condition to be treated. An effective amount of the subject active will vary with the particular condition being treated, the severity of the condition, the duration of the treatment, the nature of concurrent treatment, and like factors.

The term "anhydrous" as used herein means substantially free of added or free water. From a formulation standpoint, this means that the anhydrous deodorant stick compositions of the present invention contain less than about 1%, and more specifically zero percent, by weight of free or added water, other than the water of hydration typically associated with the particulate deodorant active prior to formulation.

The term "ambient conditions" as used herein refers to surrounding conditions under about one atmosphere of pressure, at about 50% relative humidity, and at about 25 °C, unless otherwise specified. All values, amounts, and measurements described herein are obtained under ambient conditions unless otherwise specified.

The term "majority" refers to greater than about 51% of the stated component or parameter.

"Substantially free of" refers to about 2% or less, about 1% or less, or about 0.1% or less of a stated ingredient. "Free of" refers to no detectable amount of the stated ingredient or thing.

The term "volatile" as used herein refers to those materials that have a measurable vapor pressure at 25 °C. Such vapor pressures typically range from about 0.01 millimeters of Mercury (mm Hg) to about 6 mmHg, more typically from about 0.02 mmHg to about 1.5 mmHg; and have an average boiling point at one (1) atmosphere of pressure of less than about 250 °C, more typically less than about 235 °C. Conversely, the term "non-volatile" refers to those materials that are not "volatile" as defined herein.

As consumers seek more natural ingredients in their deodorants, one approach to formulation is to use emollients derived from natural oils. Emollients derived from natural oils are derived from plant sources, such as palm oil or coconut oil. One example of an emollient derived from natural oils may be a liquid triglyceride, defined as liquid at 25°C. Thus, products that hope to emphasize natural ingredients may have a significant amount of a liquid triglyceride, for example.

In order to provide structure to the deodorant stick composition, the formulation may also include a number of waxes and other structurants. However, the structurants used may lead to deodorant sticks that are very hard. For example, a currently marketed product, Comparative Formula 1 below, has 34.15% liquid triglyceride, along with a number of structurants, resulting in a very hard stick, scoring 63 on a needle penetration test under ASTM D-1321 (as described herein). So while Comparative Formula 1 uses consumer-preferred natural ingredients, it does not necessarily provide a good consumer experience when used, given its hardness. Comparative Formula 2 contains even higher levels of structurants, resulting in a harder stick with an even lower hardness score. In comparison, Inventive Examples 1-5, while comprising consumer-preferred natural ingredients, have higher hardness scores, meaning they are softer products.

Table 1:

	Comparative Formula 1	Comparative Formula 2	Inventive Example 1	Inventive Example 2	Inventive Example 3	Inventive Example 4	Inventive Example 5
Hardness (Penetration mm*10)	63	56	97	90	117	86	85
Caprylic/Capric Triglyceride	34.15	31.15	43.4	44.7	42.7	46.9	46.15
Arrowroot Powder	23	23	19	23	23	19	
Stearyl Alcohol	12	12	11.5	10.7	10.7		
Ozokerite					1.5	11	11.75
Castor Wax	3	4	3	3	3		
Baking Soda	12	12	12	12	6	6	
Magnesium Hydroxide					6	6	12.0
Shea Butter	6	6	2	2	2	2	2
Coconut Oil	4.75	4.75	4.5	4.5	4.5	4.5	4.5
Polyglycerol-3 Beeswax	2.5	3.5					
Jojoba Esters 70	1	2					
Glyceryl Caprylate (and) Glyceryl Undecylenate	1	1					
Perfumes	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Cyclodextrin			4			4	4
Tapioca Starch							19.0

It is known that to formulate a solid antiperspirant or deodorant stick, the structurants generally have a melting point above 50°C to provide a stable structure to the stick. The present inventors have discovered that a deodorant stick having at least about 25% of a liquid triglyceride, and that uses a primary structurant that has a melting point of at least about 50°C, in some embodiments from about 50°C to 70°C and in still other embodiments from about 50°C to about 75°C, while limiting the amount of secondary structurants having a melting point of at least about 60°C to 8% or less, can result in a deodorant stick with a hardness from about 80 mm*10 to about 140 mm*10. Such a deodorant stick is able to comprise consumer-perceived natural ingredients, while offering a pleasant consumer experience in terms of its hardness.

A. Structurants

The deodorant compositions of the present invention comprise a suitable concentration of structurants to help provide the compositions with the desired viscosity, rheology, texture and/or product hardness, or to otherwise help suspend any dispersed solids or liquids within the composition.

The primary structurant in the present invention may have a melting point of at least about 50°C, in some embodiments from about 50°C to about 70°C, and in other embodiments from about 50°C to about 75°C, and in other embodiments from about 60°C to 80°C. A primary structurant is defined as the structurant that is present in the composition in the greatest amount (liquid triglycerides are not considered a structurant in this context). Some embodiments may have just a single structurant, so may have only a primary structurant. Other embodiments may have a primary structurant and then secondary structurants, those structurants that are used in a lesser amount than the primary structurant.

The primary structurant may comprise from about 5 % to about 20%, in some cases 7-17% of the deodorant stick. The secondary structurants may cumulatively comprise about 12% or less, or about 8% or less of the deodorant stick, in some embodiments less than about 5%, less than about 3%, or less than about 1% of the deodorant stick. In some embodiments, the deodorant stick may be free of or substantially free of any secondary structurants

In some embodiments, some secondary structurants may have a melting point less than 60°C, and then remaining secondary structurants have a melting point of at least about 60°C. The percentage of secondary structurants having a melting point less than 60°C may not be as significant as the percentage of secondary structurants having a melting point of at least about 60°C, as the higher melting structurants are what contribute more to the hardness of the deodorant stick. So in some embodiments, the secondary structurants having a melting point of at least about 60°C may cumulatively comprise 8% or less of the deodorant stick, in some embodiments less than about 5% of the deodorant stick, less than about 3% of the deodorant stick, or less than about 1% of the deodorant stick. In some embodiments, the deodorant stick may be free of or substantially free of any secondary structurants having a melting point of at least about 60°C.

The term "structurant" as used herein means any material known or otherwise effective in providing suspending, gelling, viscosifying, solidifying, and/or thickening properties to the composition or which otherwise provide structure to the final product form.

Waxes with melting points between 50°C and 70°C include Japan wax, lemon wax, grapefruit wax, beeswax, ceresine, paraffin, hydrogenated jojoba, ethylene glycol distearate,

stearyl stearate, palmityl stearate, stearyl behenate, cetearyl behenate, hydrogenated high erucic acid rapeseed oil, and stearyl alcohol.

Waxes with melting points above 70°C include ozokerite, candelilla, carnauba, espartograss, cork wax, guaruma, rice oil wax, sugar cane wax, ouricury, montan ester wax, sunflower wax, shellac, ozocerite, microcrystalline wax, sasol wax, polyethylenes, polymethylenes, ethylene glycol dipalmitate, ethylene glycol di(12-hydroxystearate), behenyl behenate, glyceryl tribehenate, hydrogenated castor oil (castor wax), and behenyl alcohol.

Waxes with melting points that could vary and possibly fall into either of the two previous groups (depending on factors such as chain length) include C18-C36 triglyceride, Fischer-Tropsch waxes, silicone waxes, C30-50 alkyl beeswax, C20-40 alkyl erucates, C18-38 alkyl hydroxy stearoyl stearates, C20-40 dialkyl esters of dimer acids, C16-40 alkyl stearates, C20-40 alkyl stearates, cetyl ester wax, and spermaceti.

Suitable gelling agents include fatty acid gellants such as fatty acid and hydroxyl or alpha hydroxyl fatty acids, having from about 10 to about 40 carbon atoms, and ester and amides of such gelling agents. Non-limiting examples of such gelling agents include, but are not limited to, 12-hydroxystearic acid, 12-hydroxylauric acid, 16-hydroxyhexadecanoic acid, behenic acid, eurcic acid, stearic acid, caprylic acid, lauric acid, isostearic acid, and combinations thereof. Preferred gelling agents are 12-hydroxystearic acid, esters of 12-hydroxystearic acid, amides of 12-hydroxystearic acid and combinations thereof.

These solid structurants include gelling agents, and polymeric or non-polymeric or inorganic thickening or viscosifying agents. Such materials will typically be solids under ambient conditions and include organic solids, crystalline or other gellants, inorganic particulates such as clays or silicas, or combinations thereof.

The concentration and type of solid structurant selected for use in the deodorant compositions will vary depending upon the desired product hardness, rheology, and/or other related product characteristics. For most structurants suitable for use herein, the total structurant concentration ranges from about 5% to about 35%, more typically from about 10% to about 30%, or from about 7% to about 20%, by weight of the composition.

Non-limiting examples of suitable primary structurants include stearyl alcohol and other fatty alcohols; hydrogenated castor wax (e.g., Castorwax MP80, Castor Wax, etc.); hydrocarbon waxes include paraffin wax, beeswax, carnauba, candelilla, spermaceti wax, ozokerite, ceresin, baysberry, synthetic waxes such as Fisher-Tropsch waxes, and microcrystalline wax; polyethylenes with molecular weight of 200 to 1000 daltons; solid triglycerides; behenyl alcohol, or combinations thereof. The deodorant stick may further comprise one or more structural

elements selected from the group consisting of waxes, natural oils, coconut oil, fractionated coconut oil, jojoba seed oil, olive oil, soybean oil, sunflower oil, and combinations thereof.

Other non-limiting examples of primary structurants suitable for use herein are described in U.S. Pat. No. 5,976,514 (Guskey et al.) and U.S. Pat. No. 5,891,424 (Bretzler et al.), the descriptions of which are incorporated herein by reference.

Non-limiting examples of suitable additional structurants include stearyl alcohol and other fatty alcohols; hydrogenated castor wax (e.g., Castorwax MP80, Castor Wax, etc.); hydrocarbon waxes include paraffin wax, beeswax, carnauba, candelilla, spermaceti wax, ozokerite, ceresin, baysberry, synthetic waxes such as Fisher-Tropsch waxes, and microcrystalline wax; polyethylenes with molecular weight of 200 to 1000 daltons; and solid triglycerides; behenyl alcohol, or combinations thereof.

Other non-limiting examples of additional structurants suitable for use herein are described in U.S. Pat. No. 5,976,514 (Guskey et al.) and U.S. Pat. No. 5,891,424 (Bretzler et al.).

B. Emollients

As discussed, an effective and consumer-preferred emollient may be a liquid triglyceride. Derived directly from plant sources, they are often short chains. Longer chain triglycerides may be used as structurants in deodorant or antiperspirant sticks, but the triglycerides of the present invention are liquid at room temperature (25°C) and tend to be shorter chains. An example may be caprylic / capric triglyceride (coconut oil fractionated).

The present inventive deodorant sticks may comprise at least about 25% of one or more liquid triglyceride, in some embodiments, at least about 30%, at least 35%, at least about 40% , at least about 45%, or at least about 50% liquid triglyceride, by weight of the composition. In some embodiments, the deodorant stick comprises from about 25% to about 60%, by weight of the composition, of one or more liquid triglyceride, from about 25% to about 50%, from about 30% to about 50%, from about 35% to about 60%, from about 35% to about 50%, from about 40% to about 60%, or from about 40% to about 50%, by weight of the composition, of one or more liquid triglyceride. In general, the greater amount of liquid in the formulation, the softer the deodorant stick may be. The more solids in the formulation leads to greater hardness. Because achieving a sufficient softness in a deodorant stick with natural ingredients can be a challenge, it can be beneficial to formulate with higher amounts of liquids such as liquid triglyceride. The level of liquid triglyceride as referred to herein may be the sum total of one or more types of liquid triglyceride in a particular deodorant stick.

In some embodiments, additional emollients may be used, such as plant oils (generally used at less than 10%) including olive oil, coconut oil, sunflower seed oil, jojoba seed oil, avocado oil, canola oil, and corn oil. Additional emollients including mineral oil; shea butter, PPG-14 butyl ether; isopropyl myristate; petrolatum; butyl stearate; cetyl octanoate; butyl myristate; myristyl myristate; C12-15 alkylbenzoate (e.g., Finsolv.TM.); octyldodecanol; isostearyl isostearate; octododecyl benzoate; isostearyl lactate; isostearyl palmitate; isobutyl stearate; dimethicone, and any mixtures thereof.

C. Antimicrobials

The present invention may include one or more antimicrobial compositions. For example, antimicrobials may include, without being limited to, baking soda, hexamidine, magnesium carbonate, zinc carbonate, thymol, magnesium hydroxide, magnesium hydroxide and magnesium carbonate hydroxide (dead sea salt), partially carbonated magnesium hydroxide, sodium carbonate, calcium carbonate, magnesium carbonate hydroxide, cinnamon essential oil, cinnamon bark essential oil, cinnamic aldehyde, polyvinyl formate, salycilic acid, niacinamide, phenoxyethanol, eugenol, linolenic acid, dimethyl succinate, citral, triethyl citrate, sepiwhite, an substituted or unsubstituted 2-pyridinol-N-oxide material (piroctone olamine), and combinations thereof. The deodorant stick may be free of or substantially free of a substituted or unsubstituted 2-pyridinol-N-oxide material.

In general, the total amount of antimicrobial used in the present invention may be from about 0.1% to about 30%, by weight, of the deodorant. Some antimicrobials may be used in amounts as low as about 0.1%, by weight of the deodorant stick, such as if using piroctone olamine or hexamidine as the primary antimicrobial, while others could be as high as about 25% if using magnesium hydroxide or magnesium hydroxide and magnesium carbonate hydroxide as the primary antimicrobial (primary antimicrobial being the antimicrobial present in the composition in the highest amount). In the latter cases, baking soda might still be used at a lower level, such as from about 0.1% to about 6%, as a secondary antimicrobial, or not at all.

Any of the antimicrobials of the present invention may be used as powders. It is believed that antimicrobial powders may provide a better deposition and have more longevity on the skin than antimicrobials delivered in a different form. In addition, it is believed that antimicrobial powders of a certain average particle size, typically from about 1 micron to about 5 microns, may provide a significant increase in antimicrobial efficacy.

Many antimicrobials can be effective at minimizing the skin surface bacteria. However, as a leave-on product where odor may not occur until later, even hours after application, deodorant

antimicrobials are needed that will be effective for long periods of time. So while deodorant antimicrobials may be effective immediately upon application on the skin, it is believed that odor comes back quickly because the bacteria living around the hair follicle can quickly repopulate the skin surface bacteria. Historical approaches using high skin penetrating liquid antimicrobials to affect this region (for example, hexanediol) can cause irritation. Therefore, the present invention may target methods and mechanisms that can more effectively deliver antimicrobials not only to the skin surface, but to the bacteria in and around the hair follicle. While not wanting to be bound to the theory, the inventors of the present inventor believe that powders, specifically powders with an average particle size of less than about 10 microns, in some cases from about 1 micron to about 5 microns, are more efficient at getting into the hair follicle where the bacteria live and repopulate the skin surface. In some embodiments, the antimicrobials may be a combination of larger sized particles and smaller particles that are from 1 to 10 microns. As noted above, solids such as powders can impact the overall hardness of the deodorant stick. In general, greater amounts of powders and structurants increase the deodorant stick's hardness.

The present inventors have discovered that the water solubilities of certain components in the solid stick deodorant have great importance. Some deodorant ingredients will bring in moisture to the batch, which can solvate these components to different extents when the water evaporates and subsequently recondenses as free water in the batch. Certain batch processing conditions (such as a closed top on the tank) could more effectively trap this water in the tank, where it is then free to interact with components of the batch. For example, highly water soluble alkaline powders can contribute negatively towards natural and essential oil stability when dissolved. This is because many natural and essential oils contain a broad range of perfume chemicals, many of which can undergo degradation reactions when exposed to extreme pH or heat. This is why many natural and essential oils have shorter shelf lives than many commercial synthetic chemicals or perfumes. And certain antimicrobials may cause irritation due to high water solubility. Further, high water solubility can lead to grittier products as the more water soluble powders can agglomerate when exposed to moisture released from powders during the heat of manufacture.

Thus, embodiments of the present invention may include an antimicrobial with a low water solubility. An antimicrobial with a low water solubility may be, in some embodiments, an antimicrobial with a water solubility of at most 90 g/L at 25°C, in other embodiments at most 75 g/L at 25°C, or in still other embodiments at most 50 g/L at 25°C.

Materials with a water solubility above 90 g/L @25°C include but are not limited to: potassium carbonate, potassium bicarbonate, sodium carbonate, sodium sesquicarbonate, triethyl citrate, and baking soda. Materials with a water solubility below 90 g/L @25°C include but are not limited to:

beryllium carbonate, magnesium carbonate, calcium carbonate, magnesium hydroxide, magnesium hydroxide and magnesium carbonate hydroxide, partially carbonated magnesium hydroxide, piroctone olamine, hexamidine, zinc carbonate, thymol, polyvinyl formate, salycilic acid, phenoxyethanol, eugenol, linolenic acid, dimethyl succinate, citral, and triethyl citrate. Each of beryllium carbonate, magnesium carbonate, calcium carbonate, magnesium hydroxide, magnesium hydroxide and magnesium carbonate hydroxide, partially carbonated magnesium hydroxide, piroctone olamine, hexamidine, zinc carbonate, thymol, polyvinyl formate, salycilic acid, phenoxyethanol, eugenol, linolenic acid, dimethyl succinate, and citral have a water solubility below 75 g/L @25°C, below 50 g/L @25°C, below 1 g/L @25°C, and below 0.2 g/L @25°C.

D. Antimicrobial Activity

Table 2 below shows the raw material microbial inhibition concentration data tested against two key underarm bacteria strains. As can be seen, the first three listed antimicrobials, lupamin, hexamidine, and piroctone olamine, perform particularly well against the bacteria as raw materials. Also performing well as raw materials are phenoxyethanol, eugenol, linolenic acid, dimethyl succinate, citral, triethyl citrate, and sepiwhite. Also performing moderately well against the bacteria as raw materials were magnesium carbonate/magnesium hydroxide and calcium carbonate.

TABLE 2:

Antimicrobial	*C. mucofaciens*	*S. epidermidis*
Lupamin	*<2 ppm*	*4 ppm*
Hexamidine 36mg/ml H2O	<2 ppm	7 ppm
100 mg/ml piroctone olamine in H2O	< 5 ppm	10 ppm
100% Phenoxyethanol	400 ppm	800 ppm%
Eugenol 99% ETOH	773 ppm	773 ppm
Linolenic Acid 70% ETOH	1093 ppm	1093 ppm
Dimethyl Succinate 98% ETOH	1531 ppm	3062 ppm
Citral 96% ETOH	1500 ppm	1500 ppm
100% Triethyl citrate	1600 ppm	1600 ppm
Sepiwhite 40 mg/ml H2O ins	2000 ppm	1000 ppm
Magnesium Carbonate & Magnesium Hydroxide 50 mg/ml H2O ins	>2500 ppm	>2500 ppm
Ca Carbonate 50 mg/ml H2O ins	>2500 ppm	>2500 ppm
Linoleic acid 100% ETOH	3125 ppm	3125 ppm
Conarom B (beta Bio) 100% ETOH	3125 ppm	3125 ppm
Hexyl Decanol 97% ETOH	6062 ppm	3031 ppm
Ajowan oil 50% ETOH	12500 ppm	6300 ppm
Oregano oil 50% ETOH	12500 ppm	6300 ppm
100% Ethylhexyl glycerin	12500 ppm	12500 ppm
Mineral oil 50% in ETOH	12500 ppm	>50000 ppm
ACH 50% in H2O	25000 ppm	25000 ppm
NaCl 250mg/ml H2O	>25000 ppm	>25000 ppm
Farnesol 95% ETOH	47500 ppm	5937 ppm
Phytol 97% ETOH	>49000 ppm	>49000 ppm
Nerolidol 98% ETOH	>49000 ppm	>49000 ppm
CaCl 500mg/ml H2O	>50000 ppm	>50000 ppm
Isopropyl Myristate 98% ETOH	>59000 ppm	>59000 ppm

While numerous antimicrobials exhibit efficacy against two main bacteria strains that antiperspirants and deodorants try to address, due to regulatory and safety reasons, there are sometimes limits as to how much of a particular antimicrobial may be put into an antiperspirant or deodorant formula. Therefore, there may be a need for multiple antimicrobials to work together in a formula to deliver enough long-term odor protection.

DEODORANT COMPOSITION

The deodorant compositions as described herein can contain a structurant, an antimicrobial, a perfume, and additional chassis ingredient(s). The deodorant composition may further comprise other optional ingredient(s). The compositions can be in the form of a solid stick. The compositions may be free of dipropylene glycol, added water, castor wax, or any combination

thereof. The compositions may be substantially free of or free of a substituted or unsubstituted 2-pyridinol-N-oxide material (piroctone olamine). The deodorant composition may be anhydrous. The deodorant composition may be free of added water.

Hardness

The deodorant compositions of the present invention may have a product or stick hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM D-1321 needle (see Hardness test method below). In some embodiments, the product hardness may be from about 80 to about 120 mm*10, and in others from about 85 to about 100 mm*10.

Perfume

Perfumes are often a combination of many raw materials, known as perfume raw materials. Any perfume suitable for use in a deodorant composition may be used herein. In some embodiments, the deodorant composition may be free of, or substantially free of a synthetic fragrance. A synthetic fragrance is one mostly derived through chemical synthesis where the starting material is no longer intact, but is converted to the new fragrance chemical.

A natural or essential oil fragrance is a result of natural sources wherein the fragrance material is not altered (chemically modified) but extracted from its natural source. These sources can include, but are not limited to, bark, flowers, blossoms, fruits, leaves, resins, roots, bulbs, and seeds. Natural or essential oils go through an extraction process instead of chemical synthesis. Extraction processes include, but are not limited to, maceration, solvent extraction, distillation, expression of a fruit peel, or effleurage.

Additional Chassis Ingredients

Starch

The deodorant composition may comprise a starch powder for dry feel or wetness absorption. Examples include but are not limited to arrowroot powder, tapioca starch, and corn starch.

Solvent

Non-Volatile Organic Fluids

Non-volatile organic fluids may be present, for example, in an amount of about 15% or less, by weight of the composition.

Non-limiting examples of nonvolatile organic fluids include mineral oil, PPG-14 butyl ether, isopropyl myristate, petrolatum, butyl stearate, cetyl octanoate, butyl myristate, myristyl

myristate, C12-15 alkylbenzoate (e.g., Finsolv.TM.), octyldodecanol, isostearyl isostearate, octododecyl benzoate, isostearyl lactate, isostearyl palmitate, and isobutyl stearate.

Other Optional Ingredients

The anhydrous deodorant compositions of the present invention may further comprise any optional material that is known for use in antiperspirant and deodorant compositions or other personal care products, or which is otherwise suitable for topical application to human skin.

One example of an optional ingredient is a scent expression material. Scent expression or release technology may be employed with some or all of the fragrance materials to define a desired scent expression prior to use and during use of the deodorant products. Such scent expression or release technology can include cyclodextrin complexing material, like beta cyclodextrin. Other materials, such as, for example, starch-based matrices or microcapsules may be employed to "hold" fragrance materials prior to exposure to bodily-secretions (e.g., perspiration). The encapsulating material may have release mechanisms other than via a solvent; for example, the encapsulating material may be frangible, and as such, rupture or fracture with applied shear and/or normal forces encountered during application and while wearing. A microcapsule may be made from many materials, one example is polyacrylates.

Another example of optional materials are clay mineral powders such as talc, mica, sericite, silica, magnesium silicate, synthetic fluorphlogopite, calcium silicate, aluminum silicate, bentonite and montomorillonite; pearl pigments such as alumina, barium sulfate, calcium secondary phosphate, calcium carbonate, titanium oxide, finely divided titanium oxide, zirconium oxide, zinc oxide, hydroxy apatite, iron oxide, iron titrate, ultramarine blue, Prussian blue, chromium oxide, chromium hydroxide, cobalt oxide, cobalt titanate, titanium oxide coated mica; organic powders such as polyester, polyethylene, polystyrene, methyl methacrylate resin, cellulose, 12-nylon, 6-nylon, styrene-acrylic acid copolymers, poly propylene, vinyl chloride polymer, tetrafluoroethylene polymer, boron nitride, fish scale guanine, laked tar color dyes, laked natural color dyes; and combinations thereof.

Talc, if used at higher levels can produce a significant amount of white residue which has been found to be a consumer negative for product acceptance. Therefore it is best to limit the composition to less than 10%, less than about 8%, less than about 6%, or less than about 3%, by weight of the composition.

Nonlimiting examples of other optional materials include emulsifiers, distributing agents, antimicrobials, pharmaceutical or other topical active, preservatives, surfactants, chelants, and so forth. Examples of such optional materials are described in U.S. Pat. No. 4,049,792 (Elsnau); U.S.

Pat. No. 5,019,375 (Tanner et al.); and U.S. Pat. No. 5,429,816 (Hofrichter et al.); which descriptions are incorporated herein by reference.

Method of Making

The deodorant stick products of the present invention may be made by mixing all the components of the products in an open-top or vented tank. Many powders come with bound moisture, especially naturally high moisture powders like starches. In a mostly anhydrous process with waxes, melting the waxes above their melt point can release this bound water as the batch temperature increases. In a closed tank process this water vapor will condense in the tank and drip back into the batch as water. This water can interact with the most water soluble ingredients in the batch to have negative effects on the product, including releasing the pH of any antimicrobial ingredient, which can then degrade any perfume ingredients in the batch. Additionally, the condensed water can interfere with the wax and produce a stick softer than intended.

The present invention reduces the risk of these negative consequences. The ideal process remedy for this behavior is to produce the batches in one of four ways:

1. An open tank system where the water vapor can leave the batch tank to reduce or eliminate condensation.
2. A vented closed tank to also remove water vapor during the batch process.
3. A dual phase process where the moisture containing powders can be put into the cold phase separate from the wax phase which is heated. These two phases are then mixed before filling.
4. A low residence time batch process for a closed system, where the product has less than 3 hours residence time above 50°C to reduce the rate of reaction from the moisture.

A method of making a deodorant composition or stick may comprise the steps of combining any of the herein described deodorant composition components in an open tank system or a vented closed tank. The components may be mixed, heated, and then cooled into a stick product. In some embodiments, the deodorant components may comprise at least about 40% of a liquid triglyceride, by weight of the composition, and an antimicrobial in an open tank system, heating the components, mixing the components, and cooling the components.

Test Methods

Hardness Test Method – Penetration measurement for deodorant finished products

The penetration test is a physical test method that provides a measure of the firmness of waxy solids and extremely thick creams and pastes with penetration values not greater than 250

when using a needle for D1321. The method is based on the American Society for Testing and Materials Methods D-5, D1321 and D217 and DIN 51 579 and is suitable for all solid antiperspirant and deodorant products.

A needle or polished cone of precisely specified dimensions and weight is mounted on the bottom of a vertical rod in the test apparatus. The sample is prepared as specified in the method and positioned under the rod. The apparatus is adjusted so that the point of the needle or cone is just touching the top surface of the sample. Consistent positioning of the rod is critical to the measured penetration value. The rod is then released and allowed to travel downward, driven only by the weight of the needle (or cone) and the rod. Penetration is the tenths of a millimeter travelled following release.

APPARATUS	SUGGESTED TYPE (OR EQUIVALENT)
Penetrometer with Timer (see attached drawings Attachment 1 and Attachment 2)	Penetrometer Suitable For ASTM D-5 and D-1321 methods; Examples: Precision or Humboldt Universal Penetrometer (Humboldt Manufacturing, Schiller Park, IL USA) *or* Penetrometer Model PNR10 or PNR12 (Petrolab USA or PetroTest GmbH).
Penetration Needles	ANTIPERSPIRANT or DEODORANT SOLIDS can use: • Needles as specified for ASTM Method D-5, NIST Certified, Fisher Scientific #01-512. • Needles as specified for ASTM Method D 1321 /DIN 51 579, Officially certified, Taper-Tipped needle, No. H-1310, Humboldt Mfg.

General Instructions – All Penetrometers - Keep the instrument and needles/probes clean at all times, free from dust and grime. When not in use, store needles in a suitable container to avoid damage. Periodic calibration should confirm:

Electronic Timer is correctly set. Verify against an independent stopwatch if unsure.

Shaft falls without visible signs of frictional resistance.

Ensure the total weight of the shaft and needle is 50 ± 0.2 grams when the shaft is in free fall. Note: for modern, automated or digital systems this may be performed automatically and confirmed through annual calibration.

At time of use confirm:

Electronic Timer is correctly set to 5.0 seconds.

The appropriate needle is installed and is clean, straight and without obvious defects (visual inspection)

The penetrometer is level and the shaft is clean, straight and falls freely (visual inspection)

Once level, avoid shifting the position of the unit to maintain level.

Sample Preparation and Measurement

1. On a deodorant stick that has cooled ambiently to a temperature between 22ºC and 26°C for at least 24 hours, slice off top ½ inch of product to achieve a flat surface with a wire cutter drawn across the upper lip of the canister.

2. For the first sample to be tested, lubricate the needle by gently wiping with a lint-free tissue coated with a small amount of the product to be tested. This small amount is typically taken from the shaved top.

3. Place the canister in the appropriate location for the measurement. Locate the sample so the needle will penetrate the product 9-11mm from the inside of the canister wall on the long axis.

4. Using the coarse and fine adjustments, align the height of the penetrometer mechanism head so that the point of the penetrating needle is just touching the surface of the sample.

A weak light at the side of the penetrometer which casts a shadow of the needle on the surface of the sample may be helpful in determining this contact. When a light area on the sample cannot be seen at the end of the tip of the needle's shadow, the needle height over the sample is correctly adjusted. The light should not be strong enough to heat or melt the sample surface. The needle should be just close enough to scratch the sample surface.

5. Perform the penetration measurement at this location by releasing the needle. Record the result.

6. Repeat Steps 2 through 4 at the other test point, i.e., at the other point 9-11mm inside of the canister wall on the long axis.

To report results, units for penetration are tenths of a millimeter (1/10mm = 100microns). For example, a result of 80 units is 80 mm*10 or 8 mm. Report the average results of at least 4 total measurements from 2 different sticks, report to the nearest tenth of a millimeter.

Tier 1 Anaerobic MIC Assay

The data in Table 2 above was generated with the following test method. The purpose of this assay is to determine if a compound or formulation has an antimicrobial effect *in vitro*.

It is understood that when not specifically noted in this procedure:

a) All materials, reagents and equipment required for this procedure are of appropriate design and condition of cleanliness and/or sterility as determined by their intended use.

b) The operator has been trained in aseptic technique and has been qualified to perform the procedure and accurately interpret the results.

c) All media required for this procedure was manufactured by a reputable commercial source egg. Difco, Merck etc. and has been stored and prepared as per manufacturer's instructions.

d) All routine laboratory controls, including but not limited to, media function and growth promotion tests, verification of sterility and use of positive and negative controls are being conducted.

Procedure: (All procedures performed in anaerobic chamber except where noted)

1. Apparatus

Incubator at 37°C; 20-200ul 12 channel pipette; 5-50ul 12 channel pipette; 1250ul 8 channel Thermo Scientific Matrix pipette; 96 well plate shaker (located in incubator); Beckman Coulter deep well cap mat #267005; Beckman Coulter deep 96 well plates #267007; Falcon 96 well tissue culture plates #353072; Vortexer ; Culture tubes/caps Disposable sterile gloves; Sterile petri dishes; Standard microbiological lab equipment (sterile pipettes, syringes, tips, loops, etc.); Glass bottles/flasks for media; Autoclave; Parafilm; Spectrophotometer.

2. Media

0.9% or 0.85% saline solution

BHI agar supplemented with 1% Tween 80

BHI media supplemented with 1% Tween 80

3. Microbial Strains

Staphylococcus epidermidis (clinical isolate)

Corynebacterium mucofaciens (clinical isolate)

4. Test Procedure

<u>Inoculum Preparation</u>

Prior to testing streak organisms for isolation on BHI with 1% Tween 80 plates, wrap with parafilm and place in 37°C incubator. When isolated colonies appear remove one

representative colony from each plate and place each in 5 ml of BHI with 1% Tween 80 media. Incubate at 37^{O}C with shaking overnight. Inoculate 20ml BHI with 1% Tween 80 (per 96 deep well plate to be tested) with 20ul of the overnight culture (1-1000 dilution).

Master Plate Preparation

Compounds/formulations to be tested are diluted across a 96 deep well plate as shown below (for a 1% stock solution). 800ul of 0.85% saline is added to wells A1 and B1 (as these will be the negative and positive control respectively). 800ul each 1% stock solution + positive control are added to wells C1 through H1. 400ul 0.85% saline are added to all other wells. 400ul is then removed from #1 well added to the #2 well and mixed. This is then continued across the plate resulting in a 50% dilution between wells across the plate (this can be easily accomplished with an automatic 8 channel Matrix pipette set to withdraw, dispense and mix).

	1	2	3	4	5	6	7	8	9	10	11	12
A	800ul saline	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
B	800ul saline	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
C	800ul + control	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
D	800ul compound 1	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
E	800ul compound 2	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
F	800ul compound 3	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
G	800ul compound 4	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl
H	800ul compound 5	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl	400ul NaCl

	1	2	3	4	5	6	7	8	9	10	11	12
A	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank	Media Blank
B	Pos	Pos	Pos	Pos	Pos	Pos	Pos	Pos	Pos	Pos	Pos	Pos
C	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05
D	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05
E	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05
F	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05
G	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05
H	0.1	0.05	0.025	0.0125	0.00625	0.003125	0.0015625	0.00078125	0.000390625	0.0001953313	9.765625E-05	4.882812E-05

Test Plate Preparation

In row A of a 96 deep well plate pipette 180ul of sterile BHI with 1% Tween 80 as a negative growth control. All other wells receive 180ul of inoculum. From the master plate introduce 20ul to the corresponding row in the test plate using an 8-channel pipette. Loaded plates are placed on a plate shaker in the 37^{O}C incubator and incubated overnight. The next day read the O.D. 600 on a plate reader. The MIC is the last well from the right that has no bacterial growth.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm."

It should be understood that every maximum numerical limitation given throughout this specification will include every lower numerical limitation, as if such lower numerical limitations were expressly written herein. Every minimum numerical limitation given throughout this specification will include every higher numerical limitation, as if such higher numerical limitations were expressly written herein. Every numerical range given throughout this specification will include every narrower numerical range that falls within such broader numerical range, as if such narrower numerical ranges were all expressly written herein.

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DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

Title of Invention	DEODORANT COMPOSITIONS

As the below named inventor, I hereby declare that:

This declaration is directed to:

☐ The attached application, or

■ United States application or PCT international application number 16/021,965 filed on June 28, 2018.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

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LEGAL NAME OF INVENTOR

Inventor: David Arthur Sturgis Date (Optional): 7/18/18

Signature: /David Arthur Sturgis/

Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form or must have been previously filed. Use an additional PTO/AIA/01 form for each additional inventor.

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☒ United States application or PCT international application number 16/021,965 filed on June 28, 2018.

The above-identified application was made or authorized to be made by me.

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LEGAL NAME OF INVENTOR

Inventor: Lindsey Michelle Britt Date (Optional): 07/18/2018

Signature: /Lindsey Michelle Britt/

Note: An application data sheet (PTO/SB/14 or equivalent), including naming the entire inventive entity, must accompany this form or must have been previously filed. Use an additional PTO/AIA/01 form for each additional inventor.

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

Secrecy Order 37 CFR 5.2:

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Inventor Information:

Inventor 1 Remove

Legal Name

Prefix	Given Name	Middle Name	Family Name	Suffix
	David	Arthur	Sturgis	

Residence Information (Select One) ● US Residency Non US Residency Active US Military Service

City	Montgomery	State/Province	OH	Country of Residence	US

Mailing Address of Inventor:

Address 1	One Procter & Gamble Plaza
Address 2	
City	Cincinnati
State/Province	OH
Postal Code	45202
Country	US

Inventor 2 Remove

Legal Name

Prefix	Given Name	Middle Name	Family Name	Suffix
	Lindsey	Michelle	Britt	

Residence Information (Select One) ◉ US Residency Non US Residency Active US Military Service

City	West Chester	State/Province	OH	Country of Residence	US

Mailing Address of Inventor:

Address 1	One Procter & Gamble Plaza
Address 2	
City	Cincinnati
State/Province	OH
Postal Code	45202
Country	US

All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the **Add** button. Add

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below.
For further information see 37 CFR 1.33(a).

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

☐ **An Address is being provided for the correspondence Information of this application.**

Customer Number	27752		
Email Address	Carter.ky@pg.com	Add Email	Remove Email

Application Information:

Title of the Invention	DEODORANT COMPOSITIONS		
Attorney Docket Number	15149C	**Small Entity Status Claimed**	☐
Application Type	Nonprovisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)		**Suggested Figure for Publication (if any)**	

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

Publication Information:

☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)

☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application **has not and will not** be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32).
Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

Please Select One:	● Customer Number	US Patent Practitioner	○ Limited Recognition (37 CFR 11.9)
Customer Number	27752		

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.
When referring to the current application, please leave the "Application Number" field blank.

Prior Application Status	Pending		Remove
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)
	Continuation of	16/021965	2018-06-28
Prior Application Status	Expired		Remove
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)
16/021965	Claims benefit of provisional	62/527180	2017-06-30
Prior Application Status	Expired		Remove
Application Number	Continuity Type	Prior Application Number	Filing or 371(c) Date (YYYY-MM-DD)
16/021965	Claims benefit of provisional	62/647104	2018-03-23

Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the **Add** button. Add

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)[i] the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Remove

Application Number	Country[i]	Filing Date (YYYY-MM-DD)	Access Code[i] (if applicable)

Additional Foreign Priority Data may be generated within this form by selecting the **Add** button. Add

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

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☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.
NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Applicant 1 — Remove

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

Clear

● Assignee	Legal Representative under 35 U.S.C. 117	Joint Inventor
Person to whom the inventor is obligated to assign.	Person who shows sufficient proprietary interest	

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

Name of the Deceased or Legally Incapacitated Inventor:

If the Applicant is an Organization check here. ☒

Organization Name	The Procter & Gamble Company		
Mailing Address Information For Applicant:			
Address 1	Attention: Chief Patent Counsel		
Address 2	One Procter & Gamble Plaza		
City	Cincinnati	**State/Province**	OH
Country	US	Postal Code	45202
Phone Number	513-983-2114	Fax Number	
Email Address	Carter.ky@pg.com		

Additional Applicant Data may be generated within this form by selecting the Add button. Add

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
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Assignee 1

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

Remove

If the Assignee or Non-Applicant Assignee is an Organization check here. ☐

Prefix	**Given Name**	Middle Name	**Family Name**	Suffix

Mailing Address Information For Assignee including Non-Applicant Assignee:

Address 1			
Address 2			
City		**State/Province**	
Country i		Postal Code	
Phone Number		Fax Number	
Email Address			

Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button. Add

Signature:

Remove

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). **However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).**

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

Signature	/Kathleen Y. Carter/			Date (YYYY-MM-DD)	2019-12-17
First Name	Kathleen Y.	Last Name	Carter	Registration Number	60,583

Additional Signature may be generated within this form by selecting the Add button. Add

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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	15149C
	Application Number	
Title of Invention	DEODORANT COMPOSITIONS	

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1 The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3 A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent CooperationTreaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Doc Code: PA..
Document Description: Power of Attorney

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TRANSMITTAL FOR POWER OF ATTORNEY TO ONE OR MORE REGISTERED PRACTITIONERS

NOTE: This form is to be submitted with the Power of Attorney by Applicant form (PTO/AIA/82B) to identify the application to which the Power of Attorney is directed, in accordance with 37 CFR 1.5, unless the application number and filing date are identified in the Power of Attorney by Applicant form. If neither form PTO/AIA/82A nor form PTO/AIA82B identifies the application to which the Power of Attorney is directed, the Power of Attorney will not be recognized in the application.

Application Number	Not Yet Assigned
Filing Date	December 17, 2019
First Named Inventor	David Arthur STURGIS
Title	DEODORANT COMPOSITIONS
Art Unit	Not Yet Assigned
Examiner Name	Not Yet Assigned
Attorney Docket Number	15149C

SIGNATURE of Applicant or Patent Practitioner

Signature	/Kathleen Y. Carter/	Date (Optional)	December 17, 2019
Name	Kathleen Y. Carter	Registration Number	60,583
Title (if Applicant is a juristic entity)			
Applicant Name (if Applicant is a juristic entity)	The Procter & Gamble Company		

NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4(d) for signature requirements and certifications. If more than one applicant, use multiple forms.

☑ *Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.131, 1.32, and 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Doc Code: PA..
Document Description: Power of Attorney

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POWER OF ATTORNEY BY APPLICANT

I hereby revoke all previous powers of attorney given in the application identified in either the attached transmittal letter or the boxes below.

Application Number	Filing Date

(Note: The boxes above may be left blank if information is provided on form PTO/AIA/82A.)

☑ I hereby appoint the Patent Practitioner(s) associated with the following Customer Number as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above: 27752

OR

☐ I hereby appoint Practitioner(s) named in the attached list (form PTO/AIA/82C) as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the patent application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above. (Note: Complete form PTO/AIA/82C.)

Please recognize or change the correspondence address for the application identified in the attached transmittal letter or the boxes above to:

☑ The address associated with the above-mentioned Customer Number

OR

☐ The address associated with Customer Number:

OR

☐ Firm or Individual Name					
Address					
City		State		Zip	
Country					
Telephone		Email			

I am the Applicant (if the Applicant is a juristic entity, list the Applicant name in the box):

The Procter & Gamble Company

☐ Inventor or Joint Inventor (title not required below)

☐ Legal Representative of a Deceased or Legally Incapacitated Inventor (title not required below)

☑ Assignee or Person to Whom the Inventor is Under an Obligation to Assign (provide signer's title if applicant is a juristic entity)

☐ Person Who Otherwise Shows Sufficient Proprietary Interest (e.g., a petition under 37 CFR 1.46(b)(2) was granted in the application or is concurrently being filed with this document) (provide signer's title if applicant is a juristic entity)

SIGNATURE of Applicant for Patent

The undersigned (whose title is supplied below) is authorized to act on behalf of the applicant (e.g., where the applicant is a juristic entity).

Signature	/Jay A Krebs/	Date (Optional)	
Name	Jay A. Krebs		
Title	Senior Counsel, Global Patent Systems Manager, Assistant Secretary, The Procter & Gamble Company		

NOTE: Signature - This form must be signed by the applicant in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. If more than one applicant, use multiple forms.

☑ Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.131, 1.32, and 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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POWER OF ATTORNEY BY APPLICANT

No more than ten (10) patent practitioners total may be appointed as set forth below by name and registration number. This page need not be submitted if appointing the Patent Practitioner(s) associated with a Customer Number (see form PTO/AIA/82B):

Name	Registration Number

Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.	:	Not Yet Assigned
Inventor(s)	:	STURGIS, et al.
Filed	:	December 17, 2019
Art Unit	:	Not Yet Assigned
Examiner	:	Not Yet Assigned
Docket No.	:	15149C
Confirmation No.	:	Not Yet Assigned
Customer No.	:	27752
Title	:	DEODORANT COMPOSITIONS

INFORMATION DISCLOSURE STATEMENT

Pursuant to 37 C.F.R. §§ 1.56, 1.97 and 1.98, it is respectfully requested that the document(s) cited in this submission be carefully considered by the Examiner and made of record in this case. As provided in §1.97(g), no representation is made or intended that a thorough art search was made. As provided in 37 C.F.R. §1.97(h), this Information Disclosure Statement does not constitute an admission of any kind, and specifically is not an admission that the document(s) cited in this submission is (are), or is (are) considered to be, material to the patentability of the above-identified patent application, as defined in 37 C.F.R. §1.56(b).

This Information Disclosure Statement is being submitted under 37 C.F.R. §1.97(b)(1) together with the patent application or within three months of the filing date of the patent application, or under 37 C.F.R. §1.97(b)(2) within three months of entry into the national stage in an international application. Therefore, no fee is believed to be due.

Additional Items to be Noted by the Examiner

In addition to the cited references that were previously considered in prior application U.S. Patent Application Serial No. 16/021,965, filed 06/28/2018, additional references are cited. A copy of each newly cited foreign and/or non-patent reference is provided with this statement. All other documents cited in this submission are U.S. patent documents. Priority is claimed to said application under 35 U.S.C. §120. Accordingly, copies of previously considered foreign and/or non-patent references are not provided with this Statement, pursuant to 37 C.F.R. §1.98(d). The scope of the claims of the present application may be different in scope from any prior applications, including any previous disclaimers. Thus, the Patent Office is reminded that the art cited in any prior application may need to be revisited for the examination of the claims in the present application.

Respectfully submitted,

THE PROCTER & GAMBLE COMPANY

/Kathleen Y. Carter/
Kathleen Y. Carter
Registration No. 60,583
(513) 983-2114

Date: December 17, 2019
Customer No. 27752

PTO/SB/08a (03-15)
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Filing Date	2019-12-17
First Named Inventor	David Arthur Sturgis
Art Unit	
Examiner Name	
Attorney Docket Number	15149C-US

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Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4049792		1977-09-20	Elsnau	
	2	5019375		1991-05-28	Tanner , et al.	
	3	5429816		1995-07-04	Hofrichter , et al.	
	4	5891424		1999-04-06	Bretzler, deceased , et al.	
	5	5976514		1999-11-02	Guskey , et al.	

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OR

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The information provided by you in this form will be subject to the following routine uses:

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Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	
Examiner Name	
Attorney Docket Number	–15149C-US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4906454		1990-03-06	Melanson, Jr.	
	2	5972319		1999-10-26	Linn , et al.	
	3	6177066	B1	2001-01-23	Pataut	
	4	6503944		2003-01-07	Chanchani	
	5	7425321	B2	2008-09-16	Lemoine	
	6	8460720	B2	2013-06-11	Tom's of Maine, Inc.	
	7	8574559	B2	2013-11-05	Banowski	
	8	8672067	B2	2014-03-18	Toyota Jidosha Kabushiki Kaisha	

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	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

	9	9468596	B2	2016-10-18	Eizen	
	10	9949920	B2	2018-04-24	Hakim Noha N.	

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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20030235546	A1	2003-12-25	Mattai, Jairajh ; et al.	
	2	20050281767	A1	2005-12-22	Walling et al.	
	3	20110076309	A1	2011-03-31	MISNER H STEVEN; FAN AIXING; KILPATRICK-LIVERMAN L	
	4	20110196786	A1	2017-07-13	Avon Products, Inc.	
	5	20160235661	A1	2016-08-18	AXIM Biotechnologies, Inc.	
	6	20190000730	A1	2019-01-03	DEAD SEA BROMINE COMPANY LTD.	

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Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1	International Journal Cosmetic Science, 2003, 25, pp. 127-135 - Iron Sequestration on Skin: A New Route to Improved Deodorancy	
	2	Nivea Fresh Natural Deodorant, pulled from the Internet (www.GNPD.com) 3 pages	

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EXAMINER SIGNATURE

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Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	5744146	A	1998-04-28	Peters, Kimberly T	
	2	9517193	B2	2016-12-13	Fares, Hani M.	Equiv. for WO 2013/052454 A1
	3	6485717	B1	2002-11-26	Scavone, et al.	
	4	7033579	B1	2006-04-25	Scavone	

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	1	20170252288	A1	2017-09-07	Lesniak, Ewelina	Equiv. for WO 2016/048425 A1
	2	20020086039	A1	2002-07-04	Lee, et al.	

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	3	20070203240	A1	2007-08-30	Oblong, et al.	
	4	20190000747	A1	2019-01-03	Sturgis, et al.	

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	1	International Search Report; International Application No. PCT/US2019/022660; date of mailing 4 June 2019; 14 pages.	

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Signature		Date (YYYY-MM-DD)	
Name/Print		Registration Number	

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Electronic Patent Application Fee Transmittal

Application Number:	
Filing Date:	
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur STURGIS
Filer:	Kathleen Yates Carter/Kelley Werdman
Attorney Docket Number:	15149C

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
UTILITY APPLICATION FILING	1011	1	300	300
UTILITY SEARCH FEE	1111	1	660	660
UTILITY EXAMINATION FEE	1311	1	760	760
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Miscellaneous:				
			Total in USD ($)	**1720**

Electronic Acknowledgement Receipt

EFS ID:	38048919
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur STURGIS
Customer Number:	27752
Filer:	Kathleen Yates Carter/Kelley Werdman
Filer Authorized By:	Kathleen Yates Carter
Attorney Docket Number:	15149C
Receipt Date:	17-DEC-2019
Filing Date:	
Time Stamp:	06:36:27
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$1720
RAM confirmation Number	E2019BG736466963
Deposit Account	162480
Authorized User	Kelley Werdman

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.16 (National application filing, search, and examination fees)

37 CFR 1.17 (Patent application and reexamination processing fees)

37 CFR 1.19 (Document supply fees)

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Transmittal of New Application	15149C_Transmittal.pdf	279583 5ecfeb13061d484fc351a4e214acfec3d2acf210	no	2
Warnings:					
Information:					
2		15149C_APP.pdf	549899 7420a30195962ec3da92a7c23e20973307f97d0b	yes	23

Multipart Description/PDF files in .zip description

Document Description	Start	End
Abstract	23	23
Claims	21	22
Specification	1	20

Warnings:					
Information:					
3	Oath or Declaration filed	15149C_DEC.pdf	152498 e6a6cd0ccf1ad0479bd68a75ff2dd28dc8057d80	no	2
Warnings:					
The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing					
Information:					
4	Application Data Sheet	15149C_ADS.pdf	1793468 c78951db73d14c839588b0d53e9e60c98693bd1f	no	9
Warnings:					
Information:					

5	Power of Attorney	15149C_Power_of_Attorney_and_Transmittal.pdf	196740 9889ecb04d941b94b2278b33e6bab18b9632dc46	no	4
Warnings:					
Information:					
6	Transmittal Letter	15149C_IDS_Letter.pdf	17746 0f4a9806833d3d44e50d69ebb924a2b2027f78fb	no	2
Warnings:					
Information:					
7	Information Disclosure Statement (IDS) Form (SB08)	15149C_IDSList_10-15-18.pdf	1051054 afcaa16a6aec8ea335015130195e96240b58c5d0	no	4
Warnings:					
Information:					
8	Information Disclosure Statement (IDS) Form (SB08)	15149C_IDSList_02-07-19.pdf	1053398 933242572aef3baa5a640e9d8b0ee2fe8cbe2e06	no	5
Warnings:					
Information:					
9	Information Disclosure Statement (IDS) Form (SB08)	15149C_SIDS2_1.pdf	1079103 c837d746ffdddb7fb2f4a0b91ccfa3670ca7fc21	no	4
Warnings:					
Information:					
10	Fee Worksheet (SB06)	fee-info.pdf	34805 bbae638d38c002a75eda575dcd5aa20be47d22e9	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	6208294		

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 16/716,542

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY FEE($)
BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A			N/A	300
SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A	N/A			N/A	660
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A			N/A	760
TOTAL CLAIMS (37 CFR 1.16(i))	15 minus 20 =	*			OR	x 100 =	0.00
INDEPENDENT CLAIMS (37 CFR 1.16(h))	1 minus 3 =	*				x 460 =	0.00
APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						0.00
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))							0.00
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL			TOTAL	1720

APPLICATION AS AMENDED - PART II

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY ADDITIONAL FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY ADDITIONAL FEE($)
AMENDMENT A	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	RATE($)	ADDITIONAL FEE($)		RATE($)	ADDITIONAL FEE($)
AMENDMENT B	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".
The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.

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UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
16/716,542	12/17/2019	1616	1720	15149C	15	1

CONFIRMATION NO. 3081

FILING RECEIPT

27752
THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

OC000000114093594

Date Mailed: 01/23/2020

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

Please verify the accuracy of the data presented on this receipt. If an error is noted on this Filing Receipt, please submit a written request for a corrected Filing Receipt, including a properly marked-up ADS showing the changes with strike-through for deletions and underlining for additions. If you received a "Notice to File Missing Parts" or other Notice requiring a response for this application, please submit any request for correction to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections provided that the request is grantable.

Inventor(s)

David Arthur Sturgis, Montgomery, OH;
Lindsey Michelle Britt, West Chester, OH;

Applicant(s)

The Procter & Gamble Company, Cincinnati, OH;

Power of Attorney: The patent practitioners associated with Customer Number 27752

Domestic Priority data as claimed by applicant

This application is a CON of 16/021,965 06/28/2018 PAT 10543164
which claims benefit of 62/527,180 06/30/2017
and claims benefit of 62/647,104 03/23/2018

Foreign Applications for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 01/21/2020

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 16/716,542**

Projected Publication Date: 04/30/2020

Non-Publication Request: No

Early Publication Request: No

Title

DEODORANT COMPOSITIONS

Preliminary Class

424

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at http://www.uspto.gov/web/offices/pac/doc/general/index.html.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, http://www.stopfakes.gov. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific

countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign AssetsControl, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

SelectUSA

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop

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UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
16/716,542	12/17/2019	David Arthur Sturgis	15149C

CONFIRMATION NO. 3081

27752
THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

PUBLICATION NOTICE

OC000000116634706

Title:DEODORANT COMPOSITIONS

Publication No.US-2020-0129415-A1
Publication Date:04/30/2020

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

The publication may be accessed through the USPTO's publically available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently http://www.uspto.gov/patft/.

The publication process established by the Office does not provide for mailing a copy of the publication to applicant. A copy of the publication may be obtained from the Office upon payment of the appropriate fee set forth in 37 CFR 1.19(a)(1). Orders for copies of patent application publications are handled by the USPTO's Public Records Division. The Public Records Division can be reached by telephone at (571) 272-3150 or (800) 972-6382, by facsimile at (571) 273-3250, by mail addressed to the United States Patent and Trademark Office, Public Records Division, Alexandria, VA 22313-1450 or via the Internet.

In addition, information on the status of the application, including the mailing date of Office actions and the dates of receipt of correspondence filed in the Office, may also be accessed via the Internet through the Patent Electronic Business Center at www.uspto.gov using the public side of the Patent Application Information and Retrieval (PAIR) system. The direct link to access this status information is currently https://portal.uspto.gov/pair/PublicPair. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

Further assistance in electronically accessing the publication, or about PAIR, is available by calling the Patent Electronic Business Center at 1-866-217-9197.

Office of Data Managment, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	12/17/2019	David Arthur Sturgis	15149C	3081

27752 7590 08/04/2020

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

EXAMINER	
MERCIER, MELISSA S	

ART UNIT	PAPER NUMBER
1615	

NOTIFICATION DATE	DELIVERY MODE
08/04/2020	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

centraldocket.im@pg.com
mayer.jk@pg.com
pair_pg@firsttofile.com

Office Action Summary	Application No. 16/716,542	Applicant(s) Sturgis et al.	
	Examiner MELISSA S MERCIER	Art Unit 1615	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☐ Responsive to communication(s) filed on _____.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL.** 2b) ☑ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5) ☑ Claim(s) 1-15 is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6) ☐ Claim(s) _____ is/are allowed.

7) ☑ Claim(s) 1-15 is/are rejected.

8) ☐ Claim(s) _____ is/are objected to.

9) ☐ Claim(s) _____ are subject to restriction and/or election requirement

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov.**

Application Papers

10)☐ The specification is objected to by the Examiner.

11)☐ The drawing(s) filed on _____ is/are: a)☐ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☐ All b)☐ Some** c)☐ None of the:

1.☐ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☑ Notice of References Cited (PTO-892)

2) ☑ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date _____.

4) ☐ Other: _____.

DETAILED ACTION

Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

Information Disclosure Statement

Receipt of the Information Disclosure Statements (3) filed on December 19, 2019 is acknowledged. Signed copies are attached to this office action. US Patent 8,672,067 drawn to a Vehicle Front Portion Structure and US 2011/0196786 drawn to determining trustworthiness and familiarity of users of an electronic billing and payment system have not been considered on one of the IDS dated December 19, 2019.

Claim Objections

Claim 2 is objected to because of the following informalities: salicylic is spelled incorrectly as "salycilic". Appropriate correction is required.

Double Patenting

The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have

been obvious over, the reference claim(s). See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); *In re Longi*, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); *In re Van Ornum*, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); *In re Vogel*, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); *In re Thorington*, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on nonstatutory double patenting provided the reference application or patent either is shown to be commonly owned with the examined application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement. See MPEP § 717.02 for applications subject to examination under the first inventor to file provisions of the AIA as explained in MPEP § 2159. See MPEP §§ 706.02(l)(1) - 706.02(l)(3) for applications not subject to examination under the first inventor to file provisions of the AIA. A terminal disclaimer must be signed in compliance with 37 CFR 1.321(b).

The USPTO Internet website contains terminal disclaimer forms which may be used. Please visit www.uspto.gov/patent/patents-forms. The filing date of the application in which the form is filed determines what form (e.g., PTO/SB/25, PTO/SB/26, PTO/AIA/25, or PTO/AIA/26) should be used. A web-based eTerminal Disclaimer may be filled out completely online using web-screens. An eTerminal Disclaimer that meets all requirements is auto-processed and approved immediately upon submission. For more information about eTerminal Disclaimers, refer to www.uspto.gov/patents/process/file/efs/guidance/eTD-info-I.jsp.

Claims 1-15 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of U.S. Patent No. 10,543,164. Although the claims at issue are not identical, they are not patentably distinct from each other because the pending claims are a narrower scope of the patented claims. The patented claims fully encompass the pending claims.

Claims 1-15 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-19 of U.S. Patent No. Allowed Application 16/715,542(not yet assigned). Although the claims at issue are not identical, they are not patentably distinct from each other because the pending claims are a narrower scope of the patented claims. The patented claims fully encompass the pending claims.

Examiner's Note

The closest prior art is that of Lee et al. (US 2002/0086039). Lee discloses cosmetic compositions, for example antiperspirant and deodorant products that can be in the form of a stick. Lee additionally discloses components such as PPG-14 butyl ether, baking soda, dimethicone, isopropyl myristate, mineral oil, octyldodecanol and stearyl alcohol can be included in said sticks. Lee, however, discloses active ingredients such as aluminum zirconium tetrachlorohydrex gly or aluminum chlorohyrate are included.

Additional prior art includes Oblong et al. (US 2007/0203240). Oblong discloses personal care compositions comprising niacinamide, stearyl alcohol, dimethicone, and petrolatum. However, the composition is a moisturizing skin cream/lotion. Examples of solid antiperspirants comprise aluminum zirconium tetrachlorohydrex glycinate.

Scavone (US 6,485,717) and (US 7,033,579) discloses anhydrous antiperspirant and deodorant composition comprising stearyl alcohol, niacinamide, and dimethicone, however, Scavone also requires the use of aluminum zirconium tetrachlorohydrex glycinate.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to MELISSA S MERCIER whose telephone number is (571)272-9039. The examiner can normally be reached on M-F 5:30 am to 4 pm EST.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Robert Wax can be reached on 571-272-0623. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to

the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/MELISSA S MERCIER/
Examiner, Art Unit 1615

Notice of References Cited	Application/Control No. 16/716,542	Applicant(s)/Patent Under Reexamination Sturgis et al.	
	Examiner MELISSA S MERCIER	Art Unit 1615	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	CPC Classification	US Classification
*	A	US-20020086039-A1	07-2002	Lee, Sean	C03C3/097	424/401
*	B	US-20070203240-A1	08-2007	Oblong; John Erich	A61K8/43	514/561
*	C	US-6485717-B1	11-2002	Scavone; Timothy Alan	A61K8/11	424/400
*	D	US-7033579-B1	04-2006	Scavone; Timothy Alan	A61K8/02	424/400
*	E	US-10543164-B2	01-2020	Sturgis; David Arthur	A61K8/0241	1/1
	F					
	G					
	H					
	I					
	J					
	K					
	L					
	M					

FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	CPC Classification
	N					
	O					
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	
	V	
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	16/716,542	Sturgis et al.
	Examiner	Art Unit
	MELISSA S MERCIER	1615

CPC - Searched*		
Symbol	Date	Examiner
no classified search performed	07/31/2020	MMercier

CPC Combination Sets - Searched*		
Symbol	Date	Examiner

US Classification - Searched*			
Class	Subclass	Date	Examiner

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

Search Notes		
Search Notes	Date	Examiner
east- see attached	07/31/2020	MMercier
google-natural deodorant	07/31/2020	MMercier
google-deodorant SAME triglyceride SAME antimicrobial	07/31/2020	MMercier
palm-inventor search	07/31/2020	MMercier

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner

/MELISSA S MERCIER/ Examiner, Art Unit 1615	

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	David Arthur Sturgis
Art Unit	
Examiner Name	
Attorney Docket Number	15149C-US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4049792		1977-09-20	Elsnau	
	2	5019375		1991-05-28	Tanner , et al.	
	3	5429816		1995-07-04	Hofrichter , et al.	
	4	5891424		1999-04-06	Bretzler, deceased , et al.	
	5	5976514		1999-11-02	Guskey , et al.	

If you wish to add additional U.S. Patent citation information please click the Add button. Add

U.S.PATENT APPLICATION PUBLICATIONS Remove

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	David Arthur Sturgis
Art Unit	
Examiner Name	
Attorney Docket Number	15149C-US

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							

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NON-PATENT LITERATURE DOCUMENTS Remove

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1		

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Examiner Signature	/MELISSA S MERCIER/	Date Considered	07/30/2020

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[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	
Examiner Name	
Attorney Docket Number	–15149C-US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	5744146	A	1998-04-28	Peters, Kimberly T	
	2	9517193	B2	2016-12-13	Fares, Hani M.	Equiv. for WO 2013/052454 A1
	3	6485717	B1	2002-11-26	Scavone, et al.	
	4	7033579	B1	2006-04-25	Scavone	

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Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20170252288	A1	2017-09-07	Lesniak, Ewelina	Equiv. for WO 2016/048425 A1
	2	20020086039	A1	2002-07-04	Lee, et al.	

/MELISSA S MERCIER/ 07/30/2020

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

	3	20070203240	A1	2007-08-30	Oblong, et al.	
	4	20190000747	A1	2019-01-03	Sturgis, et al.	

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Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							

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	1	International Search Report; International Application No. PCT/US2019/022660; date of mailing 4 June 2019; 14 pages.	

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ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

Bibliographic Data

Application No: 16/716,542

Foreign Priority claimed: ○ Yes ◉ No

35 USC 119 (a-d) conditions met: ☐ Yes ☑ No ☐ Met After Allowance

Verified and Acknowledged: /MELISSA S MERCIER/ Examiner's Signature | Initials

Title: DEODORANT COMPOSITIONS

FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.
12/17/2019	424	1615	15149C
RULE			

APPLICANTS

The Procter & Gamble Company, Cincinnati, OH,

INVENTORS

David Arthur Sturgis, Montgomery, OH, UNITED STATES

Lindsey Michelle Britt, West Chester, OH, UNITED STATES

CONTINUING DATA

This application is a CON of 16021965 06/28/2018 PAT 10543164

16021965 has PRO of 62647104 03/23/2018

16021965 has PRO of 62527180 06/30/2017

FOREIGN APPLICATIONS

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UNITED STATES

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THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202
UNITED STATES

FILING FEE RECEIVED

$1,720

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	373	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME (stick solid)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/07/30 16:49
S1	194	"4049792"	USPAT	OR	OFF	2019/05/30 06:43
S2	117	"ASTM D-1321"	USPAT	OR	OFF	2019/05/30 06:47
S3	117	"ASTM D-1321"	USPAT	OR	ON	2019/05/30 06:47
S4	12	"ASTM D-1321" AND antimicrobial	USPAT	OR	ON	2019/05/30 06:50
S5	33	"ASTM D-1321" AND antimicrobial	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 06:50
S6	10	"ASTM D-1321" AND deodorant	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:23

EAST Search History (Prior Art)

S7	122	"ASTM D-1321" AND hardness	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:25
S8	82	"ASTM D-1321" SAME hardness	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:26
S9	467	"3792068"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:26
S10	174	"3792068"	USPAT	OR	ON	2019/05/30 08:27
S11	162	"3887692"	USPAT	OR	ON	2019/05/30 08:27
S12	167	"3904741"	USPAT	OR	ON	2019/05/30 08:28
S13	187	"5019375"	USPAT	OR	ON	2019/05/30 08:29
S14	17	"529806"	USPAT	OR	ON	2019/05/30 08:30
S15	144	"5429816"	USPAT	OR	ON	2019/05/30 08:30
S16	54	"5891424"	USPAT	OR	ON	2019/05/30 08:32
S17	65	"5976514"	USPAT	OR	ON	2019/05/30 08:33
S18	18	"4906454"	USPAT	OR	ON	2019/05/30 08:34
S19	41	"5972319"	USPAT	OR	ON	2019/05/30 08:35
S20	20	"6177066"	USPAT	OR	ON	2019/05/30 08:35
S21	1	"8460720"	USPAT	OR	ON	2019/05/30 08:42
S22	1	"8574559"	USPAT	OR	ON	2019/05/30 08:44
S23	6	"8672067"	USPAT	OR	ON	2019/05/30 08:45
S24	1	"9468596"	USPAT	OR	ON	2019/05/30 08:50
S25	3	"9949920"	USPAT	OR	ON	2019/05/30 09:04
S26	0	"20030235546"	USPAT	OR	ON	2019/05/30 09:04
S27	1	"20030235546"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:05
S28	1	"20050281767"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:05
S29	1	"20110076309"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:06

EAST Search History (Prior Art)

S30	1	"20110196786"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:06
S31	1	"20160235661"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:11
S32	1	"20190000730"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:12
S33	978,061	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone)	US-PGPUB; USPAT	OR	ON	2019/05/30 09:18
S34	220,470	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:06
S35	224,991	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss)	US-PGPUB; USPAT	OR	ON	2019/05/30 12:10

EAST Search History (Prior Art)

S36	509,700	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:15
S37	215,206	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") AND ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:55

EAST Search History (Prior Art)

S38	47,436	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") AND ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND (stick hardness)	US-PGPUB; USPAT	OR	ON	2019/05/30 13:18

EAST Search History (Prior Art)

S39	38,478	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	US-PGPUB; USPAT	OR	ON	2019/05/30 13:19

EAST Search History (Prior Art)

S40	15,506	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	USPAT	OR	ON	2019/05/30 13:20

EAST Search History (Prior Art)

S41	1,327	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND stick	USPAT	OR	ON	2019/05/30 13:29

EAST Search History (Prior Art)

S42	198	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide") AND stick	USPAT	OR	ON	2019/05/30 13:31

EAST Search History (Prior Art)

S43	438	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant	USPAT	OR	ON	2019/05/30 13:32

EAST Search History (Prior Art)

S44	127	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	USPAT	OR	ON	2019/05/30 13:33

EAST Search History (Prior Art)

S45	399	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 14:20
S46	218	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/13 15:50

EAST Search History (Prior Art)

S47	218	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/14 07:07
S48	5	"4005186"	US-PGPUB; USPAT	OR	ON	2019/06/17 14:23
S49	5	"6485717"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/17 19:39
S50	17	"5744146"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:47
S51	1	"9517193"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:55
S52	1	"20170252288"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:57
S53	6	"6231842"	US-PGPUB; USPAT	OR	ON	2020/07/28 13:38
S54	3	"10543164"	US-PGPUB; USPAT	OR	ON	2020/07/28 13:42

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	
Examiner Name	
Attorney Docket Number	–15149C-US

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Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4906454		1990-03-06	Melanson, Jr.	
	2	5972319		1999-10-26	Linn , et al.	
	3	6177066	B1	2001-01-23	Pataut	
	4	6503944		2003-01-07	Chanchani	
	5	7425321	B2	2008-09-16	Lemoine	
	6	8460720	B2	2013-06-11	Tom's of Maine, Inc.	
	7	8574559	B2	2013-11-05	Banowski	
	8	8672067	B2	2014-03-18	Toyota Jidosha Kabushiki Kaisha	

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

	9	9468596	B2	2016-10-18	Eizen	
	10	9949920	B2	2018-04-24	Hakim Noha N.	

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	1	20030235546	A1	2003-12-25	Mattai, Jairajh ; et al.	
	2	20050281767	A1	2005-12-22	Walling et al.	
	3	20110076309	A1	2011-03-31	MISNER H STEVEN; FAN AIXING; KILPATRICK-LIVERMAN L	
	4	20110196786	A1	2017-07-13	Avon Products, Inc.	
	5	20160235661	A1	2016-08-18	AXIM Biotechnologies, Inc.	
	6	20190000730	A1	2019-01-03	DEAD SEA BROMINE COMPANY LTD.	

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	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							

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Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1	International Journal Cosmetic Science, 2003, 25, pp. 127-135 - Iron Sequestration on Skin: A New Route to Improved Deodorancy	
	2	Nivea Fresh Natural Deodorant, pulled from the Internet (www.GNPD.com) 3 pages	

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Examiner Signature	/MELISSA S MERCIER/	Date Considered	07/30/2020

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[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99**)	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature		Date (YYYY-MM-DD)	
Name/Print		Registration Number	

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

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ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	
Examiner Name	
Attorney Docket Number	–15149C-US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4906454		1990-03-06	Melanson, Jr.	
	2	5972319		1999-10-26	Linn , et al.	
	3	6177066	B1	2001-01-23	Pataut	
	4	6503944		2003-01-07	Chanchani	
	5	7425321	B2	2008-09-16	Lemoine	
	6	8460720	B2	2013-06-11	Tom's of Maine, Inc.	
	7	8574559	B2	2013-11-05	Banowski	
	8	8672067	B2	2014-03-18	Toyota Jidosha Kabushiki Kaisha	

/MELISSA S MERCIER/ 07/30/2020

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

	9	9468596	B2	2016-10-18	Eizen	
	10	9949920	B2	2018-04-24	Hakim Noha N.	

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U.S.PATENT APPLICATION PUBLICATIONS Remove

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20030235546	A1	2003-12-25	Mattai, Jairajh ; et al.	
	2	20050281767	A1	2005-12-22	Walling et al.	
	3	20110076309	A1	2011-03-31	MISNER H STEVEN; FAN AIXING; KILPATRICK-LIVERMAN L	
	4	20110196786	A1	2017-07-13	Avon Products, Inc.	
	5	20160235661	A1	2016-08-18	AXIM Biotechnologies, Inc.	
	6	20190000730	A1	2019-01-03	DEAD SEA BROMINE COMPANY LTD.	

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99**)	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							

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NON-PATENT LITERATURE DOCUMENTS Remove

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]
	1	International Journal Cosmetic Science, 2003, 25, pp. 127-135 - Iron Sequestration on Skin: A New Route to Improved Deodorancy	
	2	Nivea Fresh Natural Deodorant, pulled from the Internet (www.GNPD.com) 3 pages	

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Examiner Signature	/MELISSA S MERCIER/	Date Considered	07/30/2020

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99)**	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

Signature		Date (YYYY-MM-DD)	
Name/Print		Registration Number	

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

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PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	16716542
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	1615
Examiner Name	Melissa S Mercier
Attorney Docket Number	--15149C-US

U.S.PATENTS

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	3792068	A	1974-02-12	Luedders, W	
	2	3887692	A	1975-06-03	Gilman, William S	
	3	3904741	A	1975-09-09	Jones, John L	
	4	4120948	A	1978-10-17	Shelton, David Lee	
	5	4359456	A	1982-11-16	Gosling, Keith	
	6	5298640	A	1994-03-29	Callaghan, David T	
	7	5675013	A	1997-10-07	Hani, Rahim	
	8	6624126	B1	2003-09-23	Kasuga et al.	

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	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	9	7033576	B2	2006-04-25	Chevallier, Yvonick	
	10	10555884	B2	2020-02-11	Sturgis, David Arthur	

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U.S.PATENT APPLICATION PUBLICATIONS

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20010046479	A1	2001-11-29	Landa, Andrew Sjaak	
	2	20070003499	A1	2007-01-04	Shen, Yanfei	
	3	20130045907	A1	2013-02-21	Lanzalaco, Anthony Charles	
	4	20130045910	A1	2013-02-21	Miracle, Gregory Scot	
	5	20140154189	A1	2014-06-05	Polson et al.	
	6	20150196477	A1	2015-07-16	Stark, Cynthia Marie	
	7	20160074300	A1	2016-03-17	Salvador, Charlie Reyes	

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	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	1615
	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	8	20160326091	A1	2016-11-10	Rudolph, Thomas	
	9	20170172873	A1	2017-06-22	Banowski, Bernhard	
	10	20190000736	A1	2019-01-03	Sturgis, David Arthur	
	11	20190276389	A1	2019-09-12	Wos, John August	Equiv. for WO 2017/106279 A1
	12	20200000694	A9	2020-01-02	Sturgis, David Arthur	
	13	20200214957	A1	2020-07-09	Sturgis, David Arthur	

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FOREIGN PATENT DOCUMENTS

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	825146	BE	A1	1975-08-04	Procter & Gamble		☒
	2	1347950	GB	A	1985-03-20	Bristol-Myers		☐
	3	2048229	GB	A	1980-12-10	Gillette Co		☐

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	Art Unit	1615
	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	4	2144992	GB	A	1985-03-20	Gillette Co		☐
	5	2008110999	JP	A	2008-05-15	Mandom Corp		☒
	6	2004089092	WO	A1	2004-10-21	Idemitsu Technofine Co Ltd		☒
	7	2014139449	WO	A1	2014-09-18	Procter & Gamble		☐

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NON-PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T5
	1	All Office Actions, U.S. Patent Appl. No. 16/021,877 (14861M), SEE PAIR	☐
	2	All Office Actions, U.S. Patent Appl. No. 16/021,931 (15150), SEE PAIR	☐
	3	All Office Actions, U.S. Patent Appl. No. 16/021,965 (15149), SEE PAIR	☐
	4	All Office Actions, U.S. Patent Appl. No. 16/716,540 (14861MC), SEE PAIR	☐
	5	International Search Report; International Application No. PCT/US2018/039972; date of mailing 25 March 2019; 10 pages.	☐

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	Attorney Docket Number	--15149C-US

	6	Kontoghiorghes, George J., "2-Hydroxypyridine-N -Oxides: Effective New Chelators in Iron Mobilisation", Biochimica et Biophysica Acta, Vol. 924, No. 1, April 16, 1987, pp. 13-18.	☐

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Zirconium or hafnium oxyhalide anti-perspirant cpds - used in compsns with aluminium cpds and amino acids

AB

(FR2259587)
Anti-perspirant cpds. (I) of formula MO(OH2)2-aXa.nH2O (where M is Zr or Hf; X is Cl, Br or I; a = 1.5-1.87 and n = 1-7) of mol. wt. pref. 300-25,000 partic. 30-1500, Also compositions of the above are used (0.15-1.5 pts) in compsns. contg. a cpd. (Al2(OH)6-mXm n1H2O (where m = 0.8-2; and n1 = 0-12) (1 pts) and an aminocarboxylic acid with an equal number of carboxy and gps. (0.06-0.6 pts) pref. alanine, serine, 2-aminobutyric acid, valine, beta-(phenyl)alanine, methionine, tryptophane and esp. glycerine (I) are prepd. by dissolving a Zr or Hf salt, pptg. with a strong base, washing and dissolving in a haloacid; the compsn. is prepd. by dissolving the components and concentrating to 1-30%. Also an aerosol compsn. contg. 1-12% of the above anti-perspirant compsn. 0.1-5% suspension agent, 1-15% liq. vehicle and 70.7-93.9% propellant. Prods. are more effective, and the Zr is non-irritant to skin or damaging to clothing.

List of publications

	Publication number	Publ. date	Appl. Number	Appl. Date	Document type
	BE825146	1975-08-04	1975BE-0153024	1975-02-04	A - Patent
	NL7501279	1975-08-06	1975NL-0001279	1975-02-04	A - Patent application laid open
	FR2259587	1975-08-29	1975FR-0003259	1975-02-03	A1 - Application for patent of invention, (first publ.)
	DE2504381	1975-12-18	1975DE-2504381	1975-02-03	A1 - Doc. laid open (First publication)
	FR2259587	1978-07-13	1975FR-0003259	1975-02-03	B1 - Patent of invention (second publication)

IN SCHMITZ JEROME JOSEPH

PA PROCTER & GAMBLE*

PAH (DE2504381)
THE PROCTER & GAMBLE CO.

(FR2259587)
PROCTER AND GAMBLE CY,US
PROCTER AND GAMBLE CY

(NL7501279)
THE PROCTER & GAMBLE COMPANY TE CINCINNATI, VER.ST.V.AM.

RP (FR2259587)
CABINET HARLE ET LECHOPIEZ

PR 1974US-05438976 1974-02-04

TECD Materials, metallurgy
Pharmaceuticals

IC	A61K* A61K-033/24 C01G-025/04 C01G-027/04 C04B-035/50
CPC	A61K-008/19 A61K-008/26 A61K-008/28 A61K-008/44 A61Q-015/00*
EC	A61K-008/19 A61K-008/26 A61K-008/28 A61K-008/44 A61Q-015/00

CLMS

(FR2259587)

CLAIMS

1. Composed of antitranspiration, characterized in that he answers the formula empiriqueMO (OH) 2 centered, nH20 in which M is a metal chosen among zirconium eti' hafnium, X is selected among chlorine, bromine and iodine, has is a number from approximately 1,5 to 1,87 and N is a number from approximately 1 to 7.

2. Composed according to claim 1, characterized in that its average molecular weight is approximately 300 to 25.000 and in what M represents zirconium and X represent chlorine.

3. Composed according to one of the claims 1 or 2, characterized in that the average molecular weight of the compound is approximately 300 to 1500.

4. Complexed' antitranspiration, characterized in that it includes/understands: (A) 0,15 to 1,5 partied' a composerepondan-t with the formula empiriqueMO (OH) 2 centered, nH20 such as defined in claim 1; (B) 1 part of a basic aluminum compound of formula empirical A12 (0H) 6 m, nH20 in which X is selected among lesore, bromine and iodine, m has a median value of environ0,8 with 2,0 etn has a value of environO with 12; and (C) O, o6 a0,60 started from an amino acid containing an equal number of amino groups and carboxyl groups.

5. Complex according to claim 4, characterized in that the compound (A) has an average molecular weight from approximately 300 to 25.000 M represents zirconium and X chlorine; in what the basic aluminum compound is the aluminum chlorohydroxyde; and in what the amino acid is selected among the following compounds: glycine, alanine, alanine, serine, acide2-aminobutyric, valin, p-phe- nylalanine, methionine and tryptophan.

6. Complex according to one from the revendications4 or 5, characterized in that the average molecular weight of the element (A) is of environ300 at 1500.

7. Complex according to claim 6, characterized in that the amino acid is the glycine.

8. Proceeded for obtaining an astringent compound, characterized in that it consists: (A) to dissolve in water a water-soluble salt of a metal chosen among zirconium and hafnium; (b) to add to the solution obtained in (A) a strong base in sufficient quantity to form a

precipitate; (c) to wash the aforementioned precipitate with del' water; (d) to dissolve the precipitate in a solution of an acid chosen among the hydrobromic acids, iodhy doric and hydrochloric, the aforementioned acid solution containing approximately 1,509 1,87 mole of acid per mole of zirconium in the precipitate, the temperature during the aforementioned process being maintained at a value below de100 OC approximately.

9. Proceeded according to claim 8, characterized in that water-soluble metal salt is a water-soluble salt of zirco nium and the aforementioned acid is the hydrochloric acid.

10. Proceeded according to one from the claims 8 or 9, caracte rized in what water-soluble metal salt is 1 ' zirconium oxychloride, the hydroxychlorure of zirconyle, the zirconium tetrachloride, the bromide of zirconyle, the iodide of zirconyle, the sulfate fate of zirconium or the zirconium nitrate.

11. Complex-of powder antitranspiration, characterized in that it is obtained by the process which consists in (A) dissolving together in water (A) 1 part of-compound Al2 (OH) 6-mXm in which X and m are such as defined in claim 4, (b) approximately 0,15 to 1,5 part of a compound such as defined in claim 1, (C) - approximately 0,06 to 0,60 part of a selected neutral amino acid among the following compounds: gly movies, dl-tryptophan, dl-p - phenylalanine, di-valin, dl-serine, dl-methionine, dl-alanine, p - alanine and acid 2-aminobutyric; and (R) to dry the solution until a moisture content from approximately 1 to 30% in weight.

12. Complex powders some according to the revendication11, characterized of what one prepares it by a process according to which one chooses the compound MO (OH) 2-centered, nH2O so that M is zirconium, X is chlorine and that the average poidsmoleculaire of made up the soitd' approximately 300 a25.000, the amino acid being the glycine.

13. Complex according to larevenditation 12, characterized in that the aforementioned average molecular weight lies between approximately 300et 1500.

14. Composition of powder antitranspiration for aerosol, characterized in that it includes/understands (A) approximately 1 to 12% of a COM plexed' antitranspiration such as defined in the revendicationll; (B) approximately 0,1 to 5% in weight of an agent of suspension; (C) envi r6n I-at 15% in weight of a liquid vehicle and (D) environ70,7 to 93,9% in pcfide a liquefiable anhydrous gas engine.

15. Composition for aerosol according to the claim 14, Ca racterized in what metal M is zirconium, X is chlorine and the average molecular weight of the compound is approximately 300 a25. QQO

DESC

The present invention relates to new useful compounds to prepare anti-perspiring or antiperspirant active agents, the terms antiperspirant and anti-perspiring having the mAeme significance within the meaning of the present invention; the invention also has as an aim a antitranspirant process for obtaining complex an active of a remarkable effectiveness, quel' one can incorporate in antitranspirantes compositions or compositions of antitranspiration, compositions which are neither irritating for the skin nor corrosive for the textiles.

Many chemical compounds capablesd' to prevent or to delay the exudation of perspiration already were used or proposed for use in the compositionsd' antit-ranspi-ration. At present, one very usually uses the selsd' aluminum of the strong acids, especially the chlorides. Aluminum chlorohydroxyde, whose empirical formula estA12 (0H) 5G1,2h0 constitutes the product generally selected by the manufacturers of the agents of antitranspiration. Recently, one suggested the use of various derivatives of zirconium alone or in combination with aluminum salts in the products of antitranspiration. The zirconium salts used before in the products of antitranspiration etaienti' oxychloride of zirconium (formula empiriqueZrOC12,8 NR 0) or hydroxychlorure of zirconyle (formula empiriqueZrO (OH) Cl,)) O).

Although one proposed various derivatives of zirconium usable in the compositions of antitranspiration, more efficacessont salts of the strong acids, which form solutions of a so intense acidity, that they deviennentextremement irrivantes for the skin and also damage lesvAtements. One can use zirconium oxychloride in the condition however of combining it with a complex of chlorohydroxydedlalumi- nium and one - neutral amino acid (see patent U. S. 2.814.585). The complex of aluminum chlorohydroxyde and the amino acid play the part of agents of plugging or agents plugs.

It was unfortunately noted that the zirconium oxychloride presents some inconvenientsmeme when one uses it within a composition of plugged antitranspiration in a suitable way. It is difficult to dry the mixtures described above to obtain a dry powder because of the presence of the 8:2 O dansltoxychlorure of zirconium (water of hydration). Deme- me, when a dry powder is obtained, the latter estextrAeme- lies hygroscopic. Although the addition of a more neutral amino acid makes it possible to reduce the hygroscopic character, the quantity of amino acid necessary to obtain an adapted composition, ctest-with-to say of which the use is without danger, quintendom- magus not clothing and who is compatible with the bottles of conditioning and the perfumes, is so large that it results from it a strong increase in price and a fall from effectiveness.

In addition, the zirconium oxychloride undergoes in an aqueous solution of the long and complicated reactions of hydrolysis.

It is thus difficult to maintain a pH stable in solutionaqueuse of oxychloride of zirconium before drying and, in addition, the pH of a batch can be different from celuidalot

according to. I1 results from it from the products which are different in an inherent way after incorporation in final compositions.

The hydroxychlorure of zirconyle does not present the same disadvantages as zirconium oxychloride. I1 is less acid, therefore less irritant and contains more a small number of groups of H20 hydration, so that it is less hygroscopic. However, the hydroxychlorure of-zirconyle is notably less effective than zirconium oxychloride like agent of antitranspiration and the considerations of safety require despite everything the use of certain agent-plugs, such as chlorohydroxyde of aluminum and a neutral amino acid (see for this purpose the US patent 2.854.382).

The solutionsd' hydroxychlorure of zirconyle, of chlorohydroxyde of aluminum and amino acid produce a poudrepre6- that white with drying. Unfortunately, the produitd' antitrans- piration thus obtained is less effective than the combination oxychloride dezirconium/chiorohydroxyde of aluminium/amino acid, although more effective than aluminum chlorohydroxyde alone.

Tests with a certain number of less acid zirconium salts, for example with carbonate, the lactate, the glycolate, or the zirconium acetate or the trioxychlorure dedizrco- nium, showed that all these products are less effective quel' hydroxychlorure of zirconyle with regard to the antitranspirant capacity.

Present invention concerns new compound of zirconium which is highly effective like agentd' antiranspira- tion and qu one can to incorporate in compositions sold with users which is likely to not irritate the skin, not to damage clothing and pascerroder containers of idiot ditionnement present invention has also for object complex pulverulent homogeneous of antitranspiration, which has a great effectiveness to prevent or delay the exudation of the transpi ration and that one can incorporate in compositions sold to the users, who are likely to not irritate the skin, not to damage clothing and not to corrode the containers of conditioning.

Another object of the invention is consisted a highly effective compound of antitranspiration, which one can use faci lement to prepare of the products of antitranspiration whose USA Ge is without danger and whose characteristics are attractive for the users, these products being able to be/liquid, of the lotions, the creams or the aerosols such as aerosols in dry louse dres.

Other objects of the invention will bring out delta descrip tion hereafter, in which all the parts, the percentages and the reports/ratios are in weight except contrary stipulation.

The present invention is founded on the discovery of nou made up calves answering the empirical formula: MO (OH) 2 centered, nH20 in which M represents zirconium or hafnium, X represents chlorine, bromine. or iodizes it, has is unnombre of environ1 > 50 at approximately 1,87 and N is a number from 1 to approximately 7. It is conceived that the number of H20 groups of hydration has importance only when the compound is dried.

Cepenayant, generally one does not dry the compound but one it utili in the form of a solution in the later stages of treatment.

One usefully uses the new compounds according to the invention in combination with an astringent aluminum salt and a neutral friend noacide in the capacity as composed of antitranspiration which one can incorporate in products of antitranspiration containing of other traditional ingredients.

One noted that the objects of the invention can be realized sil' one uses an empirical compound of formula MO (OH) 2 centered, nH20 in which M accounts for zirconium or 1 ' hafnium, X representel chlorine, bromine or iodine, has is a number of 1,50 al, approximately 87 etn is a del number to approximately 7.

The compounds according to the invention can present very varied molecular weights. One prefers however that the average molecular weight lies between approximately 300 and 25.000 and, better still, between approximately 300 and 1500.

A general-purpose element, such as zirconium, estes has to form polymeric compounds. In fact, the zirconium compounds are in general polymeric in the aqueous solutions except with a very strong acidity. The molecular weights of zirconium polymers depend on the processes used in breadth treatment and also on the purity of the raw materials being used to prepare the polymeric compounds. For example, a strongly acid zirconium oxychloride is a polymer of a rather low molecular weight.

One can neutralize it in aqueous solutions to obtain a polymer having a molecular weight more raised.

It is thought that the process pour10 obtaining composed according to the invention is critical for the manufacture of the recommended products. One noted that to prepare the compounds containing of-zirconium according to the invention, one can use the following process which consists in (A) dissolving in water a water-soluble zirconium salt, for example the zirconium oxychloride; (B) to add a strong base slowly, such as NaOH, KOH, ouNH4OH with the solution of (A) until the formation of a hydrated zirconia precipitate; (C) to insulate the precipitate by adapted means, for example by filtration; (D) to wash the aforementioned precipitate with water; and (E) to dissolve the precipitate in a hydrochloric acid solution, this solution containing 1,50 a1,87 mole of HC1 per mole of zirconium in the precipitate.

During the process, one must advantageously maintain the temperature below de10000 and, better still, between 20 et1000C.

The composede - zirconium thus obtained is a very effective agent of antitranspiration but it is not also hygroscopic or as irritating for the human skin as 1 ' zirconium oxychloride.

Like water-soluble examples of zirconium salts usable in the stage (A), one can quote sulfate, nitrate, and zirconium halides for example the oxychloride of zirco nium, the hydroxychlorure, the chloride, the bromide etl' iodized zirconyle and the zirconium tetrachloride.

One can obviously use in the place of made up tells nant zirconium and chlorine, respectively, equivalent des2roportions of hafnium and another halide (such as bromine ou1' iodine), However, the preferred compounds are those containing zirconium and of chlorine.

One noted that the effectiveness of a complex aluminum zirconium/amino acid is strongly increased, without aueun harmful side-effect, for example without increase in effect irritating, when one uses the compounds according to the invention.

Thus, the complexes of antitranspiration which are highly effective and not irritants include/understand: (A) 0,15 to 1,5 part of a new compound according to the invention; (B) 1 part of a basic aluminum compound of formula empiri that: Al2 (OH) 6-mXm, nH2OJ in which X is chlorine, bromine or iodine, m has a median value from approximately 0,8 to 2,0 and N is a number of O with approximately 12; and (C) 0,06 has 0,60 part of a neutral amino acid, as described further.

It was noted that the empirical aluminum halogenohydroxydes of formula Al2 (OH) 6-mXm, nH2O, in which X is chlorine, bromine or iodine, m has a value moyeane approximately 0,8 to 1,5 and N of approximately O with 12, are facilementapplicahles for purposes de1 T invention although one can use a basic unspecified aluminum compound within the limits defined above and ob to hold of the satisfactory results. It will be noticed that these composes~asiq7ues aluminum possedenteux-same of the properties of anti perspiration.

Like basic examples of aluminum compounds, one will quote aluminum lechlorhydroxyde, the hydroxybromure D "aluminum and the basic aluminum chloride hydrated with 75% of formula Al2 (OH) 4,5Cl1,5. One prefers in particular chlorohydroxyde of aluminum minium of formula empiriqueAl2 (OH) 5cl, 2H0. The aluminum chlorohydroxyde is a well-known salt which is effective against the trans piration and which is available in the trade (sold for example by Reheis Chemical Company).

The amino acids which are appropriate for purposes of the invention are those in which the number of the amino groups is equal to that of the carboxyl groups in the molecule. With an aim decommoditeces amino acids are appeles1, amino acids neutral in the descriptive presentmdmoire. Although one can use an unspecified neutral amino acid of the typederini to lead above to the objectives of the invention, one one prefers the neutral amino acids which are solu- coms in aqueous solutions, for example the glycine, alanine, laP - analine, serine, acid 2-aminobutyric, valin, lal) - phenylalanine, methionine or tryptophan. One can use form 1, the form D or the shape dl of these compounds.

The especially preferred amino-acid is the glycine.

One adds neutral amino acid during the preparation of the complexes according to the invention in a proportion allowing to prevent the premature gelation of the complex in solution. Silton uses an insufficient quantity of amino acid, the solution of the salt of zirconium or of hafnium plus the aluminum compound a gelation at the end of very little time will undergo. If, in addition, one uses an excessive proportion of amino acid, one observes a baissed' effectiveness, and certain zirconium salt combinations/aluminum salts end up gelling. It is understood that in all the cases, it is advisable to incorporate amino acid in a pourenpecher sufficient quantity the gelation of the active ingredient of antitranspiration or composition containing this active ingredient. This consideration is extremely important because the gel has so limited properties of antitranspiration that their use is regarded as practically impossible in this field.

In the preparation of the end products usable by the consumers, starting from the complexes according to the invention, the concentration of the complex can be approximately 1 with 30ffi in weight compared to the total composition.

Patent U. S. 3.792.068 described of the complexesdtantitrans piration out of powder, the methods of preparation of these complexes and the products usable by the consumers containing such complexes. The lesson of this patent is expressly built-in as reference in the present request. One can replace the zirconium salts described in the above mentioned patent by the new hafnium and zirconium compounds according to the present invention, to obtain complexes of high antitranspiration lies effective and that one can use to prepare composi tions of antitranapiration of a use without danger-and of an aspect cosmetically attractive for the sale with the consumers.

Thus, to form a complex of antitranspiration in louse DRE having a great effectiveness, one procedecomme follows: one jointly dries an aluminum subsalt, a composedthafnium or of zirconium according to the invention and a neutral amino acid of the type defined above, these components beforehand having been me langes together in an aqueous solution; one dries the complex until a final content of moisture of approximately I with 50% in weight, of preferenceenviron 5 to 20%. The pulverulent complex thus formed has unexpected higher properties than one ob does not hold silion dries the components separately and than then one combines them by a simple process of mechanical mixture.

To prepare a pulverulent complex of antitranspiration one can as follows operate (A) one dissolves together in water (1) 1 part dcA12 (0H) 6 mXm, formula in which X is an ion halide chosen among the ions chloride, bromide, or iodizes and N is a number from approximately 0,8 to 2,0; (2) environ0,15 with 1,5 part of the new compound according to invention; and (3) approximately 0,06 to 0,60 part of a selected neutral amino acid among the glycine, dl-tryptophan, ladl- - phenyl-ana line, dl-valin, it - dl-serine, dl-methionine, the dl alanine, laP - alanine or thebutyric one; (B) one dries this solution until a moisture content of envi ron I to 30 ffi in weight and, possibly (C) one crushes the

complex dried to form an impalpable uniform powder before incorporating the complex in a compositionsd' aerosol.

RN what relates to the process of drying, one can use a sechageordinaire out of drying oven, a drying out of drum, a drying by pulverization, a drying by the vacuum, a freeze-drying, a drying with the microwaves, etc. Although the choice of the process of drying is not critical, one prefers drying by pulverized tion. During drying, one chauffepastde preference, the COM plexe antitranspirant above de270 " C in consequence of the risk a decomposition amino acid and aluminum or zirconium oxide formation insolu undesirable corns at a more high temperature. In the operation of drying per pulverization, the temperature of the produitest senstihlement lamAme that of the air at the removal from the enclosure of drying and not that of the air al' in tree of this enclosure. Thus, whereas the temperature of the ingoing air can be higher than 2300C, it is preferred that the outgoing air is at a temperature below de2300C.

Among the adapted processes of crushing, one will mention crushing with the balls, crushing with the rollers, the passage through a broyeurcolloidal, crushing with energy of fluid by impact etc. According to the nature of the equipment of crushing, it can be or pasatre necessary to make pass the powder crushed through a sieve to ensure the uniformity and to eliminate the too large granules from it.

If one must incorporate the powder in a packing or container for aerosol, the granulometry of the dry pulverulent complex must be sufficiently low to allow a uniform pulverization without risk of an operating fault of the adjutage or valve. Although it is possible to use particles reaching 100 microns in diameter, preferred granulometry lies between approximately 5 and 40 microns.

It is preferred that the atomic report/ratio de1' abliminium with zirconium in the complex described is approximately 4,2:1 with 2,5:1.

The neutral amino acid must be present in a sufficient quantity to prevent the gelation or formation of freezing of the solutions of zirconium and aluminum salts. The amino acid neutreprefere D is the glycine that one advantageously incorporates in a molar report/ratio aluminum/glycine of approximately 1,75:1 a5,55: 1.

A pulverulent composition of antitranspiration in the form of an aerosol, containing the complex describes higher, can be prepared by combining the following ingredients (A) approximately 1 to 12% in poidsdtun complex of antitranspiration of the type describes above, (B) approximately 0,1 to 5% in weight of an agent of suspension; (C) approximately 1 to 15% in weight of a liquid vehicle; and (D) a sufficient quantity of a propellent anhydrous gas to produce a pulverization of the aerosol type.

Component has For purposes of the invention, the preferred compound of antitranspiration is a mineral/organic complex which was already described endetdl previously.

One incorporates approximately 1 to 12% in weight, preferably 2,5 with 6ffi approximately of this compound in a composition aerosol and one obtains an excellent effectiveness of antitranspiration. A lower proportion has approximately 1ss reduitl4efficacity antitranspiran- you. Above 12, ia composition is not economically profitable in so far as its effectiveness "does not increase proportionally with the incorporation of an additional quantity - and one encounters new problems of handling etd' atomisation.

Component B the agent of suspension maintains the complex pulverulent of antitranspiration in suspension in lacomposition, Although a certain deposit can avoirieu, the complex of antitranspiration remains in an easy state with redisperser. Among the adapted agents of suspension, one will preferably quote silica colloidale (of a granulometry below de0,05 micron diameter, for example the product known under the trade description " Cab-O-Sil M-5 " (produced sold by CabotCorp.), the products described in the patent application in Netherlands N 66/13943 published. - on April 4th, 1968 by Spitzer and submicroscopic particulate silica prepared in a hot gas environment (1100 C) by the hydrolysis of the higher phase of a silicon compound.

Other suitable agents of suspension are hydrophobic clays, which inflate in the organic solvents, for example the product known under the trade description " Bentone-38 (produced sold by NR. L. Industries) which is a clay montmorillonite modified by organic way, primary education aliphatic amines containing from 12 to 20 carbon atoms, parexemple laurylamine, latetradeeylamine, the hexedecylamine, the octadecylamine etl' eicosylamine; soaps forming of greases like aluminum stearate; smoked alumina; and of aliphatic monoalkylol-amides saturescomportant unechaise of carbon atoms of acids grasd' approximately 12 to 20 carbon atoms and a channealicylol of approximately with 3 carbon atoms, examples of these amides being copra-monoethanolamide, the octadecyl-monoethanolamide, COM describes me in the patent belgen0 760.957.

The agents of suspension which have just been described or their peuventetre mixtures used at a rate of approximately 0,1 to 5% in weight. One prefers a proportion from 0,3 to approximately 0,8%. The agents of suspension which one particularly prefers with fine the deltinvention are the argileshydrophobes which inflate in lessolvants organic, for examples the argilesconnues under the denomination commercialettSentone-38 ".

Component C One uses a liquid vehicle slightly volatile so that the emanating current of the container of aerosol is a wet pulverization and not a cloud of particles or dust. Indeed, one thus obtains a more pleasant feeling on the skin and one reduces the risks of an inspiration of the poudreseche liquid vehicle also improves the effectiveness because it pareffrittement maintains the composed1antitranspiration in contact with the skin under conditions not allowing the fall of made up or by washing. Among the suitable liquid vehicles, one will indicate carboxylic esters, such as the myristate or the palmitate of isopropyl; liquid hydrocarbons like lettradecane; fatty alcohols like alcohol laurylic, hexadecylic or oleylic; fatty-acids such as the lauryque or oleic acid; lanolin and its derivatives such as the acetylated lanolin; and silicone oils, such as the dimethylpolysiloxane. Others made up organics being able to be used as liquid vehicles

are those containing of the multiple esters groups, for example the organic diesters of diacids and made up containing multiple esters groups, for example the phthalate of di-N-butyl, the sebacate of diethyle, the adipate of diisopropyle etl' ethylcarbomethyl-phtalated" ethyl (ortho-CZH, OOC-C0OCH2COOC2H,).

Of autresvehicules - liquid suitable are the polyethyleneglycol monolaurate and ethoxyle or propoxyle butanol, for example the products known under the trade description " Ucon-HB-66O and Ucon HB-5100 (Union Carbide).

Among the liquid vehicles which were enumerated, one prefers the organic compounds containing of multiple esters. One preferetout especially the dedi-N-butyl phthalate, the sebacate dediethyle, the adipate of diisopropyle etl' ethylcarbomethyl- phthalate of ethyl.

Described liquid vehicles peuventAetre used at a rate of environ1 with 15ffi. Below approximately 1% the quantity is insufficient to obtain a wet pulverization and the latter is thus troppoussiereuse or granulous and biend the skin does not adhere. Above approximately 15%, the composition deposited on the skin gives a fatty and oily unpleasant feeling. One prefers a liquid proportion of vehicle of approximately 2-to 8%.

ComposantD the propellent gas used can be a liquefiable gas unspecified qu one usually implements in the aerosols Like examples of propellent gases usable, one will quote the trichlorofluoromethane, ledichlorodifluoromethane, ledichlorotetrafluoro ethane, the monochlorodifluoromethane, letrichlorotrifluoroethane, propane, butane etl' isobutane, only or in mixtures. One prefers especially letrichlorofluoromethane, the dichlorodifluoromdthane, the dichlorotetrafluoroethane etl' isobutane, only or in mixtures.

The proportion dugazFropulsor to be used is determined by lesfaeteursusuels well-known specialists in the aerosols.

I1 is satisfactory to regard propellent gas as formant the complement of the composition, it be-8-to say the quantity in addition to the other components studied in higher detail. Consequently, the preferred proportions of the engine are of environ70,7 a93,9 X, and particularly from 80 to approximately 92%.

I1 is obvious that other ingredients can be built-in minor amounts in the composition, without influencing the nature of the invention. An example of such an additional ingredient is a perfume in a proportion going up to approximately 0,8%.

The following examples are used to illustrate the invention without at all limiting the range of it.

EXAMPLE I One as follows prepares a compound of antitranspiration containing zirconium, according to 1 ' invention: one dissolves 32,2 zirconium gdloxychlorure,

ZrOCl2,8H2O, in water 32,2ml distilled under agitationet one adds slowly to the solution above, 20 G of a solution at 40% of sodium hydroxide.

Il is formed a precipitate which one separates from the solution by filtration, then one washes this precipitate which contains 9,12 G of zirconium with approximately 2 liters of water, one adds 17,3 G of a solution at hydrochloric acid 37% to this wet precipitate, one heats a60 C and one agitates until dissolution of the precipitate. One subjects then the solution to a vacuum evaporation to eliminate 12,5 parts of water and onobtienVainsi a solution at 40% from composed of desired zirconium.

This zirconium compound presents a report/ratio Cl: Zr de1,76.

and its empirical formula estZrO (OH) 0,24C11,76. The compound is not dried but one leaves it in the solution and for this reason one does not indicate the number of H20 groups of hydration.

EXAMPLE 2 to prepare a complex of powder antitranspiration, one proceeds as follows: one introduces into a becher 15 G of an aqueous solution with 50% of aluminum chlorohydroxyde ("Chlorhydrol" registered trademark of Reheis Chemical Company). One adds glycine at a rate of 2,0 G and one dissolves completely under agitation.

One adds ensuite13,6 G of the aqueous solution (to approximately 40%) of zirconium the desired compound of example 1, with continuous agitation, Jusqd with a codissolution of all the components. One dries by pulverization the solution thus obtained, the inlet temperature being of 2100C and the temperature of exit de920C. The moisture content final of the resulting powder is of approximately 10%. One makes pass this powder through a sieve of 44 microns to obtain a product of a uniform granulometry. This product is an active complex of antitranspiration being able to be built-in a compositiondaerosol.

EXAMPLE 3 One uses the following components to prepare a pulverizable poudreanti perspiring by Composantsffi aerosol in weight propellent Agent (1) 30,00 citric Acid " Bentone 38" 0,60 monohydrate 0,02 Mixture of ethanol and water 95/5ffi in weight) 0,27 Myristatedtisopropyle 3,50 ZAG (2) 5,00 Perfume 0,20 Fluid propellent (3) q.s.p. 100ss (1) CC1 F (2) Complex of antitranspiration powder prepared selonltexem- ple 2.

3) CCIDF: CC12F2 (40/60) in weight.

One as follows prepares an aerosol of antitranspiration by using the composition described above has 8,16 kg of trichlorofuloromethane, one adds 272 G of " Bentone-38", 9 G of citric acid and 122 G of an aqueous solution with ethanol 95g, then one mixes by using a mixer with strong shearing during 3 minutes approximately with 10OC, To freezing thus formed, one adds 1,58 kg of myristatedtisopropyle, 2,27 kg of pulverulent complex active ZAG of the 2,90g example of perfume and 5,44 kg of trichlorofluoromethane. One agitates carefully during 5 minutes at 10 C then one crushes the load by making it pass through a filter to fall down in a collecting container. One

uses this concentrate to fill a container with aerosol and then one adds propellent gas using a device of filling assembled under the cap.

EXAMPLE 4 One prepares a pulverizable dry powder by aerosol of an agent of antitranspiration starting from the ingredients indicated cidessous and practically according to the same process as in example 3.

Component % (in weight) Propellent (1) 30,00 "Cab-o-Sil" (M-5 or H-5) 0,60 Acidecirrique monohydrate 0,02 AlcoolethyliqueXeau (95/5 in weights) 0,27 Myristatedtisopropyle 3,50 ZAG (2) 5,00 perfume 0,20 Engine (3) q.s.p. 100 (1) CCl F Gomplexe of powder of antitranspiration prepared selonltexem ple 2.

3) CCl3F: CCl2F2 (40/60 in weight) -.

After distribution starting from a container for ae rosoi, une dry film of highly effective powder of antitranspiration is formed on the skin.

EXEMPIE 5 One uses the following components to prepare a pulverizable powder of antitranspiration in aerosol, by same the procedequ1a the example 3 Composantffi in weight Propulseur (i) 30,00 ZAG (2) 5,00 Acid citric monohydrate 0,02 Phthalate of dibutyle3,50 Ethanol/water (95/5% in weights) 0,27 "Bentone-38 " Cab-O-Sil" M-5 or H-5 (50/50) 0,60 Perfume 0,20 Engine (3) q.s.p. 100- (i) CG13F (2) Complex of powder of antitranspiration of the example 2.

3) CC13F: CC12F2 (40/60 in weight).

One obtains a highly effective powder of aerosol for pulverization of a product of antitranspiration.

One prepares compositions identical to that del' exem- ple 5 by leprocede describes in example 3 except that one replaces dibutyl phthalate by the isopropyl myristate, the isopropyl palmitate, the tetradecane, alcohol laurylic, alcohol hexadecylic, alcohol oleylic, the lauric acid, the oleic acid, the lanolin, the acetylated lanolin, the dimethylpolysiloxane, the sebacate of diethyle, adipate of diisopropyle and ethyl ethylcarbomethyl-phthalate (orthoC2H5OOC- - COOCH2COOC2H5). One prepares these compositions according to a process practically identical to that described in example 3 and one obtains in each case of the results practically identical to those of example 5.

One prepares compositionsanalogists - with those del' exem- ple 5 except that one replaces the mixture " Bentone-38"/"Cab-O-Sil" by the stearoyl-monoethanolamide, the laurylamine, the stearylamine, the tetradecylamine, 11hexadecylamine, the octadecylamine, I. E.(internal excitation) Co syla- mine, copra-monoethanolamide and the octadecylmonoethanolamide.

One prepares these compositions by the same process that dansl' exem- ple 3 and one always obtains results equivalent to those of example 5.

In addition to the uses described in examples 1 to 5, for the powders of antitranspiration in aerosol, it is obvious quel' in vention includes the liquid solutions of the complexes aluminum/zirconium/glycine which were described above in prepared tions cosmetic, liquid, semi-fluid or of autrestypes In what follows, one presents a formula representative of a cream and one antitranspirant liquid which be-prepared in each case with the complexes of antitranspiration according to the invention.

Component % in aqueous Solution weight to 40% of zirconium the desired compound of example 1 13,6 aqueous Solution with 50ffi of aluminum chlorohydroxyde 15,0 Glycine 2,0 distilled Water 43,0 Perfume 0,2 Monostearate of glyceryle 10,0 Waxes spermaceti 4,0 Mineral oil (15 Cs a380C) 4,0 Stearate of polyoxyethylene (50) "Myrj 53" 5,0 Glycerin 3,0 titanium 0,2 Dioxide Composantffi LIQUID in aqueous Solution weight to 40% of zirconium the desired compound of example 1 1D, 6 aqueous Solution with 50ss of aluminum chlorohydroxyde 15,0 Glycine 2,0 Monooleate of polyoxyethylene (20) - sorbitane "Twen 80" 2,0Ethan ol 2,0 Perfume 0,2 Water 65,2

VE.

ROYAUME DE BELGIQUE

MINISTERE DES AFFAIRES ECONOMIQUES

BREVET D'INVENTION

N° 825.146

Classif. internat.: A 61 K

Mis en lecture le: 4-8-1975

Le Ministre des Affaires Economiques,

Vu la loi du 28 mai 1854 sur les brevets d'invention;

Vu la Convention d'Union pour la Protection de la Propriété Industrielle;

Vu le procès-verbal dressé le 4 février 1975 à 14 h.

au Service de la Propriété Industrielle;

ARRÊTE :

Article 1. — *Il est délivré* à la Sté dite: THE PROCTER & GAMBLE COMPANY, 301, East Sixth Street, Cincinnati, Ohio (Etats-Unis d'Amérique)

repr. par le Cabinet Bede à Bruxelles,

un brevet d'invention pour: Complexes d'antitranspiration,

qu'elle déclare avoir fait l'objet d'une demande de brevet déposée aux Etats-Unis d'Amérique le 4 février 1974 sous le n° 438.976 au nom de Mr. J.J. Schmitz dont elle est l'ayant droit.

Article 2. — *Ce brevet lui est délivré sans examen préalable, à ses risques et périls, sans garantie soit de la réalité, de la nouveauté ou du mérite de l'invention, soit de l'exactitude de la description, et sans préjudice du droit des tiers.*

Au présent arrêté demeurera joint un des doubles de la spécification de l'invention (mémoire descriptif et éventuellement dessins) signés par l'intéressé et déposés à l'appui de sa demande de brevet.

Bruxelles, le 4 août 1975

PAR DELEGATION SPECIALE:

R. RAUX

GR 75 18

Case 2050 R

B R E V E T D ' I N V E N T I O N

" Complexes d'antitranspiration "

Société dite :
THE PROCTER & GAMBLE COMPANY

C.I.: Demande de brevet des Etats-Unis d'Amérique n° 438.976 déposée le 4 février 1974 au nom de Jerome Joseph Schmitz dont la demanderesse est l'ayant droit.

ms

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La présente invention concerne des nouveaux composés utiles pour préparer des agents actifs anti-transpirants ou anti-perspirants, les termes antiperspirant et anti-transpirant ayant la même signification au sens de la présente invention ; l'invention a également pour objet un procédé pour l'obtention d'un complexe antitranspirant actif d'une efficacité remarquable, que l'on peut incorporer dans des compositions antitranspirantes ou compositions d'antitranspiration, compositions qui ne sont ni irritantes pour la peau ni corrosives pour les textiles.

De nombreux composés chimiques capables d'empêcher ou de retarder l'exsudation de la transpiration ont déjà été utilisés ou proposés pour utilisation dans les compositions d'antitranspiration. A l'heure actuelle, on utilise très couramment les sels d'aluminium des acides forts, surtout les chlorures. Le chlorohydroxyde d'aluminium, dont la formule empirique est $Al_2(OH)_5Cl,2H_2O$ constitue le produit le plus souvent choisi par les fabricants des agents d'antitranspiration. Depuis peu de temps, on a suggéré l'utilisation de divers dérivés de zirconium seuls ou en combinaison avec les sels d'aluminium dans les produits d'antitranspiration. Les sels de zirconium utilisés antérieurement dans les produits d'antitranspiration étaient l'oxychlorure de zirconium (formule empirique $ZrOCl_2,dH_2O$) ou l'hydroxychlorure de zirconyle (formule empirique $ZrO(OH)Cl,3H_2O$).

Bien qu'on ait proposé divers dérivés du zirconium utilisables dans les compositions d'antitranspiration, les plus efficaces sont les sels des acides forts, qui forment des solutions d'une acidité tellement intense, qu'elles deviennent extrêmement irritantes pour la peau et endommagent également les vêtements. On peut utiliser l'oxychlorure de zirconium à la condition cependant de le combiner avec un complexe de chlorohydroxyde d'aluminium et un aminoacide neutre (voir brevet U.S. 2.814.585). Le complexe de chlorohydroxyde d'aluminium et l'aminoacide jouent le rôle d'agents de tamponnement ou agents tampons.

On a malheureusement constaté que l'oxychlorure de zirconium présente certains inconvénients même quand on l'utilise au sein d'une composition d'antitranspiration tamponnée de façon appropriée. Il est difficile de sécher les mélanges décrits ci-dessus pour obtenir une poudre sèche en raison de la présence des 8 H_2O dans l'oxychlorure de zirconium (eau d'hydratation). De même, quand on obtient une poudre sèche, cette dernière est extrême-

ment hygroscopique. Bien que l'addition d'un aminoacide plus neutre permette de réduire le caractère hygroscopique, la quantité de l'aminoacide nécessaire pour obtenir une composition appropriée, c'est-à-dire dont l'usage est sans danger, qui n'endommage pas les vêtements et qui est compatible avec les flacons de conditionnement et avec les parfums, est tellement grande qu'il en résulte une forte augmentation de prix et une baisse d'efficacité.

D'autre part, l'oxychlorure de zirconium subit dans une solution aqueuse des réactions longues et compliquées d'hydrolyse. Il est donc difficile de maintenir un pH stable dans une solution aqueuse d'oxychlorure de zirconium avant le séchage et, par ailleurs, le pH d'un lot peut être différent de celui du lot suivant. Il en résulte des produits qui sont différents de façon inhérente après incorporation dans des compositions finales.

L'hydroxychlorure de zirconyle ne présente pas les mêmes inconvénients que l'oxychlorure de zirconium. Il est moins acide, donc moins irritant et contient un plus petit nombre de groupes d'hydratation H_2O, de sorte qu'il est moins hygroscopique. Toutefois, l'hydroxychlorure de zirconyle est notablement moins efficace que l'oxychlorure de zirconium comme agent d'antitranspiration et les considérations de sécurité exigent malgré tout l'utilisation de certains agents-tampons, tels que le chlorohydroxyde d'aluminium et un aminoacide neutre (voir à cet effet le brevet US 2.854.382).

Les solutions d'hydroxychlorure de zirconyle, de chlorohydroxyde d'aluminium et d'aminoacide produisent une poudre presque blanche au séchage. Malheureusement, le produit d'antitranspiration ainsi obtenu est moins efficace que la combinaison oxychlorure de zirconium/chlorohydroxyde d'aluminium/aminoacide, bien que plus efficace que le chlorohydroxyde d'aluminium seul.

Des essais avec un certain nombre de sels de zirconium moins acides, par exemple avec le carbonate, le lactate, le glycolate, ou l'acétate de zirconium ou le trioxychlorure de dizirconium, ont montré que tous ces produits sont moins efficaces que l'hydroxychlorure de zirconyle en ce qui concerne le pouvoir antitranspirant.

La présente invention concerne un nouveau composé de zirconium qui est hautement efficace comme agent d'antitranspiration et qu'on peut incorporer dans des compositions vendues aux

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utilisateurs qui sont de nature à ne pas irriter la peau, ne pas endommager les vêtements et ne pas corroder les récipients de conditionnement .

La présente invention a également pour objet un complexe pulvérulent homogène d'antitranspiration, qui possède une grande efficacité pour empêcher ou retarder l'exsudation de la transpiration et qu'on peut incorporer dans des compositions vendues aux utilisateurs, qui sont de nature à ne pas irriter la peau, ne pas endommager les vêtements et ne pas corroder les récipients de conditionnement.

Un autre objet de l'invention est constitué par un composé d'antitranspiration hautement efficace, qu'on peut utiliser facilement pour préparer des produits d'antitranspiration dont l'usage est sans danger et dont les caractéristiques sont attrayantes pour les utilisateurs, ces produits pouvant être des liquides, des lotions, des crèmes ou des aérosols tels que des aérosols en poudres sèches.

D'autres objets de l'invention ressortiront de la description ci-après, dans laquelle toutes les parties, les pourcentages et les rapports sont en poids sauf stipulation contraire.

La présente invention est fondée sur la découverte de nouveaux composés répondant à la formule empirique : $MO(OH)_{2-a}X_a.nH_2O$ dans laquelle M représente le zirconium ou l'hafnium, X représente le chlore, le brome ou l'iode, a est un nombre d'environ 1,50 à environ 1,87 et n est un nombre de 1 à 7 environ. On conçoit que le nombre de groupes H_2O d'hydratation ne présente de l'importance que lorsqu'on sèche le composé. Cependant, le plus souvent on ne sèche pas le composé mais on l'utilise sous forme d'une solution dans les étapes ultérieures de traitement.

On utilise utilement les nouveaux composés selon l'invention en combinaison avec un sel d'aluminium astringent et un aminoacide neutre en qualité de composés d'antitranspiration qu'on peut incorporer dans des produits d'antitranspiration renfermant d'autres ingrédients classiques.

On a constaté que les objets de l'invention peuvent être réalisés si l'on utilise un composé de formule empirique : $MO(OH)_{2-a}X_a.nH_2O$ dans laquelle M représente le zirconium ou l'hafnium, X représente le chlore, le brome ou l'iode, a est un nombre de 1,50 à 1,87 environ et n est un nombre de 1 à 7 environ.

Les composés selon l'invention peuvent présenter des poids moléculaires très variés. On préfère cependant que le poids moléculaire moyen soit compris entre environ 300 et 25.000 et, mieux encore, entre environ 300 et 1500.

Un élément polyvalent, tel que le zirconium, est capable de former des composés polymères. En fait, les composés de zirconium sont en général polymères dans les solutions aqueuses sauf à une très forte acidité. Les poids moléculaires des polymères de zirconium dépendent des procédés utilisés dans le traitement et aussi de la pureté des matières premières servant à préparer les composés polymères. Par exemple, un oxychlorure de zirconium fortement acide est un polymère d'un assez bas poids moléculaire. On peut le neutraliser dans des solutions aqueuses pour obtenir un polymère ayant un poids moléculaire plus élevé.

On pense que le procédé pour l'obtention des composés selon l'invention est critique pour la fabrication des produits préconisés. On a constaté que pour préparer les composés contenant du zirconium selon l'invention, on peut utiliser le procédé suivant qui consiste :

(A) à dissoudre dans l'eau un sel hydrosoluble de zirconium, par exemple l'oxychlorure de zirconium;

(B) à ajouter lentement une base forte, telle que NaOH, KOH, ou NH_4OH à la solution de (A) jusqu'à la formation d'un précipité de zircone hydratée;

(C) à isoler le précipité par des moyens appropriés, par exemple par filtration;

(D) à laver ledit précipité avec de l'eau; et

(E) à dissoudre le précipité dans une solution d'acide chlorhydrique, cette solution contenant 1,50 à 1,87 mole de HCl par mole de zirconium dans le précipité.

Pendant le procédé, on doit maintenir avantageusement la température au-dessous de 100°C et, mieux encore, entre 20 et 100°C.

Le composé de zirconium ainsi obtenu est un agent d'anti-transpiration très efficace mais il n'est pas aussi hygroscopique ou aussi irritant pour la peau humaine que l'oxychlorure de zirconium.

Comme exemples de sels hydrosolubles de zirconium utilisables dans l'étape (A), on peut citer le sulfate, le nitrate, et les halogénures de zirconium par exemple l'oxychlorure de zirco-

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nium, l'hydroxychlorure, le chlorure, le bromure et l'iodure de zirconyle et le tétrachlorure de zirconium.

On peut évidemment utiliser à la place des composés contenant du zirconium et du chlore, respectivement, des proportions équivalentes d'hafnium et d'un autre halogénure (tel que le brome ou l'iode). Cependant, les composés préférés sont ceux à base de zirconium et de chlore.

On a constaté que l'efficacité d'un complexe aluminium/zirconium/aminoacide est fortement augmentée, sans aucun effet secondaire nuisible, par exemple sans accroissement de l'effet irritant, quand on utilise les composés selon l'invention.

Ainsi, les complexes d'antitranspiration qui sont hautement efficaces et non irritants comprennent :

(A) 0,15 à 1,5 partie d'un nouveau composé selon l'invention;

(B) 1 partie d'un composé basique d'aluminium de formule empirique : $Al_2(OH)_{6-m}X_m.nH_2O$, dans laquelle X est le chlore, le brome ou l'iode, m a une valeur moyenne d'environ 0,8 à 2,0 et n est un nombre de 0 à 12 environ; et

(C) 0,06 à 0,60 partie d'un aminoacide neutre, tel que décrit plus loin.

On a constaté que les halogénohydroxydes d'aluminium de formule empirique $Al_2(OH)_{6-m}X_m.nH_2O$, dans laquelle X est le chlore, le brome ou l'iode, m a une valeur moyenne d'environ 0,8 à 1,5 et n d'environ 0 à 12, sont facilement applicables aux fins de l'invention bien qu'on puisse utiliser un composé basique d'aluminium quelconque dans les limites définies ci-dessus et obtenir des résultats satisfaisants. On remarquera que ces composés basiques d'aluminium possèdent eux-mêmes des propriétés d'antitranspiration.

Comme exemples de composés basiques d'aluminium, on citera le chlorhydroxyde d'aluminium, l'hydroxybromure d'aluminium et le chlorure hydraté d'aluminium basique à 75 % de formule $Al_2(OH)_{4,5}Cl_{1,5}$. On préfère spécialement le chlorohydroxyde d'aluminium de formule empirique $Al_2(OH)_5Cl.2H_2O$. Le chlorohydroxyde d'aluminium est un sel bien connu qui est efficace contre la transpiration et qui est disponible dans le commerce (vendu par exemple par Rahcis Chemical Company).

Les aminoacides qui conviennent aux fins de l'invention sont ceux dans lesquels le nombre des groupes amino est égal à celui des groupes carboxyle dans la molécule. Dans un but de commodité, ces

aminoacides sont appelés "aminoacides neutres" dans le présent mémoire descriptif. Bien qu'on puisse utiliser un aminoacide neutre quelconque du type défini ci-dessus pour aboutir aux objectifs de l'invention, on préfère les aminoacides neutres qui sont solubles dans des solutions aqueuses, par exemple la glycine, l'alanine, la β-analine, la sérine, l'acide 2-aminobutyrique, la valine, la β-phénylalanine, la méthionine ou le tryptophane. On peut utiliser la forme l, la forme d ou la forme dl de ces composés. L'amino-acide spécialement préféré est la glycine.

On ajoute l'aminoacide neutre lors de la préparation des complexes selon l'invention en une proportion permettant d'empêcher la gélification prématurée du complexe en solution. Si l'on utilise une quantité insuffisante de l'aminoacide, la solution du sel de zirconium ou d'hafnium plus le composé d'aluminium subira une gélification au bout de très peu de temps. Si, d'autre part, on utilise une proportion excessive de l'aminoacide, on observe une baisse d'efficacité, et certaines combinaisons de sel de zirconium/sels d'aluminium finissent par se gélifier. On comprend que dans tous les cas, il convient d'incorporer l'aminoacide en une quantité suffisante pour empêcher la gélification du principe actif d'antitranspiration ou de la composition contenant ce principe actif. Cette considération est extrêmement importante car les gels possèdent des propriétés d'antitranspiration tellement limitées que leur usage est considéré comme pratiquement impossible dans ce domaine.

Dans la préparation des produits finals utilisables par les consommateurs , à partir des complexes selon l'invention, la concentration du complexe peut être d'environ 1 à 30 % en poids par rapport à la composition totale.

Le brevet U.S. 3.792.068 décrit des complexes d'antitranspiration en poudre, des procédés de préparation de ces complexes et les produits utilisables par les consommateurs contenant de tels complexes. Les enseignements de ce brevet sont expressément incorporés à titre de référence dans la présente demande. On peut remplacer les sels de zirconium décrits dans le brevet précité par les nouveaux composés de zirconium et d'hafnium selon la présente invention, pour obtenir des complexes d'antitranspiration hautement efficaces et qu'on peut utiliser pour préparer des compositions d'antitranspiration d'un usage sans danger et d'un aspect cosmétiquement attrayant pour la vente aux consommateurs.

Ainsi, pour former un complexe d'antitranspiration en poudre possédant une grande efficacité, on procède comme suit : on sèche conjointement un sel basique d'aluminium, un composé d'hafnium ou de zirconium selon l'invention et un aminoacide neutre du type défini ci-dessus, ces composants ayant été préalablement mélangés ensemble dans une solution aqueuse ; on sèche le complexe jusqu'à une teneur finale en humidité d'environ 1 à 30 % en poids, de préférence d'environ 5 à 20 %. Le complexe pulvérulent ainsi formé possède des propriétés supérieures inattendues qu'on n'obtient pas si l'on sèche séparément les composants et qu'ensuite on les combine par un simple procédé de mélange mécanique.

Pour préparer un complexe pulvérulent d'antitranspiration on peut opérer comme suit :

(A) on dissout ensemble dans de l'eau :

(1) 1 partie de $Al_2(OH)_{6-m}X_m$, formule dans laquelle X est un ion halogénure choisi parmi les ions chlorure, bromure, ou iodure et m est un nombre d'environ 0,8 à 2,0 ;

(2) environ 0,15 à 1,5 partie du composé nouveau selon l'invention; et

(3) environ 0,06 à 0,60 partie d'un aminoacide neutre choisi parmi la glycine, le dl-tryptophane, la dl-β-phényl-analine, la dl-valine, la dl-sérine, la dl-méthionine, la dl-alanine, la β-alanine ou l'acide 2-amino-butyrique;

(B) on sèche cette solution jusqu'à une teneur en humidité d'environ 1 à 30 % en poids et, éventuellement ,

(C) on broie le complexe séché pour former une poudre uniforme impalpable avant d'incorporer le complexe dans une composition d'aérosol.

En ce qui concerne le procédé de séchage, on peut utiliser un séchage ordinaire en étuve, un séchage en tambour, un séchage par pulvérisation, un séchage par le vide, une lyophilisation, un séchage aux micro-ondes, etc... Bien que le choix du procédé de séchage ne soit pas critique, on préfère le séchage par pulvérisation. Pendant le séchage, on ne chauffe pas, de préférence, le complexe antitranspirant au-dessus de 230°C par suite du risque d'une décomposition de l'aminoacide et de la formation d'oxydes insolubles indésirables de zirconium ou d'aluminium à une température plus élevée. Dans l'opération de séchage par pulvérisation, la température du produit est sensiblement la même que celle de l'air à la sortie de l'enceinte de séchage et non celle de l'air à l'en-

trée de cette enceinte. Ainsi, alors que la température de l'air d'entrée peut être supérieure à 230°C, on préfère que l'air sortant soit à une température au-dessous de 230°C.

Parmi les procédés de broyage appropriés, on mentionnera le broyage aux boulets, le broyage aux rouleaux, le passage à travers un broyeur colloïdal, le broyage à énergie de fluide par impact etc... Selon la nature de l'équipement de broyage, il peut être ou ne pas être nécessaire de faire passer la poudre broyée à travers un tamis pour en assurer l'uniformité et éliminer les granules trop gros.

Si l'on doit incorporer la poudre dans un emballage ou récipient pour aérosol, la granulométrie du complexe pulvérulent sec doit être suffisamment faible pour permettre une pulvérisation uniforme sans risque d'un fonctionnement défectueux de l'ajutage ou de la soupape. Bien qu'il soit possible d'utiliser des particules atteignant 100 microns de diamètre, la granulométrie préférée est comprise entre environ 5 et 40 microns.

On préfère que le rapport atomique de l'aluminium au zirconium dans le complexe décrit soit d'environ 4,2:1 à 2,5:1. L'aminoacide neutre doit être présent en une quantité suffisante pour empêcher la gélification ou formation de gel des solutions des sels d'aluminium et de zirconium. L'aminoacide neutre préféré est la glycine qu'on incorpore avantageusement dans un rapport molaire aluminium/glycine d'environ 1,75:1 à 5,55:1.

Une composition pulvérulente d'antitranspiration sous forme d'un aérosol, contenant le complexe décrit plus haut, peut être préparée en combinant les ingrédients suivants :
(A) environ 1 à 12 % en poids d'un complexe d'antitranspiration du type décrit ci-dessus,
(B) environ 0,1 à 5 % en poids d'un agent de suspension;
(C) environ 1 à 15 % en poids d'un véhicule liquide; et
(D) une quantité suffisante d'un gaz anhydre propulseur pour produire une pulvérisation du type aérosol.

Composant A

Aux fins de l'invention, le composé préféré d'antitranspiration est un complexe minéral/organique qui a déjà été décrit en détail précédemment.

On incorpore environ 1 à 12 % en poids, de préférence 2,5 à 6 % environ de ce composé dans une composition aérosol et on obtient une excellente efficacité d'antitranspiration. Une proper-

tion inférieure à environ 1 % réduit l'efficacité antitranspirante. Au-dessus de 12 %, la composition n'est pas économiquement rentable pour autant que son efficacité n'augmente pas proportionnellement avec l'incorporation d'une quantité supplémentaire et on se heurte à de nouveaux problèmes de manipulation et d'atomisation.

Composant B

L'agent de suspension maintient le complexe pulvérulent d'antitranspiration en suspension dans la composition. Bien qu'un certain dépôt puisse avoir lieu, le complexe d'antitranspiration demeure dans un état facile à redisperser. Parmi les agents de suspension appropriés, on citera la silice colloïdale (de préférence d'une granulométrie au-dessous de 0,03 micron de diamètre, par exemple le produit connu sous la dénomination commerciale "Cab-O-Sil M-5" (produit vendu par Cabot Corp.), les produits décrits dans la demande de brevet aux Pays-Bas n° 65/13943 publiée le 4 avril 1968 par Spitzer et la silice particulaire submicroscopique préparée dans un environnement gazeux chaud (1100°C) par l'hydrolyse de la phase supérieure d'un composé de silicium. D'autres agents appropriés de suspension sont les argiles hydrophobes, qui gonflent dans les solvants organiques, par exemple le produit connu sous la dénomination commerciale "Bentone-38" (produit vendu par N.L. Industries) qui est une argile montmorillonite modifiée par voie organique, les amines aliphatiques primaires contenant de 12 à 20 atomes de carbone, par exemple la laurylamine, la tétradécylamine, l'hexédécylamine, l'octadécylamine et l'eicosylamine; des savons formant des graisses comme le stéarate d'aluminium; l'alumine fumée; et des monoalkylol-amides aliphatiques saturés comportant une chaîne d'atomes de carbone d'acides gras d'environ 12 à 20 atomes de carbone et une chaîne alkylol d'environ 2 à 3 atomes de carbone, des exemples de ces amides étant le coprah-monoéthanolamide, l'octadécyl-monoéthanolamide, comme décrit dans le brevet belge n° 760.957.

Les agents de suspension qui viennent d'être décrits ou leurs mélanges peuvent être utilisés à raison d'environ 0,1 à 5 % en poids. On préfère une proportion de 0,3 à 0,8 % environ. Les agents de suspension qu'on préfère tout particulièrement aux fins de l'invention sont les argiles hydrophobes qui gonflent dans les solvants organiques , par exemples les argiles connues sous la dénomination commerciale "Bentone-38".

Composant C

On utilise un véhicule liquide faiblement volatil pour que le courant émanant du récipient d'aérosol soit une pulvérisation humide et non un nuage de particules ou de poussière. En effet, on obtient ainsi une sensation plus agréable sur la peau et on réduit les risques d'une inspiration de la poudre sèche. le véhicule liquide améliore également l'efficacité car il maintient le composé d'antitranspiration en contact avec la peau dans des conditions ne permettant pas la chute du composé par effritement ou par lavage. Parmi les véhicules liquides appropriés, on indiquera les esters carboxyliques, tels que le myristate ou le palmitate d'isopropyle; les hydrocarbures liquides comme le tétradécane; les alcools gras comme l'alcool laurylique, hexadécylique ou oléylique; les acides gras tels que l'acide lauryque ou oléique; la lanoline et ses dérivés tels que la lanoline acétylée; et les huiles de silicones, telles que le diméthylpolysiloxane. D'autres composés organiques pouvant servir de véhicules liquides sont ceux contenant des groupes esters multiples, par exemple les diesters de diacides organiques et des composés contenant des groupes esters multiples, par exemple le phtalate de di-n-butyle, le sébacate de diéthyle, l'adipate de diisopropyle et l'éthylcarbométhylphtalate d'éthyle (ortho-C_2H_5OOC-Ø-$COOCH_2COOC_2H_5$).

D'autres véhicules liquides appropriés sont le monolaurate de polyéthylène-glycol et le butanol éthoxylé ou propoxylé, par exemple les produits connus sous la dénomination commerciale ®Ucon-HB-660 et Ucon HB-5100 (Union Carbide).

Parmi les véhicules liquides qui ont été énumérés, on préfère les composés organiques contenant des esters multiples. On préfère tout spécialement le phtalate de di-n-butyle, le sébacate de diéthyle, l'adipate de diisopropyle et l'éthylcarbométhylphtalate d'éthyle.

Les véhicules liquides décrits peuvent être utilisés à raison d'environ 1 à 15 % . Au-dessous d'environ 1 % la quantité est insuffisante pour obtenir une pulvérisation humide et cette dernière est donc trop poussiéreuse ou granuleuse et n'adhère pas bien à la peau. Au-dessus de 15 % environ, la composition déposée sur la peau donne une sensation désagréable grasse et huileuse. On préfère une proportion de véhicule liquide d'environ 2 à 8 %.

Composant D

Le gaz propulseur utilisé peut être un gaz liquéfiable quelconque qu'on met en oeuvre couramment dans les aérosols. Comme exemples de gaz propulseurs utilisables, on citera le trichlorofluorométhane, le dichlorodifluorométhane, le dichlorotétrafluoroéthane, le monochlorodifluorométhane, le trichlorotrifluoroéthane, le propane, le butane et l'isobutane, seuls ou en mélanges. On préfère spécialement le trichlorofluorométhane, le dichlorodifluorométhane, le dichlorotétrafluoroéthane et l'isobutane, seuls ou en mélanges.

La proportion du gaz propulseur à utiliser est déterminée par les facteurs usuels bien connus des spécialistes des aérosols. Il est satisfaisant de considérer le gaz propulseur comme formant le complément de la composition, c'est-à-dire la quantité en plus des autres composants étudiés en détail plus haut. En conséquence, les proportions préférées du propulseur sont d'environ 70,7 à 93,9 % , et tout particulièrement de 80 à 92 % environ.

Il est évident que d'autres ingrédients peuvent être incorporés en petites quantités dans la composition, sans influer sur la nature de l'invention. Un exemple d'un tel ingrédient supplémentaire est un parfum en une proportion allant jusqu'à 0,8 % environ.

Les exemples suivants servent à illustrer l'invention sans aucunement en limiter la portée.

EXEMPLE 1

On prépare comme suit un composé d'antitranspiration à base de zirconium, selon l'invention : on dissout 32,2 g d'oxychlorure de zirconium, $ZrOCl_2,8H_2O$, dans 32,2 ml d'eau distillée sous agitation et on ajoute lentement à la solution ci-dessus, 20 g d'une solution à 40 % d'hydroxyde de sodium.

Il se forme un précipité qu'on sépare de la solution par filtration, puis on lave ce précipité qui contient 9,12 g de zirconium avec environ 2 litres d'eau, on ajoute 17,3 g d'une solution à 37 % d'acide chlorhydrique à ce précipité humide, on chauffe à 60°C et on agite jusqu'à dissolution du précipité. On soumet ensuite la solution à une évaporation sous vide pour éliminer 12,5 parties d'eau et on obtient ainsi une solution à 40 % du composé de zirconium désiré.

Ce composé de zirconium présente un rapport Cl:Zr de 1,76 et sa formule empirique est $ZrO(OH)_{0,24}Cl_{1,76}$. On ne sèche pas le

composé mais on le laisse dans la solution ; c'est pour cette raison que l'on n'indique pas le nombre de groupes H_2O d'hydratation.

EXEMPLE 2

Pour préparer un complexe d'antitranspiration en poudre, on procède comme suit : on introduit dans un bécher 15 g d'une solution aqueuse à 50 % de chlorohydroxyde d'aluminium ("Chlorhydrol" marque déposée de Reheis Chemical Company). On ajoute de la glycine à raison de 2,0 g et on dissout complètement sous agitation. On ajoute ensuite 13,6 g de la solution aqueuse (à environ 40 %) du composé désiré de zirconium de l'exemple 1, avec agitation continue, jusqu'à une codissolution de tous les composants. On sèche par pulvérisation la solution ainsi obtenue, la température d'entrée étant de 210°C et la température de sortie de 92°C. La teneur en humidité finale de la poudre résultante est d'environ 10 %. On fait passer cette poudre à travers un tamis de 44 microns pour obtenir un produit d'une granulométrie uniforme. Ce produit est un complexe actif d'antitranspiration pouvant être incorporé dans une composition d'aérosol.

EXEMPLE 3

On utilise les composants suivants pour préparer une poudre antitranspirante pulvérisable par aérosol :

Composants		% en poids
Agent propulseur (1)		30,00
"Bentone 38"		0,60
Acide citrique monohydraté		0,02
Mélange d'alcool éthylique et d'eau (95/5 % en poids)		0,27
Myristate d'isopropyle		3,50
ZAG (2)		5,00
Parfum		0,20
Fluide propulseur (3)	q.s.p.	100 %

(1) CCl_3F

(2) Complexe d'antitranspiration pulvérulent préparé selon l'exemple 2.

(3) CCl_3F:CCl_2F_2 (40/60) en poids.

On prépare comme suit un aérosol d'antitranspiration en utilisant la composition décrite ci-dessus :

A 8,16 kg de trichlorofluorométhane , on ajoute 272 g de "Bentone-38" , 9 g d'acide citrique et 122 g d'une solution aqueuse à 95 % d'éthanol, puis on mélange en utilisant un mélangeur à fort cisaillement pendant 3 minutes environ à 10 °C. Au gel ainsi formé, on ajoute 1,58 kg de myristate d'isopropyle, 2,27 kg du complexe pulvérulent actif ZAG de l'exemple 2, 90g de parfum et 5,44 kg de trichlorofluorométhane. On agite soigneusement pendant 5 minutes à 10 °C puis on broie la charge en la faisant passer à travers un filtre pour retomber dans un récipient collecteur. On utilise ce concentré pour remplir un récipient d'aérosol et ensuite on ajoute le gaz propulseur à l'aide d'un dispositif de remplissage monté sous le capuchon.

EXEMPLE 4

On prépare une poudre sèche pulvérisable par aérosol d'un agent d'antitranspiration à partir des ingrédients indiqués ci-dessous et pratiquement selon le même procédé que dans l'exemple 3.

Composant		% (en poids)
Propulseur (1)		30,00
"Cab-O-Sil" (M-5 ou H-5)		0,60
Acide citrique monohydraté		0,02
Alcool éthylique/eau (95/5% en poids)		0,27
Myristate d'isopropyle		3,50
ZAG (2)		5,00
Parfum		0,20
Propulseur (3)	q.s.p.	100

(1) CCl_3F

(2) Complexe de poudre d'antitranspiration préparé selon l'exemple 2.

(3) CCl_3F:CCl_2F_2 (40/60 en poids).

Après distribution à partir d'un récipient pour aérosol, une pellicule sèche de poudre d'antitranspiration hautement efficace se forme sur la peau.

EXEMPLE 5

On utilise les composants suivants pour préparer une poudre

d'antitranspiration pulvérisable en aérosol, par le même procédé qu'à l'exemple 3.

Composant		% en poids
Propulseur (1)		30,00
ZAG (2)		5,00
Acide citrique monohydraté		0,02
Phtalate de dibutyle		3,50
Alcool éthylique/eau (95/5 % en poids)		0,27
"Bentone-38"Cab-O-Sil" M-5 ou H-5 (50/50)		0,60
Parfum		0,20
Propulseur (3)	q.s.p.	100

(1) CCl_3F

(2) Complexe de poudre d'antitranspiration de l'exemple 2.

(3) $CCl_3F:CCl_2F_2$ (40/60 en poids).

On obtient une poudre d'aérosol hautement efficace pour pulvérisation d'un produit d'antitranspiration.

On prépare des compositions identiques à celle de l'exemple 5 par le procédé décrit dans l'exemple 3 sauf qu'on remplace le phtalate de dibutyle par le myristate d'isopropyle, le palmitate d'isopropyle, le tétradécane, l'alcool laurylique, l'alcool hexadécylique, l'alcool oléylique, l'acide laurique, l'acide oléique, la lanoline, la lanoline acétylée, le diméthylpolysiloxane, le sébacate de diéthyle, l'adipate de diisopropyle et l'éthylcarbométhyl-phtalate d'éthyle (ortho C_2H_5OOC-Ø-$COOCH_2COOC_2H_5$). On prépare ces compositions selon un procédé pratiquement identique à celui décrit dans l'exemple 3 et on obtient dans chaque cas des résultats pratiquement identiques à ceux de l'exemple 5.

On prépare des compositions analogues à celles de l'exemple 5 sauf qu'on remplace le mélange "Bentone-38"/"Cab-O-Sil" par le stéaroyl-monoéthanolamide, la laurylamine, la stéarylamine, la tétradécylamine, l'hexadécylamine, l'octadécylamine, l'eicosylamine, le coprah-monoéthanolamide et l'octadécylmonoéthanolamide. On prépare ces compositions par le même procédé que dans l'exemple 3 et on obtient toujours des résultats équivalents à ceux de l'exemple 5.

Outre les usages décrits dans les exemples 1 à 5, pour les poudres d'antitranspiration en aérosol, il est évident que l'invention englobe les solutions liquides des complexes aluminium/zirconium/glycine qui ont été décrits plus haut dans des prépara-

tions cosmétiques, liquides, semi-liquides ou d'autres types. Dans ce qui suit, on présente une formule représentative d'une crème et d'un antitranspirant liquide qui sont préparés dans chaque cas avec les complexes d'antitranspiration selon l'invention.

CREME

Composant	% en poids
Solution aqueuse à 40 % du composé désiré de zirconium de l'exemple 1	13,6
Solution aqueuse à 50 % de chlorohydroxyde d'aluminium	15,0
Glycine	2,0
Eau distillée	43,0
Parfum	0,2
Monostéarate de glycéryle	10,0
Cire de spermaceti	4,0
Huile minérale (15 cs à 38°C)	4,0
Stéarate de polyoxyéthylène (50) "Myrj 53"	5,0
Glycérine	3,0
Bioxyde de titane	0,2

LIQUIDE

Composant	% en poids
Solution aqueuse à 40 % du composé désiré de zirconium de l'exemple 1	13,6
Solution aqueuse à 50 % de chlorohydroxyde d'aluminium	15,0
Glycine	2,0
Monooléate de polyoxyéthylène(20)-sorbitane "Twen 80"	2,0
Ethanol	2,0
Parfum	0,2
Eau	65,2

REVENDICATIONS

1. Composé d'antitranspiration, caractérisé en ce qu'il répond à la formule empirique $MO(OH)_{2-a}X_a, nH_2O$ dans laquelle M est un métal choisi parmi le zirconium et l'hafnium, X est choisi parmi le chlore, le brome et l'iode, a est un nombre d'environ 1,5 à 1,87 et n est un nombre d'environ 1 à 7.

2. Composé selon la revendication 1, caractérisé en ce que son poids moléculaire moyen est d'environ 300 à 25.000 et en ce que M représente le zirconium et X représente le chlore.

3. Composé selon l'une des revendications 1 ou 2, caractérisé en ce que le poids moléculaire moyen du composé est d'environ 300 à 1500.

4. Complexe d'antitranspiration, caractérisé en ce qu'il comprend : (A) 0,15 à 1,5 partie d'un composé répondant à la formule empirique $MO(OH)_{2-a}X_a, nH_2O$ telle que définie dans la revendication 1; (B) 1 partie d'un composé d'aluminium basique de formule empirique : $Al_2(OH)_{6-m}X_m, nH_2O$ dans laquelle X est choisi parmi le chlore, le brome et l'iode, m a une valeur moyenne d'environ 0,8 à 2,0 et n a une valeur d'environ 0 à 12 ; et (C) 0,06 à 0,60 partie d'un aminoacide contenant un nombre égal de groupes amino et de groupes carboxyle.

5. Complexe selon la revendication 4, caractérisé en ce que le composé (A) a un poids moléculaire moyen d'environ 300 à 25.000 M représente le zirconium et X le chlore; en ce que le composé d'aluminium basique est le chlorohydroxyde d'aluminium; et en ce que l'aminoacide est choisi parmi les composés suivants : glycine, alanine, β-alanine, sérine, acide2-aminobutyrique, valine, β-phénylalanine, méthionine et tryptophane.

6. Complexe selon l'une des revendications 4 ou 5, caractérisé en ce que le poids moléculaire moyen de l'élément (A) est d'environ 300 à 1500.

7. Complexe selon la revendication 6, caractérisé en ce que l'aminoacide est la glycine.

8. Procédé pour l'obtention d'un composé astringent, caractérisé en ce qu'il consiste : (a) à dissoudre dans l'eau un sel hydrosoluble d'un métal choisi parmi le zirconium et l'hafnium; (b) à ajouter à la solution obtenue en (a) une base forte en quantité suffisante pour former un précipité; (c) à laver ledit précipité avec de l'eau; (d) à dissoudre le précipité dans une solution d'un acide choisi parmi les acides bromhydrique, iodhy-

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drique et chlorhydrique, ladite solution contenant environ 1,50 à 1,87 mole d'acide par mole de zirconium dans le précipité, la température pendant ledit procédé étant maintenue à une valeur au-dessous de 100°C environ.

9. Procédé selon la revendication 8, caractérisé en ce que le sel métallique hydrosoluble est un sel hydrosoluble de zirconium et ledit acide est l'acide chlorhydrique.

10. Procédé selon l'une des revendications 8 ou 9, caractérisé en ce que le sel métallique hydrosoluble est l'oxychlorure de zirconium, l'hydroxychlorure de zirconyle, le tétrachlorure de zirconium, le bromure de zirconyle, l'iodure de zirconyle, le sulfate de zirconium ou le nitrate de zirconium.

11. Complexe d'antitranspiration en poudre, caractérisé en ce qu'il est obtenu par le procédé qui consiste :

(A) à dissoudre ensemble dans de l'eau (a) 1 partie du composé $Al_2(OH)_{6-m}X_m$ dans lequel X et m sont tels que définis dans la revendication 4, (b) environ 0,15 à 1,5 partie d'un composé tel que défini dans la revendication 1, (c) environ 0,06 à 0,60 partie d'un aminoacide neutre choisi parmi les composés suivants : glycine, dl-tryptophane, dl-β-phénylalanine, dl-valine, dl-sérine, dl-méthionine, dl-alanine, β-alanine et acide 2-aminobutyrique; et

(B) à sécher la solution jusqu'à une teneur en humidité d'environ 1 à 30 % en poids.

12. Complexe en poudre selon la revendication 11, caractérisé en ce qu'on le prépare par un procédé selon lequel on choisit le composé $MO(OH)_{2-a}X_a,nH_2O$ de manière que M soit le zirconium, X soit le chlore et que le poids moléculaire moyen du composé soit d'environ 300 à 25.000 , l'aminoacide étant la glycine.

13. Complexe selon la revendication 12, caractérisé en ce que ledit poids moléculaire moyen est compris entre environ 300 et 1500.

14. Composition d'antitranspiration en poudre pour aerosol, caractérisée en ce qu'elle comprend (A) environ 1 à 12 % d'un complexe d'antitranspiration tel que défini dans la revendication 11; (B) environ 0,1 à 5 % en poids d'un agent de suspension; (C) environ 1 à 15 % en poids d'un véhicule liquide et (D) environ 70,7 à 93,9% en poids d'un propulseur gazeux anhydre liquéfiable.

15. Composition pour aérosol selon la revendication 14, caractérisée en ce que le métal M est le zirconium, X est le chlore et le poids moléculaire moyen du composé est d'environ 300 à 25.000 .

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(54) ANTI-PERSPIRANT AEROSOL SYSTEM

(71) We, BRISTOL-MYERS COMPANY, a Company organised under the laws of the State of Delaware, United States of America, of 345 Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to anhydrous aerosol antiperspirant systems comprising a powder suspension of antiperspirant material in a non-volatile, organic, oily liquid medium incorporated in an aerosol container. More particularly, it concerns a system of the aforesaid type in in which the antiperspirant material comprises a combination of water soluble aluminum compounds.

Aluminum compounds have long been known in the prior art to be effective as antiperspirants and deodorants. Thus, for example, aluminum chloride has been suggested for use as an active ingredient in antiperspirant and deodorant compositions alone or in combination with other aluminum salts such as aluminum chlorhydroxide. Some such compositions are commercially available as hydroalcoholic solutions or as emulsions in the form of creams or lotions.

With the advent of aerosol technology, efforts have been made to adopt this technology in the application of aluminum antiperspirants. However, several disadvantages and problems were soon encountered. In the first place, it was found that the hydroalcoholic solutions or emulsion, when dispensed from aerosol cans by means of propellants, gave a "wet spray" which was not well accepted by the consumer. Furthermore, although potentially the more effective antiperspirant, aluminum chloride alone, in the form of hydroalcoholic solutions or emulsions could not be employed practically, because of the highly acid nature of aluminum chloride which would tend to corrode the aerosol can and cause irritation. However, compositions such as these have been prepared in aerosol glass containers. These have circumvented the corrosion problem but they are still irritating and also wet.

In an effort to avoid this "wet spray" and can corrosion, attempts were made to formulate an anhydrous aerosol system in which the aluminum antiperspirant material was suspended as a fine powder in the aerosol liquid medium. Illustrative of these attempts are U.S. Patents 3,088,874; 3,288,681; Canadian Patent 771,633; French Patent 1,494,465 and Belgian Patent 687,228. Aluminum chloride was not employed in these attempts apparently because of its known acidic character described above and further because of its notorious hygroscopic nature. As a result of the latter, aluminum chloride would have a great tendency to pick up moisture and crystallize causing clogging of the actuator and valve system.

It has been recently suggested to incorporate aluminum chloride solution and an alcohol soluble complex compound obtained by reacting aluminum chloride or aluminum chlorhydroxide with a glycol monoether in an aliphatic monohydroxy alcohol such as ethanol (U.S. Patent 3,509,253). Although this reaction product can be incorporated in an aerosol form as an alcoholic solution, such compositions cannot be packaged in conventional metal containers because of their high corrosion potential. Moreover, they tend also to give a "wet spray".

In light of the known nature of aluminum chloride in formulating the anhydrous aerosol aluminum antiperspirant systems, the prior art tendency was to go to the less acidic water soluble aluminum salts such as aluminum chlorhydroxide and aluminum sulfate. Although these systems avoided some of the problems noted above, they left something to be desired with regard to their antiperspirant activity. For example, it has been observed that the antiperspirant activity of anhydrous

aerosol aluminum chlorhydroxide compositions is substantially lower than that observed in corresponding roll-ons, indicating that the full antiperspirant activity potential of aluminum chlorhydroxide has not been achieved in these aerosol systems.

From one aspect the present invention provides an anhydrous aerosol antiperspirant composition comprising a powdered aluminum antiperspirant mix distributed in a liquid vehicle comprising a non-volatile, organic, oily liquid having a boiling point under atmospheric pressure not lower than 120°C and a specific gravity of 0.7 to 1.6, and aerosol propellant, said powdered aluminum antiperspirant mix comprising aluminum chloride and a water soluble antiperspirant aluminum salt less acidic than aluminum chloride.

In one embodiment of the invention, the aluminium antiperspirant mix contains from 1 to 50%, and preferably from 5 to 20%, by weight of aluminum chloride, the balance being made up by the less acidic aluminum salt. In the case of aluminum chlorhydroxide, it is convenient to express the relative amounts of aluminum chloride and aluminum chlorhydroxide in terms of the molar ratios of aluminum to chloride in the dry mixture. In this case, the molar ratio of aluminum to chloride will be in the range of from 0.78:1 to 1.95:1. Preferably, this ratio will be in the range of from 1.33:1 to 1.79:1.

In preparing the antiperspirant powder mix of the present invention, in addition to the aluminum chloride any of the other water soluble aluminum compounds (less acidic than aluminum chloride) known in the prior art as effective as antiperspirant materials may be employed. By way of illustration of these, mention may be made of aluminum chlorhydroxide or its hydrates, aluminum sulfate, "Rehydrol" (trade mark) aluminum chlorhydroxide-propylene glycol complex), complex of zinc phenolsulfonate and chlorhydrol (ratio of aluminum to zinc from 12:1 to 6:1), aluminum potassium sulfate and "Wickenol 363D" (aluminum chlorhydroxide - polyethylene glycol mixture). However, the preferred aluminum compound of this character is aluminum chlorhydroxide.

The aluminum antiperspirant mixes employed in this invention may be prepared by various procedures. In one procedure they are prepared by drying aqueous solutions containing aluminum chloride and water soluble aluminum compounds. The dried powder, if need be, may be pulverized to a fine powder which passes through a 200 mesh sieve (74 microns) or finer. It is preferred that this powder pass 100% through a 270 mesh sieve (53 microns) and 99% through a 325 mesh (44 microns) sieve. In another procedure these compositions can be made by mixing aluminum chloride (i.e. $AlCl_3XH_2O$ wherein X=0—6) powder and, for example, aluminum chlorhydroxide powder. The drying technique used to remove water from the solutions to prepare the powder can be any of the conventional methods, e.g. oven-drying, vacuum oven-drying, spray drying or freeze drying.

In preparing the compositions of the invention, the antiperspirant powder mix is preferably first dispersed in the non-volatile, organic, oily liquid prior to introduction of the liquified aerosol propellant. The non-volatile organic, oily liquid is one which has a boiling point under atmospheric pressure not lower than 120°C, and a specific gravity of from 0.7 to 1.6, preferably from 0.7 to 1.2. These include such materials as liquid hydrocarbons (mineral oil, Iso-Par); fatty acid esters (isopropyl myristate, isopropyl palmitate); diesters of dicarboxylic acids (diisopropyl adipate); polyoxyalkylene glycol esters (polypropylene glycol 2000 monooleate); propylene glycol diesters of short chain fatty acids (C_2—C_{10}) (Neobee—M20); polyoxyethylene ethers ((polyoxyethylene (4) lauryl ether (Brij 30), polyoxyethylene (2) oleyl ether (Brij 92), polyoxyethylene (10) oleyl ether (Brij 96, Volpo (Trade Mark) 10)); polyoxypropylene cetyl ether (Procetyl); fatty alcohols (oleyl, hexadecyl, lauryl); propoxylated monohydric alcohol M.W. 880—930 (Fluid AP); and Silicon oils (dimethyl polysiloxane, 10—1000 centistokes) ("Brij" is a Trade Mark). Mixtures of the above non-volatile liquids are equally suitable for the purposes of this invention.

The propellant systems that may be employed in practicing the present invention are also quite varied. Any of the well-known propellants widely used in aerosol technology are applicable here. Generally, the propellant should have a boiling point lower than 75°F and a vapor pressure from 25 to 70 pounds per square inch gauge (psig) at 70°F, preferably from 30 to 40 pounds psig. A suitable vapor pressure can also be produced by blending two different propellants such as Freon 12 (70.2 psig at 70°F) and Freon 11 or Freon 11—S (Freon 11 stabilized with 0.3% nitromethane). ("Freon" is a Trade Mark).

Liquefied hydrocarbons, liquefied halogenated hydrocarbons, or a suitable mixture thereof are operative in this invention. Examples of materials which can be used as propellants either alone or in mixtures are trichloromonofluoromethane (Freon 11 or Freon 11—S), dichlorodifluoromethane (Freon 12), dichlorotetrafluoroethane (Freon 114), trichlorotrifluoroethane (Freon 113), propane, butane, pentane, and isobutane.

It is also desirable to incorporate in the aerosol compositions of this invention suspending agents to further prevent caking of the powder and to enable redispersing the powder by simple agitation. A variety of materials may be used (alone or incombination) for this purpose among which may be mentioned:

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(a) Cab-O-Sil (Cabot Co) (Fumed Silica)
(b) Bentone 34 (National Lead Co.) (dimethyl dioctadecyl ammonium bentonite)
(c) Veegum F (R. T. Vanderbilt Co.) (microfine magnesium aluminum silicate)
(d) Microthene (trade mark) (U.S.I.) (polyethylene powder)
(e) Metal soaps of fatty acids (e.g. powder aluminum stearate, aluminum octoate).

("Cab-O-Sil" and "Bentone" are Trade Marks).

In addition to the aforesaid ingredients, other auxiliary agents well known to those skilled in the art may also be employed in the present composition. These additives include:

1. Antibacterial and antifungal agents such as hexachlorophene, quaternary nitrogen compounds (Benzethonium Chloride), benzoic acid, resorcinol, monoacetate, chlorobutanol, Vancide 89RE, and Zinc Omadine.
2. Fragrance
3. Slip Agents (e.g. talc, zinc stearate)
4. Surfactants (e.g. Arlacel 80—sorbitan monooleate)
5. Pigments (e.g. titanium dioxide)
6. Fabric damage reducing agents (e.g. urea, glycine)
7. Anticorrosion agents: Gafac RM510 (ethoxylated dinonyl phenyl mono and diester of phosphoric acid); Crodafos CAP (propoxylated cetyl mono and diesters of phosphoric acid; Epoxol 8—2B (epoxidized butyl esters of linseed oil fatty acids).

"Arlacel" and "Crodafos" are Trade Marks).

The ingredients described above may be contained in the present composition in various proportions. Ordinarily, however, they will fall within a certain range. Table I below gives the typical and preferred ranges of these materials.

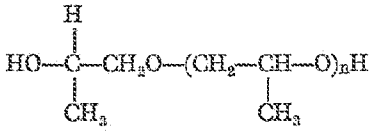
TABLE I

Ingredients	% by Weight General Range	% by Weight Preferred Conc. Range
Aluminum chloride (1—50%)—aluminum salt (99—50%) powder mix	0.2 to 20	0.5 to 8
Suspending Agents	0.01 to 4	0.05 to 1
Non-volatile, organic, oily liquids	2 to 25	3 to 15
Propellants	25 to 95	60 to 95
Auxilliary Agents	q.s.	q.s.

The following Examples are given to further illustrate the present invention. It should be understood, however, that the invention is not limited thereto. Unless otherwise indicated, all parts and percentages in the Examples and elsewhere in the specification are based on weight.

The terms given below used in the Examples or elsewhere have the following meaning:

Cab-O-Sil M-5	: fumed amorphous silica (99% SiO_2)
Freon 114	: 1,2-dichloro-1,1,2,2-tetrafluoroethane
Freon 11	: trichloromonofluoromethane
Freon 11S	: trichloromonofluoromethane stabilized with 0.3% nitromethane
Freon 12	: dichlorodifluoromethane
Neobee M-20	: propylene glycol diester of short chain fatty acids (C_8—C_{10})
Brij 30	: polyoxyethylene (4 moles) lauryl ether
Bentone 34	: dimethyl dioctadecyl ammonium bentonite
Hodag CSA 76	: diisopropyl ester of short chain fatty acid of coco fraction
Fluid AP	: propoxylated monohydric alcohol M.W. 880—930
Polypropylene glycol 2000 monooleate propylene oxide of general formula	: Polymerized



having an average molecular weight of 2000 esterified with one mole of oleic acid.

Acetulan	: acetylated lanolin alcohols; Sp. gr. 25°C. 0.867; Acid No. 0.35; Hydroxyl No. 2.0 Saponification No. 190.0 (See the Merck Index, Eighth Edition, page 9, Merck & Co. Inc., Rahway, New Jersey 1968)

EXAMPLE 1

Preparation of Aluminum Chloride—Aluminum Chlorhydroxide Powder Mix (Spray Dry Method)

Ingredients:	% by Weight
Aluminum Chloride Hexahydrate	10
Aluminum Chlorhydroxide (50% Aq. Solution)	90
	100

Procedure:

1. The aluminum chloride hexahydrate is added to the 50% aluminum chlorhydroxide solution and mixed until clear.

2. Solution (1) is spray dried under the following conditions:

Inlet temperature	150°C—160°C
Outlet temperature	115°C—125°C
Feeding rate	1 gm/min.

3. The spray dried powder mix is screened through a 325 mesh sieve (44 microns).

A suitable spray dryer for this process is the "Nerco-Niro" Portable Spray Dryer of the Nichols Engineering & Research Corporation. However, any of a number of spray dryers well known to those skilled in this art may be employed.

Following the procedure outlined above, other powder mixes can be prepared containing Al/Cl molar ratios as follows:

	Ex. 1A	Ex. 1B	Ex. 1C
Molar Ratio Al/Cl	1 : 1	1.5 : 1	1.79 : 1
% by Weight Aluminum Chloride Hexahydrate	21.21	7.24	2.56
% by Weight Aluminum Chlorohydroxide (50% Aq. Solution)	78.79	92.76	97.44

EXAMPLE 2

Preparation of Mix of Aluminum Chloride—Aluminum Chlorhydroxide (Dry Mix Method)

The aluminum antiperspirant mixes are also prepared by mixing aluminum chloride hexahydrate powder and aluminum chlorhydroxide powder. This mix is pulverized (a jet mill or similar equipment well known in the art may be used) to a fine powder which passes 100% through a 270 mesh sieve (53 microns) and 99% through a 325 mesh (44 microns) sieve.

Aluminum Chloride Hexahydrate/ Aluminum Chlorhydroxide	% by Weight Aluminum	% by Weight Chloride	Molar Ratio Al : Cl
50 parts/50 parts	17.91	30.13	0.78 : 1
25 parts/75 parts	21.28	23.15	1.21 : 1
5 parts/95 parts	23.97	17.58	1.79 : 1
1 part /99 parts	24.50	16.47	1.95 : 1

These calculations are based on aluminum chloride hexahydrate and aluminum chlorhydroxide ($Al_2(OH)_5Cl.2 1/2H_2O$).

EXAMPLE 3

The following components are used to prepare an aerosol antiperspirant powder suspension:

Ingredients	% by Weight
Aluminum Chloride—Aluminum Chlorhydroxide Powder (35/65%) (Al : Cl=1 : 1)	3.5
Isopropyl Myristate	6.0
Cab-O-Sil M-5	0.5
Hexachlorophene	0.2
Perfume	0.2
Freon 114	64.6
Freon 12	25.0
	100.0

The aluminum chloride-aluminum chlorhydroxide powder, fumed silica, perfume, hexachlorophene are dispersed in the isopropyl myristate with the aid of Lightnin' Mixer. The suspension is then homogenized to form a concentrate. The concentrate is weighed into an aerosol can, the valve is inserted and the can charged with the propellant mix. If desired, the perfume, may be omitted and replaced by an equal amount of Freon 114.

Conventional filling operations, namely, pressure filling, cold filling, under-cap filling methods can be used in this formulation or formulations described herein.

Using the same procedure outlined above for preparing the concentrate, the compositions of the following Examples are prepared:

EXAMPLE 4

Ingredients	% by Weight
Aluminum Chloride-Aluminum Chlothydroxide Powder (15/85%) (Al : Cl=1.5 : 1)	5.00
Neobee M 20	4.55
Brij 30	2.00
Cab-O-Sil M-5	0.45
Perfume	0.30
Freon 11-S	47.70
Freon 11-S	47.70
Freon 12	40.00
	100.00

EXAMPLE 5

Ingredients	% by Weight
Aluminum Chloride-Aluminum Chlorhydroxide Powder (15/85%) (Al : Cl=1.5 : 1)	5.0
Bentone 34	0.9
Hodag CSA 76	5.1
Fluid AP	1.5
Perfume	0.2
Freon 11	47.3
Freon 12	40.0
	100.0

Ingredients	EXAMPLE 6 % by Weight	EXAMPLE 7 % by Weight
Aluminum Chloride-Aluminum Chlorhydroxide Powder (5/95%) (Al : Cl=1.79 : 1)	5.0	10.0
Cab-O-Sil M-5	0.5	0.5
Bentone 34	——	0.5
Polypropylene glycol 2000 monooleate	2.0	——
Isopropyl Myristate	5.0	5.0
Isopropyl Palmitate	——	10.0
Freon 11	62.5	47.6
Freon 12	25.0	25.0
Perfume	——	0.4
Benzoic Acid	——	1.0
	100.0	100.0

Ingredients	EXAMPLE 8	EXAMPLE 9	EXAMPLE 10	EXAMPLE 11
	% by Weight			
Aluminum Chloride-Aluminum Chlorhydroxide Powder (15/85%) (Al : Cl=1.5 : 1)	1.0	4.0	7.0	3.5
Bentone 34	——	——	——	0.23
Hexachlorophene	0.1	——	0.3	——
Cab-O-Sil M-5	0.3	0.4	0.5	0.23
Isopropyl Palmitate	5.0	3.0	——	7.00
Hexadecyl Alcohol	——	3.0	——	——
Talc	——	1.0	——	——
Perfume	——	——	——	2
Diisopropyl Sebacate	——	——	9.0	——
Acetulan	——	1.0	——	——
Dimethyl Polysiloxane	——	——	0.3	——
Freon 11S	——	——	——	58.84
Freon 11	68.6	62.6	52.9	——
Freon 12	25.0	25.0	30.0	30.0
	100.0	100.0	100.0	100.0

Ingredients	EXAMPLE 12	EXAMPLE 13	EXAMPLE 14
	% by Weight		
Rehydrol/Aluminum Chloride Hexahydrate (90/10)	3.5	——	——
Wickenol 363D/Aluminum Chloride Hexahydrate (90/10)	——	3.5	——
Aluminum Sulfate . $18H_2O$/Aluminum Chloride Hexahydrate (90/10)	——	——	3.5
Cab-O-Sil M-5	0.5	0.5	0.5
Isopropyl Myristate	6.0	6.0	6.0
Freon 11	65.0	65.0	65.0
Freon 12	25.0	25.0	25.0
	100.0	100.0	100.0

To compare the relative effectiveness of the compositions of this invention with a commercial aerosol antiperspirant preparation having corresponding amounts of active antiperspirant material (i.e. 3.5% by weight of aluminum chlorhydroxide), this commercial product and the product of Example 11 of this application (containing 3.5% by weight of aluminum chlorhydroxide mix) were subjected to the following tests:

Each antiperspirant preparation was applied to one axilla of each of 11 female subjects in a standard cross-over design. The materials were applied seven times over a period of five days. Perspiration was collected (in absorbent pads placed in each axilla) seven days after the last application except for the final treatment week. The amount of material perspired was measured by weighing the pads.

After making suitable corrections for control values, the percent of inhibition of axillary sweat was calculated for each case. Thirty-two percent (32%) inhibition was observed for the 3.5% aluminum chlorhydroxide commercial preparation. In contrast to this 52% inhibition was observed with the corresponding composition of this invention.

To test the corrosion characteristics of composition of this invention an antiperspirant composition containing aluminum chloride and aluminum chlorhydroxide mix (18%/82%) with a molar ratio of Al/Cl=1.38:1 was subjected to elevated temperature storage (two months at 125°F and six months at 104°F) in a tin plate can with a vinyl/epoxy coating. This composition did not produce any corrosion other than some slight detinning, and the vinyl/epoxy coating remained intact.

WHAT WE CLAIM IS:—

1. An anhydrous aerosol antiperspirant composition comprising a powdered aluminum antiperspirant mix distributed in a liquid vehicle comprising a non-volatile, organic, oily liquid having a boiling point under atmospheric pressure not lower than 120°C and a specific gravity of 0.7 to 1.6, and an aerosol propellant, said powdered aluminum antiperspirant mix comprising aluminum chloride and a water soluble antiperspirant aluminum salt less acidic than aluminum chloride.

2. A composition according to Claim 1 including a suspending agent.

3. A composition according to Claim 2 containing the following ingredients in the percentages by weight indicated:

(a) powdered aluminum antiperspirant mix	0.2 to 20%
(b) organic oily liquid	2 to 25%
(c) propellant	25 to 95%
(d) suspending agent	.01 to 4 %

4. A composition according to Claim 2 or Claim 3 wherein said suspending agent is fumed silica.

5. A composition according to any one of the preceding claims wherein said organic, oily liquid has a specific gravity of from 0.7 to 1.2.

6. A composition according to any one of the preceding claims wherein the said powdered aluminum antiperspirant mix contains from 1 to 50% by weight of said aluminum chloride and from 99 to 50% by weight of said water soluble salt.

7. A composition according to any one of the preceding claims wherein said antiperspirant mix comprises aluminum chloride or its hydrates and aluminum chlorhydroxide or its hydrates.

8. A composition according to Claim 7 wherein the molar ratio of aluminum to chloride in said aluminum antiperspirant mix is in the range of from 0.78:1 to 1.95:1.

9. A composition according to Claim 8 in which the aluminum to chloride molar ratio is in the range of from 1.33:1 to 1.79:1.

10. A composition according to any one of the preceding claims including glycine.

11. A composition according to any one of the preceding claims wherein the organic oily liquid is a liquid hydrocarbon, a fatty acid ester, a diester of a dicarboxylic acid, a polyoxyalkylene glycol ester, a propylene glycol diester of a short chain fatty acid (C_8—C_{10}), a polyoxyethylene ether, a polyoxypropylene cetylether, a fatty alcohol, a propoxylated monohydric alcohol (molecular weight 880—930) a silicone oil or a mixture of two or more thereof.

12. A composition according to Claim 11 wherein the organic, oily liquid is isopropyl myristate.

13. An aerosol antiperspirant composition substantially as hereinbefore described with reference to any of Examples 3 to 14.

14. A process for preparing a powdered aluminium antiperspirant mix which comprises forming an aqueous solution containing aluminum chloride and a water soluble antiperspirant aluminum salt less acidic than aluminum chloride, and drying the solution to obtain a dry product.

15. A process according to Claim 14 wherein said aluminum salt, less acidic than said aluminum chloride is an aluminum chlorhydroxide.

16. A process according to Claim 14 or 15 including the step of pulverizing said dry product obtained to produce a powder suitable for suspension in an aerosol container.

17. A process according to any one of Claims 14, 15, and 16 wherein the dry product has a particle size of not more than 74 microns.

18. A process according to Claim 17 wherein the dry product has a particle size of not more than 53 microns.

19. A process for preparing a powdered aluminum antiperspirant mix comprising aluminum chloride and a water soluble antiperspirant aluminum salt less acidic than alum-

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inum chloride, substantially as hereinbefore described with reference to Example 1 or Example 2.

20. A powdered aluminum antiperspirant mix when made by the process of any one of Claims 14 to 19.

21. An aerosol antiperspirant composition according to Claim 1 including a powdered de-aluminum antiperspirant mix as claimed in Claim 21.

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(54) **Aluminium Chlorhydroxide and Preparation Thereof**

(57) A novel aluminium chlorhydroxide comprises at least 45% by weight of an Al^{3+} group of aluminium chlorhydroxide complexes which are characterised by exhibiting anomalous permeation and reaction rates in gel permeation chromatography and ferron tests. This aluminium chlorhydroxide is prepared by ageing aluminium chlorhydroxide in an aqueous medium.

The product is used in anti-perspirant compositions.

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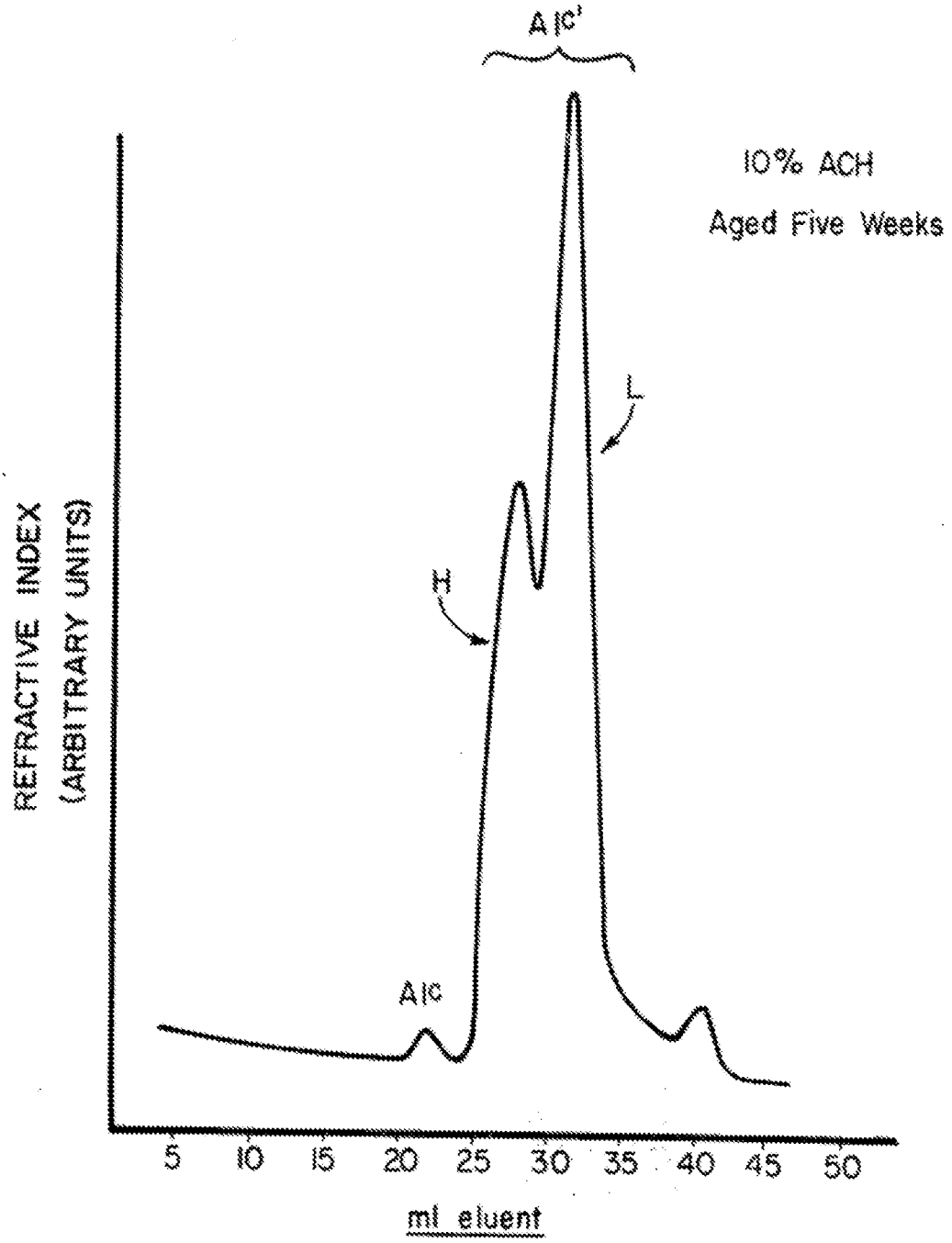

AIc'
10% ACH
Aged Five Weeks
L
H
AIc
REFRACTIVE INDEX
(ARBITRARY UNITS)
5
10
15
20
25
30
35
40
45
50
ml eluent

Fig. 1

SPECIFICATION

Aluminium Chlorhydroxide and Preparation Thereof

Aluminium chlorhydroxides have long been used as antiperspirants. In general, such chlorhydroxides are, in the presence of water, complexes made up of mixtures of polymeric species of various sizes and molecular structures. The aluminium chlorhydroxides which have been found most useful as antiperspirants may be represented by the empirical formula:

$$Al_2(OH)_5Cl$$

In the past, aluminium hydroxyl polymers which are related to such aluminium chlorhydroxides have been shown to be made up of three broad basic groups by spectrophotometrically following their complexing rates with 8-hydroxy-7-iodo-5-quinoline-sulphonic acid (ferron). Such basic groups have been referred to as Al^a, Al^b and Al^c. The first group, Al^a, has the fastest complexing rate (almost instantaneous); the Al^b group has an intermediate complexing rate (the reaction is complete in about an hour); and the Al^c group has the slowest rate (generally the reaction takes a matter of days). It is known that aluminium chlorhydroxides exhibit a similar phenomenon when reacted with ferron. For convenience, the fastest reacting group within the aluminium chlorhydroxide complexes may be referred to as Al^a, the intermediate reacting group as Al^b, and the slowest group as Al^c. It is also known that when aqueous solutions of aluminium chlorhydroxides are subjected to gel permeation chromatography, the three groups exhibit different retention times. The Al^a group has the longest retention time which is indicative that it is made up of the lowest molecular size materials, the Al^b group has an intermediate retention time, indicating it comprises complexes of intermediate molecular size, and the Al^c group has the shortest retention time indicating that it is made up of the highest molecular size complexes.

We have now found that there is a further group of complexes within the aluminium chlorhydroxides which has not previously been identified or recognized and which is more efficacious as an antiperspirant than the known groups referred to above. This more efficacious group of complexes, which for convenience may be referred to as $Al^{a'}$, is present in amounts of from 10 to 30% by weight in aluminium chlorhydroxides presently available, although its presence has not been recognized or appreciated up to now. We have further found that aluminium chlorhydroxides can be modified or produced so as to contain a substantially higher amount of the $Al^{a'}$ group and the chlorhydroxides so modified or produced are novel compositions of matter. Generally, the aluminium chlorhydroxides of this invention are distinguished from known aluminium chlorhydroxides in that they contain at least 45% by weight, preferably 60% and more preferably at least 70%, of the $Al^{a'}$ group of complexes.

Even with the advanced analytical techniques available today, it is still not possible to determine the structure of aluminium chlorhydroxide complexes. Accordingly, in defining the newly discovered $Al^{a'}$ group of complexes, it is necessary to resort to empirical procedures. Broadly, the new $Al^{a'}$ complex can be defined by its diffusion constant in gel permeation chromatography and its complexing rate in the ferron test. Using such criteria, the $Al^{a'}$ group of complexes can be broadly characterised by having a diffusion constant in gel permeation chromatography which is within that range generally found for the Al^b group of complexes and a complexing rate in the ferron test in the range found for the Al^a complexes. Such anomalous results clearly indicate that the complexes within the $Al^{a'}$ group have a distinct structure not heretofore recognized or appreciated.

According to the present invention, therefore, there is provided an aluminium chlorhydroxide comprising at least 45% by weight, preferably at least 60% by weight, and more preferably at least 70% by weight, of an $Al^{a'}$ group of aluminium chlorhydroxide complexes which are characterised by exhibiting anomalous permeation and reaction rates in gel permeation chromatography and ferron tests.

The term "$Al^{a'}$" will hereinafter be used to refer to that group of aluminium chlorhydroxide complexes which are more efficacious as antiperspirants and exhibit anomalous results in gel permeation chromatography and the ferron reaction.

Generally, the diffusion rates in gel permeation chromatography and the reaction rates in the ferron complexing test will vary with the conditions under which such procedures are conducted. Accordingly, in order to more specifically define the $Al^{a'}$ group of complexes, it is desirable to specify the conditions under which such procedures are conducted.

Generally, the $Al^{a'}$ group of complexes can be said to have a diffusion constant in gel permeation chromatogrsphy of from 0.1 to 0.7, preferably from 0.2 to 0.65, and more preferably from 0.3 to 0.55 when such permeation is carried out under the following conditions:

Eluent—0.1N KCl
Concentration of aluminium chlorhydroxide—10% by weight
Temperature—Room Temperature
Type of gel material—Em Gel PGM-2000 (Trade name of E. Merck Co. for a polyethylene glycol dimethacrylate type gel.)

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In the ferron test, the Al^{cr} group of complexes requires a minimum of about 3 days for the reaction to go to completion under the following conditions:

Reaction medium—Water
Conc. of ferron—2.8×10^{-3} M
Conc. of aluminium chlorhydroxide—2.3×10^{-4} M
Temperature—Room Temperature
pH—5
Buffer—Hydroxylamine hydrochloride, hydrochloric acid and sodium acetate.

At least in aluminium chlorhydroxides which have a pH of 4.0 to 4.7, for example $Al_2(OH)_5Cl$, the Al^{cr} group of complexes can be further characterised by the fact that in gel permeation chromatography (using an Em Gel) they produce a doublet within the peak in which they fall (for example, between diffusion constants 0.3 to 0.55). The single Figure of the accompanying drawing is a chromatogram of an aluminium chlorhydroxide within the scope of the present invention which was run using an Em Gel PGM-2000. The two peaks of the doublet have been found to be distinct fractions and the first peak, presumably comprising the larger molecules of the two fractions, has been called H and the second L. Such fractions may also be referred to as Al_H^{cr} and Al_L^{cr}.

The Al^{cr} group of complexes of this invention can be still further characterized by the fact that the molecules making up the group are less than 100Å in size in aqueous solutions.

For the purpose of this invention, the percent of the Al^{cr} group in an aluminium chlorhydroxide is determined by assuming that the Al^a group of complexes plus the Al^{cr} group of complexes is about 90% by weight of the aluminium chlorhydroxide and then multiplying 90% by the ratio of the height of the Al^{cr} peak to the combined heights of the Al^a and Al^{cr} peaks on a gel chromatogram such as shown in the accompanying drawing, i.e.

$$\%Al^{cr} = \frac{Al^{cr}}{Al^a + Al^{cr}} \times 90\%$$

The novel aluminium chlorhydroxide of this invention may be prepared by ageing the aluminium chlorhydroxide presently available (that is, those containing less than 30% of the Al^{cr} group) in an aqueous medium until the aluminium chlorhydroxide contains at least 45% of the Al^{cr} group as determined by the peak height ratio. In carrying out the ageing process, it has generally been found that the reaction is temperature, concentration and time dependent. In general, the ageing should be carried out at temperatures below 100°C, preferably at 90°C or lower and more preferably at about 80°C or lower. Generally there is no lower limit on the ageing temperature, but when the ageing is carried out at room temperature or lower the reaction rate is considerably slower, for example a 10% by weight solution at room temperature takes about five weeks. It is preferable that the ageing be carried out at a temperature above 50°C. As to the concentration, it has generally been found that the concentration should be at least 5% by weight, preferably at least 7.5% and more preferably 10% or more. As to the upper limit on concentration, it is generally less than 40% by weight, preferably less than 35% by weight and more preferably less than 30%. At between 27.5 and 7.5% and more particularly between 25% and 10% especially good results were obtained. Within the temperature and concentration ranges specified above, the reaction rate of the ageing process will generally increase as the temperature is raised and decrease when the concentration is increased. The effect of concentration and temperature on the reaction is illustrated in Table I below:

Table I

Time Required to Produce Aluminium Chlorhydroxides (ACH) which contain at least 70% Al^{cr}

	Temperature °C		
ACH Concentration	*50°*	*65°*	*80°*
10%	1 week	1 day	8 hours
15%	———	4 days	16 hours
25%	———	3 weeks	1 week

Generally the conditions which are most conducive for producing the aluminium chlorhydroxides of this invention will promote the depolymerization of the larger size polymeric molecules and the overall composition of the resulting product in water will generally be substantially free, i.e. contain less than 2% by weight, of molecules greater than 100Å in size.

After the aluminium chlorhydroxides of this invention have been produced by the ageing process described above, the reaction solution may be concentrated by distillation. Such distillation is preferably carried out at a temperature below 100°C and more preferably below about 80°C and at reduced pressure, so as not to convert the aluminium chlorhydroxide to aluminium oxide. Particularly good results are obtained by carrying out the concentration step at 34°C to 38°C and 28 mmHg pressure. As the concentration of aluminium chlorhydroxides increases by removal of water during the

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distillation, the concentration of the Al^{cr} group is believed to start to gradually decrease as the overall concentration of the aluminium chlorhydroxide begins to exceed about 30%. Thus, although it is possible to produce 50% solutions by distillation, such solutions are not stable for extended periods and the amount of the Al^{cr} present will gradually decrease until the amount present is below that specified for the aluminium chlorhydroxide of this invention. In light of such instability at higher concentrations, it has been found that if the aluminium chlorhydroxides are going to be used in water-based antiperspirant compositions, they should be concentrated to a range of from 10% to 30% in which they will generally remain stable. If it is desired to produce the aluminium chlorhydroxide as a powder, the reaction solutions may be concentrated, for example up to about 50%, and they should then be converted to the powder shortly thereafter, for example within 24 hours, under conditions in which the decrease of the Al^{cr} group does not take place to any great extent. One such method is to rapidly convert the concentrated solution to a powder, for example by spray drying. Spray drying has been successfully carried out at an inlet temperature of 150°C and an outlet temperature of 75 to 80° and a feed rate of 280 ml per minute. Another method is to convert the concentrated solution to a powder under conditions in which the decrease of the Al^{cr} group will take place very gradually, for example freeze drying. Freeze drying has been successfully carried out at —40°C and a pressure of 1 mm Hg. In drying the aqueous solution to powder, care should be taken not to remove all the bound water because as is known such total removal generally irreversibly destroys the antiperspirant activity of the material. The dried powder should preferably contain 3 to 20% by weight and more preferably 3 to 15% by weight of bound water. In powdered form the aluminium chlorhydroxides of this invention are quite stable.

The aluminium chlorhydroxides of this invention may be used as antiperspirants in the various forms well known to the art, for example solutions, creams, lotions, sticks, roll-ons, pads and aerosols. When they are used in water-based compositions or in water-alcohol based compositions, the amount of water present should preferably be such that the concentration of the aluminium chlorhydroxide in the water alone, that is exclusive of all other liquids present, is from 5 to 30% by weight, in which range the Al^{cr} group is generally stable. The especially preferred concentration is from 7.5% to 27.5%.

When the aluminium chlorhydroxides of this invention are to be used as powders in substantially anhydrous systems, it will not usually be necessary to use any special precautions because as pointed out above, they are quite stable in this form. The aluminium chlorhydroxides of this invention are especially useful in anhydrous suspension-type systems well known to the art, such as roll-ons and pump and aerosol sprays, in which the powders are suspended in hydrophobic vehicles, such as isopropyl myristate, volatile cyclic silicones, and mixtures thereof. Such systems usually also comprise a suspending agent, such as a hydrophobic bentonite, for example Bentone 34 and 38, and silica, for example Cab-O-Sil M5. When the suspensions are used in aerosol form they will also include well known propellants, such as the hydrocarbons and the halocarbons.

Unlike the presently available aluminium chlorhydroxides, the efficacy of the aluminium chlorhydroxides of this invention is concentration dependent. This can be quite advantageous in that, within limits, one may formulate antiperspirant compositions to provide a desired level of efficacy by merely varying the amount of active material present. In aqueous systems, the improved efficacy of the aluminium chlorhydroxides of this invention generally become more apparent and significant when they are used in amounts above 15%, for example 20% by weight. In such aqueous systems at concentrations of 20% or more the aluminium chlorhydroxides of this invention have been found to be at least 20% more efficacious than the aluminium chlorhydroxides presently available.

In order that the invention may be more fully understood, the following examples are given by way of illustration only.

Example I

A 10% aqueous solution of aluminium chlorhydroxide was aged at 50°C for five weeks. The resulting aluminium chlorhydroxide composition which contained about 90% of the Al^{cr} group was freeze dried to a powder in a laboratory freeze drier at about 5 mm Hg. The powder (ACH'), together with a control of standard aluminium chlorhydroxide (ACH), was formulated into water-based antiperspirant compositions containing 10, 15 and 20% by weight, respectively, of the aluminium chlorhydroxides. The resulting compositions were tested for sweat reduction using a trade-recognized gravimetric procedure on thermally stimulated human subjects. The results are set forth in Table II below:

Table II

Relative Percent Sweat Reduction—90% Confidence Levels After Six Applications*

	Relative Percent Sweat Reduction	
Concentration	*ACH*	*ACH'*
10%	36.7	40.5
15%	37.8	41.8
20%	39.3	48.1

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As can be noted, especially at the 20% level, the aluminium chlorhydroxides of this invention are significantly superior to the presently available aluminium chlorhydroxides.

* A number of applications of the product which the trade would recognize as being reflective of the typical efficacy of the antiperspirant.

Example II

300 gallons of a 10% by weight aqueous solution of a commercially available aluminium chlorhydroxide were aged at 80°C for 16 hours. At the end of the ageing period, a small aliquot of the composition was subjected to gel permeation chromotography. The chromotogram indicated that the aluminium chlorhydroxide present contained at least 90% of the Al^{IV} group. About 10 Kg of the composition was freeze dried at −40°C and 1 mm Hg to provide about 1 Kg of powdered material. A chromatogram of a 10% aqueous solution of the freeze dried material was identical to that of the mother solution thus indicating that the aluminium chlorhydroxide of the invention is stable to freeze drying.

Example III

The aluminium chlorhydroxide powder produced in Example II (ACH') was ground and sieved, using a 230 mesh sieve, and were formulated into a suspension-type, substantially anhydrous, roll-on antiperspirant composition, together with a standard powdered ACH (control). The make-up of the formulations was as follows:

Ingredients	*Formulations (Percent by Weight)* ACH'	ACH (Control)
ACH'	20.0	—
ACH	—	20.0
Volatile cyclic silicone	74.0	74.0
Bentone 38 (a hydrophobic quaternized bentonite clay)	3.50	3.50
Alcohol (200 proof)	2.31	2.31
Deionized water	0.19	0.19
	100%	100%

The resulting compositions were tested for sweat reduction using a trade-recognized gravimetric procedure on thermally stimulated human subjects. The ACH' composition was found to be significantly superior to the ACH (control) composition in reducing sweat.

Example IV

A 10% aqueous solution of a commercially available aluminium chlorhydroxide was aged as described in Example II and concentrated to about a 50% solution at a temperature of from 34°C to 38°C and a pressure of 28 mm Hg. The concentrated solution was spray dried at an inlet temperature of about 150°C, an outlet temperature of 75° to 80°C and a feed rate of about 280 ml per minute. When the resulting material was tested as an antiperspirant in a suspension-type composition similar to that of Example III, except that 25% of the material was used, it gave comparable results.

In the above Examples, the aluminium chlorhydroxide used had an empirical formula of about $Al_2(OH)_5Cl$.

Claims

1. An aluminium chlorhydroxide comprising at least 45% by weight of an Al^{IV} group of aluminium chlorhydroxide complexes which are characterised by exhibiting anomalous permeation and reaction rates in gel permeation chromatography and ferron tests.

2. An aluminium chlorhydroxide according to claim 1, which contains at least 60% by weight of the Al^{IV} group of complexes.

3. An aluminium chlorhydroxide according to claim 1, which contains at least 70% by weight of the Al^{IV} group of complexes.

4. An aluminium chlorhydroxide according to any of claims 1 to 3, which is further characterised by forming a doublet in the chromatogram when it is subjected to gel permeation chromatography using a polyethylene glycol dimethacrylate gel.

5. An antiperspirant composition comprising an aluminium chlorhydroxide according to any of claims 1 to 4.

6. An antiperspirant composition according to claim 5, which is water-based and in which the concentration of the aluminium chlorhydroxide is from 5% to 30% by weight, based on the water content thereof.

7. An antiperspirant composition according to claim 6, in which the concentration is from 7.5% to 27.5% by weight.

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8. An antiperspirant composition according to claim 5 which is substantially anhydrous and in which the aluminium chlorhydroxide is in powder form.

9. A process for producing an aluminium chlorhydroxide according to any of claims 1 to 4, which comprises ageing aluminium chlorhydroxide in an aqueous medium.

10. A process according to claim 9, in which the ageing is carried out at a concentration of from 5% to 40% by weight and at a temperature of from 50°C to 100°C.

11. A process according to claim 10, in which the concentration is from 5% to 30% by weight.

12. A process according to claim 10, in which the concentration is from 7.5% to 27.5% by weight.

13. A process according to any of claims 9 to 12, in which ageing is carried out at a temperature of from 50°C to 90°C.

14. A process according to any of claims 9 to 13, in which ageing is carried out at a temperature of from 50°C to 80°C.

15. A process according to any of claims 9 to 14, in which the resulting aqueous composition is spray dried to a powder.

16. A process according to any of claims 9 to 14, in which the resulting aqueous composition is freeze-dried to a powder.

17. An aluminium chlorhydroxide according to claim 1 substantially as herein described in any of the Examples.

18. An antiperspirant composition according to claim 5 substantially as hereindescribed in any of Examples I, III and IV.

19. A process for producing an aluminium chlorhydroxide according to claim 1 substantially as herein described in any of Examples I, II and IV.

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A5B

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(54) Antiperspirants

(57) A composition of zirconyl hydroxy chloride and aluminium chlorhydroxide in stable solid form having improved antiperspirant efficacy when dissolved in water is made by heating a 2–20% by weight aqueous solution of the composition at a temperature of at least 50°C until the ratio of the height of peak 4 to that of peak 3 as measured by gel permeation chromatography on cross-linked dextran exceeds 2:1, then subjecting the solution to rapid drying to solid form.

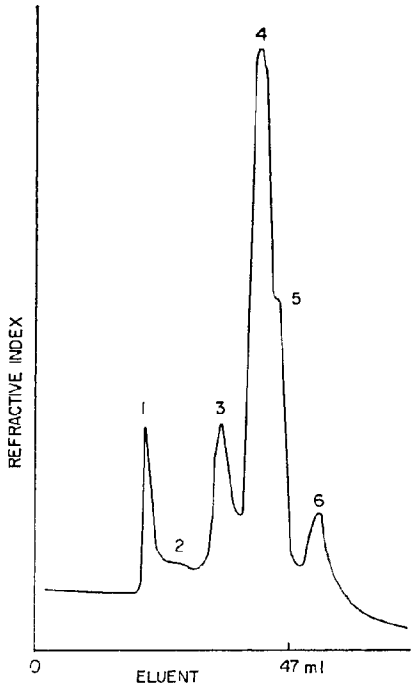


FIG 2

GB 2 144 992 A

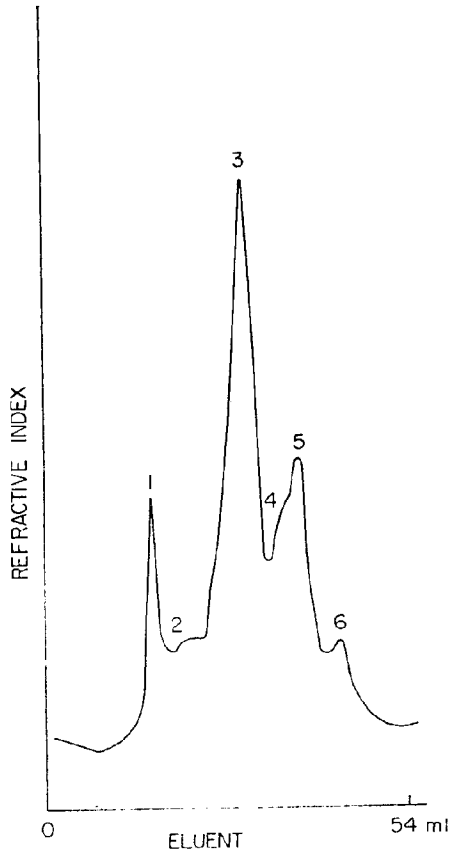

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1
2
3
4
5
6
0
ELUENT
54 ml

FIG 1

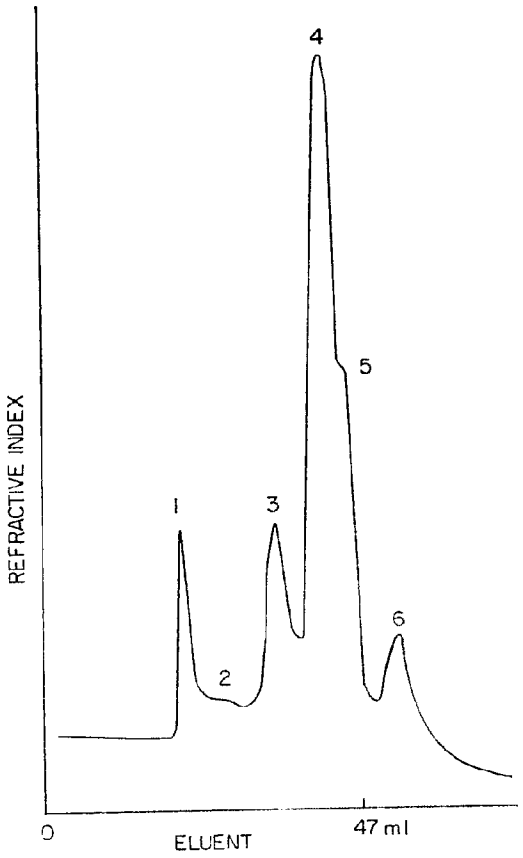

REFRACTIVE INDEX
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2
3
4
5
6
0
ELUENT
47 ml

FIG 2

FIG 3

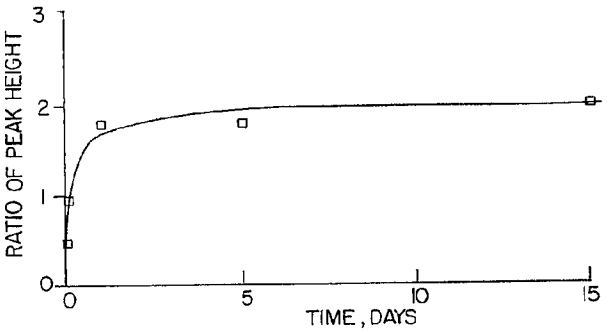


FIG 4

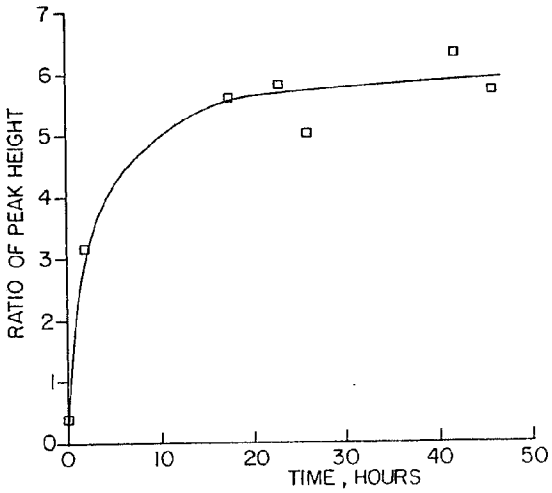
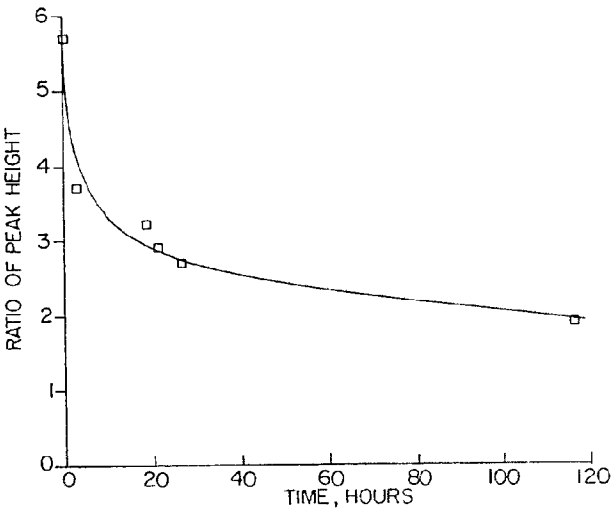


FIG 5



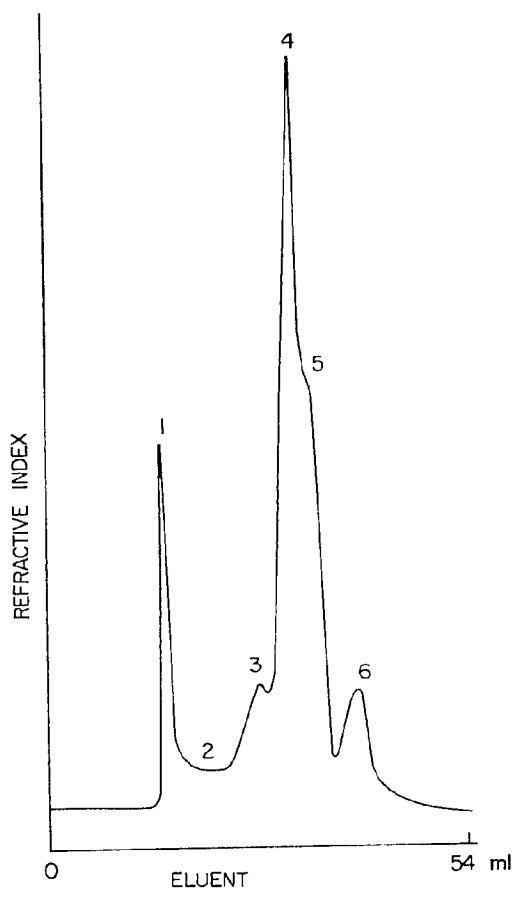

REFRACTIVE INDEX
1
2
3
4
5
6
0
ELUENT
54 ml

FIG 6A

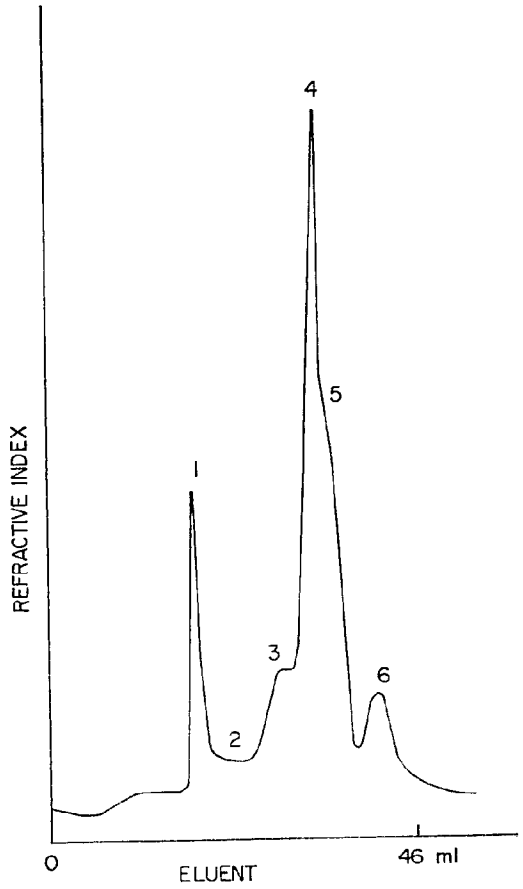

REFRACTIVE INDEX
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2
3
4
5
6
0
ELUENT
46 ml

FIG 6B

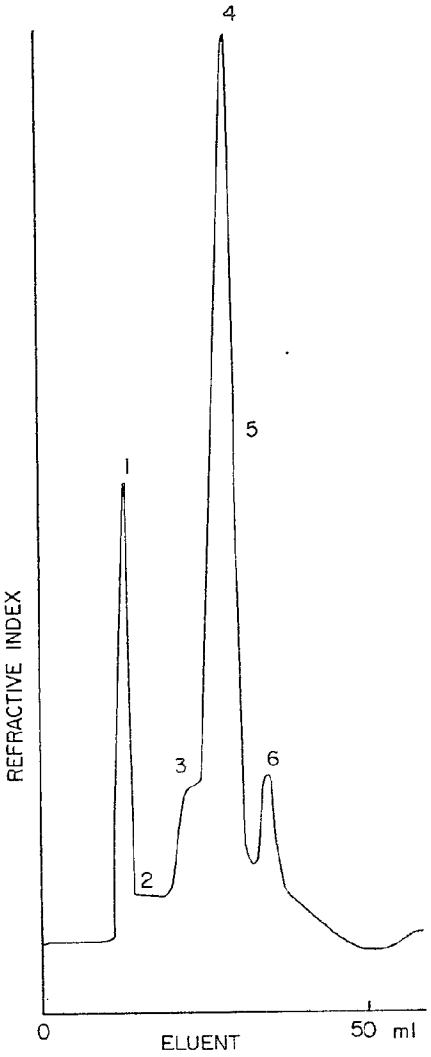

4
5
1
3
6
2
REFRACTIVE INDEX
0
ELUENT
50 ml

FIG 7A

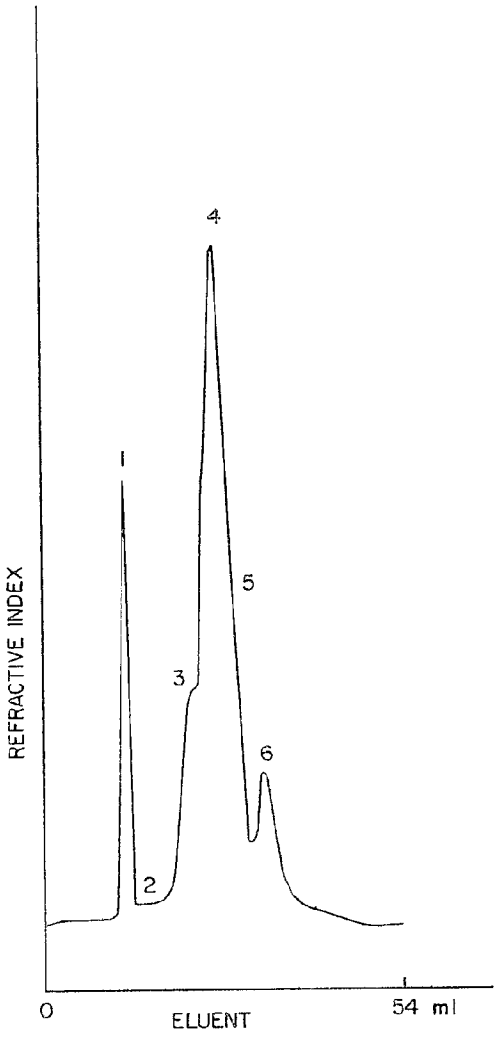

4
1
5
3
6
2
REFRACTIVE INDEX
0
ELUENT
54 ml

FIG 7B

SPECIFICATION

Antiperspirant compositions

This invention is concerned with antiperspirant compositions comprising zirconyl hydroxy chloride and aluminium chlorhydroxide and with their preparation.

It has previously been proposed to increase the antiperspirant effectiveness of aluminium chlorhydroxide by aging under specified conditions an aqueous solution containing the aluminium chlorhydroxide, as described in U.S. Patent 4,359,456 and in British Patent Application 2,048,229A, until the solution exhibits specified characteristics. In the British application, it is pointed out that the increased activity is the result of a group of complexes called $Al^{c'}$ characterized by having a diffusion constant in gel permeation chromatography which is within the range generally found for the Al^{b} group of complexes, but which displays a complexing rate in the ferron test which is in the range of the Al^{c} complexes and by having molecules which are less than 100 A in size in aqueous solution. The $Al^{c'}$ complexes thus prepared are stable in aqueous solution at concentrations in the range 10% to 30%.

It has now been found that a composition comprising zirconyl hydroxy chloride and aluminium chlorhydroxide, unlike aluminium chlorhydroxide alone, does not form a stable aqueous solution when heated, but that antiperspirant effectiveness is formed by heating such a solution. The novel complex, of lower molecular weight than $Al^{c'}$, contains no zirconium, and reverts rapidly in aqueous solution at room temperature to its initial composition, losing approximately 1/2 of its initial concentration in 48 hours. The composition containing the novel complex as described herein displays improved antiperspirant efficacy as compared to an aqueous solution made by dissolving conventional zirconyl hydroxy chloride and aluminium chlorhydroxide in water or as compared to conventional commercially available aqueous solutions containing the zirconyl and aluminium compounds. The novel complex is formed, at least in part, from the higher molecular weight $Al^{c'}$ complex and is segregated in a separate zone from $Al^{c'}$ when the aqueous solution is subjected to gel permeation chromatography. When the aqueous solution containing the composition is subjected to heating, the $Al^{c'}$ complex initially present in the solution gradually decreases in amount, while at the same time the amount of the novel complex present gradually increases. An aqueous solution containing the novel complex of the present invention in large amount is unstable at room temperature, as stated above, and the amount of the novel complex present in such a solution decreases quite rapidly, while the amount of $Al^{c'}$ increases. Because of this inherent instability, solutions of zirconyl hydroxy chloride and aluminium chlorhydroxide containing the novel complex of the present invention cannot be used as such as a commercially practical antiperspirant. However, it has been found that if the aqueous solution containing the novel complex is rapidly solidified by removal of water, the solid product retains in large part the desired complex and its high antiperspirant effectiveness. The solid product consequently can be employed as an antiperspirant in finely divided form, for example as a powder either alone or with a diluent powder vehicle, such as talc, or in the form of a cream or suspension in a non-aqueous liquid vehicle, for example a high viscosity cream for manual application, a low viscosity suspension for roll-on application, or a suspension in a liquefied propellant for aerosol application. Any conventional non-aqueous physiological and cosmetically acceptable non-toxic vehicle can be used with the particulate solid composition of the present invention to form an antiperspirant composition. Particle size may vary over a wide range, but is preferably from 1 to 100 micrometers and particulate solid are not critical. In general, the weight of particulate solid may range from 0.1 to 80% or more of the total weight of the composition including vehicle. After the finely divided solid comes into contact with the skin, it dissolves in the initial perspiration present on the skin and becomes effective as an antiperspirant.

According to one aspect of the present invention, therefore, there is provided a stable solid composition comprising zirconyl hydroxy chloride and aluminium chlorhydroxide having improved antiperspirant efficacy, said composition, when dissolved in water to form a 10% by weight solution and, after storage at room temperature for no more than 2 hours, subjected to gel permeation chromatography on cross-linked dextran having a molecular weight exclusion range of 1000 to 30,000 for globular proteins (Sephadex G-50), exhibiting a distribution pattern having at least four successive peaks in which the ratio of the height of peak 4 to that of peak 3 is at least 2:1.

According to a further aspect of the invention, there is provided a method of increasing the antiperspirant efficacy of a composition comprising zirconyl hydroxy chloride and aluminium chlorhydroxide, which comprises providing a 2% to 20% solution of such composition in water, heating the solution at a temperature of at least 50°C until the ratio of the height of peak 4 to that of peak 3 as measured by gel permeation chromatography of a 10% aqueous solution on Sephadex G-50 exceeds 2:1, then subjecting the solution to rapid drying to solid form, said solid, when dissolved in water to form a 10% by weight solution and subjected to gel

permeation chromatography on Sephadex G-50 after storage at room temperature for no more than 2 hours, exhibiting a distribution pattern having at least 4 successive peaks in which the ratio of the height of peak 4 to that of peak 3 is at least 2:1.

In the following description, reference will be made to the accompanying drawings, in which:

Figure 1 represents a chromatogram of a freshly prepared 10% by weight aqueous solution of zirconyl hydroxy chloride and aluminium chlorhydroxide;

Figure 2 represents a chromatogram of the solution of Fig. 1 after heating at 100°C for 2 hours;

Figure 3 is a plot showing the ratio of the heights of peak 4 to peak 3 of chromotograms of a 10% aqueous solution of the aluminium and zirconium compounds heated for varying time periods at 50°C;

Figure 4 is a plot showing the ratio of the heights of peak 4 to peak 3 of chromatograms of the solution of Fig. 1 when heated at reflux for varying time periods;

Figure 5 is a plot showing the ratio of heights of peak 4 to peak 3 during room temperature storage of the aluminium and zirconium compounds after previously heating at 100°C for 46 hours;

Figure 6a shows the chromatogram of a 10% aqueous solution of a mixture of zirconium and aluminium compounds of the present invention after heating at 100°C for 20 hours;

Figure 6b is a chromatogram of a 10% aqueous solution of the solid made by spray-drying the solution of Fig. 6a;

Figure 7a is a chromatogram of a 10% aqueous solution of the zirconium and aluminium compounds of the present invention after heating at 100°C for 17 hours; and

Figure 7b is a chromatogram of a 10% aqueous solution of the solid product made by freeze-drying the solution of Fig. 7a.

The compositions of zirconyl hydroxy chloride and aluminium chlorhydroxide which can be employed in the present invention include those in which the zirconyl compound has the formula $ZrO(OH)_xC1_y$, where $x + y = 2$ or a hydrate thereof and the aluminium compound has the formula $A1_2(OH)_{6-n}C1_n$ where n is from 0.8 to 2 or a hydrate thereof, n preferably being 1. The atomic proportion of aluminium to zirconium in the composition may vary from about 1:1 to 6:1. The atomic proportion of metal to chloride may vary from about 0.9 to 1.9. The inclusion in the composition of a neutral amino acid as described in U.S. Patent 2,854,382 does not have an adverse effect upon the formation of the desired novel complex. Commercially available compositions of zirconyl hydroxy chloride and aluminium chlorhydroxide mixtures frequently contain glycine as the neutral amino acid in an amount such that the molar ratio of glycine to zirconyl hydroxy chloride is about 1:1. The glycine or other neutral amino acid aids in preventing gelation of the aqueous solution before or during the heating step of the method of the present invention and its presence does not prevent formation of the desired complex and does not interfere with the conversion of the heated solution to solid form nor with the subsequent use of the solid as an antiperspirant. Solid compositions as well as aqueous solutions containing zirconyl hydroxy chloride and aluminium chlorhydroxide are available commercially, usually containing a water soluble amino acid to prevent gelling; these materials may be employed in practising the present invention. The individual zirconyl and aluminium components can also be obtained commercially and mixed in solution. It is preferred for best results to employ as starting material an aqueous solution containing zirconyl hydroxy chloride, to which are added an approximately equimolar amount of glycine, and $A1_2(OH)_5C1.2H_2O$ in an amount to provide an atomic ratio of A1:Zr equal to about 4:1.

In practising the method of the present invention, an aqueous solution containing a mixture of zirconyl hydroxy chloride and aluminium chlorhydroxide in an amount from about 2% to about 20% by weight of the total solution, preferably 8 to 15%, is heated at a temperature of at least 50°C, preferably 70°C or higher. Although temperatures above 100°C can be used, pressure vessels are needed for higher temperatures, adding to the expense, so that temperatures from 70°C to 100°C are most preferred. After heating has been continued for several hours an aliquot sample of the solution is subjected to gel permeation chromatography on a cross-linked dextran, (Sephadex G-50) using as the eluent 0.1 molar aqueous KC1 with the pH adjusted to 3.0 with HC1, and the distribution of the metal complexes is measured by monitoring the refractive index of the eluent. As appears from Fig. 1, 6 peaks occur in the case of a typical freshly prepared 10% by weight aqueous solution containing zirconyl hydroxy chloride and aluminium chlorhydroxide $(A1)_2(OH)_5C1$. Peak 1 as shown in the drawing is the eluent fraction containing the zirconium compound; this compound is excluded by the gel because of its large molecular size. Peak 2 contains the $A1^c$ complex, and peak 3 contains the $A1^{c'}$ complex. Peak 4 which appears only as a shoulder between peaks 3 and 5 contains the hitherto unrecognized novel complex which is present only in small proportion, while peaks 5 and 6 are fractions containing $A1^a$ or other lower molecular weight compounds.

As appears from Fig. 2 of the drawing, the solution characterized in Fig. 1, after heating at 100°C for 2 hours, displays a quite different distribution of complexes when subjected to gel

permeation chromatography under the same conditions, the most striking change being a large decrease in the height of peak 3 and a very large increase in the height of peak 4. It has been found that the extent of antiperspirant efficacy of the composition is generally proportional to the relative heights of peak 4 and peak 3, i.e. to the relative amounts of the complex of peak 4 present in the composition as compared to the $A1^{c'}$ complex of peak 3. Those compositions in which the ratio of the height of peak 4 to that of peak 3 is at least 2:1 display the desired superior antiperspirant efficacy.

The length of time of heating necessary to achieve the desired ratio of height of peak 4 to peak 3 depends upon the temperature and the concentration of the solution. The higher the temperature, the more rapid is the increase in ratio. To achieve a ratio of height of peak 4 to peak 3 of 3 or more, it is necessary to heat solutions of a concentration of 2–10% at a temperature of 80°C or more.

In the case of a 10% aqueous solution heated at 50°C, as shown in Fig. 3 of the drawing, the ratio of the height of peak 4 to that of peak 3 rapidly approaches a maximum and thereafter remains substantially constant as heating is continued. This is typical of the results obtained at other temperatures and concentrations, as can be seen from Fig. 4 of the drawing which shows the change in the ratio of peak 4 to peak 3 of a 10% aqueous solution when heated at reflux temperature.

The rate of reversion at room temperature of a 10% aqueous solution which had previously been heated at 100°C for 46 hours is shown in Fig. 5 of the drawing, from which it is clear that the rate of reversion is so rapid that it is not commercially practical to package for sale aqueous solutions having a high ratio of peak 4 to peak 3.

The following specific examples are intended to illustrate more fully the nature of the present invention without acting as a limitation upon its scope.

Example 1

Approximately 1 litre of an aqueous solution containing (10% solids) $ZrO(OH)C1$, $A1_2(OH)_5C1$ and glycine in which the atomic ratio of A1 to Zr was 3.6 to 1 and the ratio of Zr to glycine was 1:1 was heated at 100°C for 20 hours.

A portion of the solution, within less than 2 hours after cooling to room temperature, was subjected to gel permeation chromatography on a cross-linked dextran (Sephadex G-50) column using pH 3, 0.1M KC1 as the eluent, and the refractive index of the eluent fractions was measured. The resulting chromatogram is shown in Fig. 6a. It exhibits successive peaks in which the height of peak 4 is several times the height of peak 3, similar to that of Fig. 2.

Another portion of this heated solution was immediately subjected to spray-drying on a Buchi model 190 spray drier with an inlet temperature of 147°C and an outlet temperature of 80°C to provide a fine, dry powder.

A portion of the powder was then redissolved in water to form a 10% by weight solution and within less than 2 hours was subjected to gel permeation chromatography under the same conditions as the earlier solution. The resultant chromatogram is shown in Fig. 6b of the drawing, from which it is clear that the ratio of the height of peak 4 to that of peak 3 was not greatly changed.

Example 2

A different specimen of a 10% aqueous solution of the same mixture of zirconyl, aluminium and glycine compounds was aged 17 hours at 100°C; its gel permeation chromatographic characteristics, measured as in Example 1, are shown in Fig. 7a of the drawing. A specimen of the aged solution was immediately subjected to freeze-drying using a Virtis Unitrap Freeze-Drier. The dried residue was ground and passed through a 200 mesh sieve.

A portion of the powder was then redissolved in water to form a 10% by weight solution and subjected to gel permeation chromatography under the same conditions as the earlier solution. The resultant chromatogram is shown in Fig. 7b of the drawing. The slight reduction in ratio of height of peak 4 to that of peak 3 after freeze-drying while somewhat greater than the reduction which occurred in the case of spray-drying, still produced a product having enhanced antiperspirant activity as compared to the antiperspirant activity of the original aqueous solution characterized in Fig. 1 of the drawing.

Both the spray-dried and the freeze-dried solid powders exhibited no further substantial change in chromatographic characteristics during storage at room temperature for several months.

Typical antiperspirant formulations in which the material of the present invention may be practically employed are as follows:

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	I. *Non-aqueous Roll-On*	*Weight %*
	Finely divided solid of present invention	20.0
	Quaternium-18 Hectorite	2.7
	Anhydrous Alcohol SDA-40	1.6
	H_2O	0.2
	Cyclomethicone, a silicone oil	75.5
	Perfume	q.s
	II. *Stick Antiperspirant*	
	Finely divided solid of present invention	23.0
	Ozokerite Wax	22.4
	Myristyl Alcohol	17.2
	Cyclomethicone	17.9
	PPG-15 Stearyl ether	11.5
	Steareth-15	2.3
	Bentone Gel IPM	5.7
	Perfume	q.s
	III. *Aerosol Antiperspirant*	
	Finely divided solid of present invention	7.0
	Talc	2.0
	Anhydrous Alcohol SDA-40	4.2
	Quaternium-18 Hectorite	1.4
	Cyclomethicone	4.0
	Isopropyl myristate	3.0
	Perfume	0.1
	Propellant-A31	78.3

The product of the present invention has been evaluated for antiperspirant activity using standard Hot Room clinical testing procedures both as a 10% aqueous solution, and as a dry powder in a non-aqueous roll-on formulation. In each case, it was found to be superior to the analogous conventional material having the gel permeation characteristics as shown in Fig. 1 of the drawing.

CLAIMS

1. A stable, solid composition comprising zirconyl hydroxy chloride and aluminium chlorhydroxide having improved antiperspirant efficacy, said composition, when dissolved in water to form a 10% by weight solution and, after storage at room temperature for no more than two hours, subjected to gel permeation chromatography on cross-linked dextran having a molecular weight exclusion range of 1000 to 30,000 for globular proteins, exhibiting a distribution pattern having at least four successive peaks in which the ratio of the height of peak 4 to that of peak 3 is at least 2:1.

2. A method of increasing the antiperspirant efficacy of a composition comprising zirconyl hydroxy chloride and aluminium chlorhydroxide, which comprises providing a 2% to 20% solution of said composition in water,

heating the solution at a temperature of at least 50°C until the ratio of the height of peak 4 to that of peak 3 as measured by gel permeation chromatography of a 10% aqueous solution on cross-linked dextran having a molecular weight exclusion range of 1000 to 30,000 for globular proteins, exceeds 2:1,

then subjecting the solution to rapid drying to solid form, said solid, when dissolved in water to form a 10% by weight solution and, after storage at room temperature for no more than two hours, subjected to gel permeation chromatography on said cross-linked dextran exhibiting a distribution pattern having at least four successive peaks in which the ratio of the height of peak 4 to that of peak 3 is at least 2:1.

3. A method as claimed in claim 2, in which the solution has a concentration of 8 to 15%.

4. A method as claimed in claim 2 or 3, in which the heating temperature is 70°C to 100°C.

5. A method as claimed in any of claims 2 to 4, in which the solution is subjected to spray drying.

6. A method as claimed in any of claims 2 to 4, in which the solution is subjected to freeze

drying.

7. A method as claimed in any of claims 2 to 6, in which the solution contains a water-soluble amino acid.

8. A solid composition according to claim 1 substantially as herein described in Example 1 or 2.

9. An antiperspirant composition comprising a solid as claimed in claim 1 in finely divided form dispersed in a non-aqueous, non-toxic vehicle.

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A61K 8/43	(2006.01)	A61K	8/43	
A61K 8/34	(2006.01)	A61K	8/34	

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(54)【発明の名称】抗菌性組成物及びデオドラント剤

(57)【要約】
【課題】汗や皮脂の量や比率が異なる様々な肌状態でも、特定の化合物がそれぞれ有する抗菌活性を相乗的に増強できる抗菌性組成物、並びに該抗菌性組成物を含有するデオドラント剤を提供すること。
【解決手段】塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる２種以上の化合物を含有することを特徴とする腋臭原因菌に好適な抗菌性組成物、並びに該組成物を含有してなるデオドラント剤とする。
【選択図】なし

【特許請求の範囲】
【請求項１】
塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる２種以上の化合物を含有することを特徴とする抗菌性組成物。
【請求項２】
前記２種以上の化合物が、
トリクロサンとトリクロロカルバニリド、
トリクロサンと塩化ベンザルコニウム、
トリクロサンとジンクピリチオン、
トリクロサンとピロクトンオラミン、
イソプロピルメチルフェノールとトリクロロカルバニリド、
イソプロピルメチルフェノールと塩化ベンザルコニウム、
イソプロピルメチルフェノールとジンクピリチオン、
イソプロピルメチルフェノールとピロクトンオラミン、
イソプロピルメチルフェノールとレゾルシン、
イソプロピルメチルフェノールとサリチル酸、
トリクロロカルバニリドと塩化ベンザルコニウム、
トリクロロカルバニリドとジンクピリチオン、
トリクロロカルバニリドとピロクトンオラミン、
塩化ベンザルコニウムとピロクトンオラミン、
塩化ベンザルコニウムとトリクロサンとイソプロピルメチルフェノール、又は
ジンクピリチオンとトリクロサンとイソプロピルメチルフェノール
である請求項１に記載の抗菌性組成物。
【請求項３】
抗腋臭原因菌用である請求項１又は２に記載の抗菌性組成物。
【請求項４】
請求項１～３のいずれかに記載の抗菌性組成物を含有してなるデオドラント剤。
【発明の詳細な説明】
【技術分野】
【０００１】
本発明は抗菌性組成物、並びに該抗菌性組成物を含有したデオドラント剤に関する。
【背景技術】
【０００２】
人に不快感を与える腋臭などの体臭は、特に、汗が皮脂と混ざり、それが腋臭原因菌により分解されることにより生じるとされている。
【０００３】
そこで、従来から、体臭の発生を抑制するために、抗菌性化合物がデオドラント剤に用いられてきた（例えば、特許文献１～３を参照）。
【０００４】
しかし、人の肌や頭皮には汗腺や皮脂腺が存在し、その時々で汗と皮脂の量が異なり、汗（水分）が多い状態と皮脂が多い状態とでは、抗菌性化合物の抗菌活性が異なる可能性があった。従来のデオドラント剤では、このような肌状態の変化に対応できず、防臭・消臭効果を十分に発揮できないといった問題があった。さらには、肌状態の変化により、防臭・消臭効果が持続して得られないといった問題もあった。
【０００５】
一方、トリクロサン及びイソプロピルメチルフェノールを併用することにより、汗が多く分泌した肌状態に加え、皮脂が多く分泌した肌状態でも、腋臭原因菌に対する抗菌活性

を維持できるデオドラント剤が報告されている（特許文献４を参照）。

【０００６】

しかし、上記技術では、異なった肌状態でも腋臭原因菌に対する抗菌活性を維持できることに留まり、抗菌性化合物のそれぞれが有する抗菌活性を相乗的に増強できるものではなく、種々の肌状態でもより効果的に防臭・デオドラント効果を発揮できるデオドラント剤が望まれる。

【０００７】

【特許文献１】特開２００２－８０３３５号公報
【特許文献２】特開２００３－８１８０１号公報
【特許文献３】特開２００４－１８９６３３号公報
【特許文献４】特開２００６－９６７１９号公報

【発明の開示】

【発明が解決しようとする課題】

【０００８】

本発明は、上記従来技術に鑑みてなされたものであって、汗や皮脂の量や比率が異なる様々な肌状態でも、特定の化合物がそれぞれ有する抗菌活性を相乗的に増強できる抗菌性組成物、並びに該抗菌性組成物を含有するデオドラント剤を提供することを課題とする。

【課題を解決するための手段】

【０００９】

すなわち、本発明は、

〔１〕塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる２種以上の化合物を含有することを特徴とする抗菌性組成物、

〔２〕抗腋臭原因菌用である前記〔１〕に記載の抗菌性組成物、並びに

〔３〕前記〔１〕又は〔２〕に記載の抗菌性組成物を含有してなるデオドラント剤

に関する。

【発明の効果】

【００１０】

本発明にかかる抗菌性組成物は、汗や皮脂の量や比率が異なる様々な肌状態でも、特定の化合物のそれぞれが本来有する抗菌活性を相乗的に増強することができるという効果を奏する。

【００１１】

しかも、腋臭原因菌に対して優れた抗菌活性を発揮することから、抗腋臭原因菌効果を発揮するデオドラント剤として有用である。

【発明を実施するための最良の形態】

【００１２】

本発明にかかる抗菌性組成物は、必須成分として塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる２種以上の特定の化合物を含有する。かかる構成を有することで、腋臭などの体臭に優れた防臭・デオドラント効果を発揮するとともに、その効果を長時間持続することができる。

【００１３】

具体的には、腋臭原因菌に対して相乗的な優れた抗菌活性を発揮する観点から、以下の特定の２種又は３種の化合物を組み合わせて含有するのが好ましい。

【００１４】

トリクロサンとトリクロロカルバニリド、

トリクロサンと塩化ベンザルコニウム、
トリクロサンとジンクピリチオン、
トリクロサンとピロクトンオラミン、
イソプロピルメチルフェノールとトリクロロカルバニリド、
イソプロピルメチルフェノールと塩化ベンザルコニウム、
イソプロピルメチルフェノールとジンクピリチオン、
イソプロピルメチルフェノールとピロクトンオラミン、
イソプロピルメチルフェノールとレゾルシン、
イソプロピルメチルフェノールとサリチル酸、
トリクロロカルバニリドと塩化ベンザルコニウム、
トリクロロカルバニリドとジンクピリチオン、
トリクロロカルバニリドとピロクトンオラミン、
塩化ベンザルコニウムとピロクトンオラミン、
塩化ベンザルコニウムとトリクロサンとイソプロピルメチルフェノール、又は
ジンクピリチオンとトリクロサンとイソプロピルメチルフェノール。

【００１５】

特に、トリクロサンとピロクトンオラミン、イソプロピルメチルフェノールとレゾルシン、又はイソプロピルメチルフェノールとサリチル酸の特定の２種を組み合わせて含有するのが好ましい。

【００１６】

尚、ピロクトンオラミンとは、一般名称、１－ヒドロキシ－４－メチル－６－（２，４，４－トリメチルペンチル）－２（１Ｈ）－ピリドン　モノエタノールアミン塩の化合物である。市販品としては、商品名、オクトピロックス（クラリアント・ジャパン社製）として入手が可能である。

【００１７】

本発明にかかる抗菌性組成物は、各化合物の含有量は特に限定されないが、第一の化合物１重量部に対し、第二以降の化合物のそれぞれを０．１～１０重量部とするのが好ましく、より好ましくは０．２～５重量部、更に好ましくは０．５～２重量部となるように配合する。第一の化合物に対し第二以降の化合物が１０重量部を超えて配合すると、また、０．１重量部未満の場合、抗菌活性の相乗的な増強効果が期待できないために好ましくない。

【００１８】

例えば、第一の化合物にトリクロサンを選択し、第二のトリクロロカルバニドを選択した場合、トリクロサン１重量部に対し、トリクロロカルバニド０．１～１０重量部とするのが好ましい。また、第一の化合物に塩化ベンザルコニウム、第二の化合物にトリクロサン、第三の化合物にイソプロピルメチルフェノールを選択した場合、塩化ベンザルコニウム１重量部に対し、トリクロサン０．１～１０重量部、イソプロピルメチルフェノール０．１～１０重量部とするのが好ましい。

【００１９】

尚、本発明の抗菌性組成物には、上記した化合物の他、本発明の効果を阻害しない範囲で、溶剤、界面活性剤、酸化防止剤、金属イオン封鎖剤等を適宜配合することができる。

【００２０】

本発明の抗菌性組成物は、デオドラント剤に配合して使用することができる。その配合量は、特に限定されないが、デオドラント剤中０．００１～３重量％、好ましくは０．０１～２重量％、より好ましくは０．１～１重量％とするとよい。配合量が０．００１重量％未満の場合、抗菌効果に劣るために、また、３重量％を超えて配合すると、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

【００２１】

本発明では、更に制汗成分を含有することができる。本発明で用いられる制汗成分は、皮膚を収斂することにより汗の発生を抑制する薬剤であり、例えば、塩化アルミニウム、

硫酸アルミニウムカリウム、硫酸アルミニウム、酢酸アルミニウム、クロロヒドロキシアルミニウム、アラントインクロロヒドロキシアルミニウム、パラフェノールスルホン酸亜鉛などを例示することができる。

【００２２】

制汗成分の配合量は、本発明の効果を発揮すれば特に限定されず、デオドラント剤中０．１～２０重量％とするとよく、０．３～１５重量％とすることがより好ましい。０．１重量％未満の配合量では、防臭・デオドラント効果の持続性の効果に劣るために、また、２０重量％を超えて配合すると、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

【００２３】

本発明にかかるデオドラント剤には、本発明の効果を損なわない範囲であれば、上記した成分の他に、消臭成分を配合することができる。消臭成分とは、臭いを発する物質と反応・吸着したり、臭いをマスクしたりして、臭いを消す効果を有する薬剤であり、例えば、酸化亜鉛などの金属酸化物、アルキルジエタノールアミド、ヒドロキシアパタイト、茶抽出物、香料、酸化防止剤などを例示することができる。

【００２４】

消臭成分の配合量は、本発明の効果を発揮すれば特に限定されず、デオドラント剤中０．０１～５重量％とするとよく、０．１～２重量％とすることがより好ましい。０．０１重量％未満の配合量では、臭いのマスキングの効果に劣るために、また、５重量％を超えて配合すると、香りが強くなりすぎたり、肌への着色が生ずる場合があり、また、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

【００２５】

本発明のデオドラント剤には、本発明の効果を阻害しない範囲で、上記した成分の他、エタノール、イソプロピルアルコール等の低級アルコール；ラウリルアルコール、ステアリルアルコール等の高級アルコール；グリセリン、ジグリセリン、ジプロピレングリコール、１，３－ブタンジオール、１，２－ペンタンジオール、１，２－オクタンジオール等の多価アルコール；ベントナイト、カラギーナン、カルボキシビニルポリマー、ヒドロキシエチルセルロース等の増粘剤；トコフェロール及びその誘導体、アスコルビン酸及びその誘導体等の酸化防止剤；エデト酸塩、リン酸、ポリリン酸ナトリウム等の金属イオン封鎖剤；動物及び植物エキス、クエン酸、酒石酸等のｐＨ調整剤；シリコーン類、保湿剤、界面活性剤、防腐剤、粉体、着色剤、水等を適宜配合することもできる。

【００２６】

本発明のデオドラント剤は、ローション、エアゾール、スティック、パウダー、ロールオン、クリーム、ジェル、乳液、シート剤などの種々の形態に用いることができ、製剤化については、一般に知られている方法により製造すればよい。

【実施例】

【００２７】

＜試験例１＞

供試菌として、腋臭の原因菌であるスタフィロコッカス　エピデルミディス（Ｓｔａｐｈｙｌｏｃｏｃｃｕｓ　ｅｐｉｄｅｒｍｉｄｉｓ　ＩＡＭ１２９６）を用いた。

【００２８】

（接種菌液の調製）

接種用菌液としてスタフィロコッカス　エピデルミディスを寒天培地で３５℃で培養後、更にブイヨン培地に移植して３５℃で培養した。得られた培養液をブイヨン培地で約10^7個／ｍＬに希釈したものを接種用菌液とした。

【００２９】

（希釈系列の調製）

ピロクトンオラミン（比較例１）、及びトリクロサン（比較例２）は、７０％エタノールを希釈溶媒として１５００μｇ／ｍＬに調製し、１０段階に倍倍希釈して希釈系列を調製した。

【００３０】
また、ピロクトンオラミンとトリクロサンの等量混合物（実施例１）についても、同様に希釈系列を調製した。
【００３１】
（最小殺菌濃度（ＭＢＣ）の測定）
上記各被験物質を含む希釈系列１ｍＬに対して各寒天培地９ｍＬをシャーレに入れ、それぞれについて、上記接種用菌液を約１ｃｍの長さに画線した。培養は、３５℃で行い５日後の菌の生育の有無を判定した。このとき、生育が認められなかった濃度では、接種した画線から菌を採取し、新鮮寒天培地に移植し更に３日培養した。このとき、生育が認められなかった最小の濃度をＭＢＣとして求め、そのときの各成分の濃度を表１（トレオレイン非添加）に記した。
【００３２】
（皮脂成分の影響の検討）
皮脂成分の影響による殺菌効果を検討するために、皮脂成分としてトレオレインを添加して下記試験を実施した。即ち、上記各被験物質を含む希釈系列１ｍＬに対して、０．１重量％のトリオレインと寒天培地８．９ｍＬをシャーレに入れ、それぞれについて、上記と同様に接種用菌液を約１ｃｍの長さに画線した。培養は、３５℃で行い５日後の菌の生育の有無を判定した。このとき、生育が認められなかった濃度では、接種した画線から菌を採取し、新鮮寒天培地に移植しさらに３日培養した。このとき、生育が認められなかった最小の濃度をＭＢＣとして求め、そのときの各成分の濃度を表１（トレオレイン添加）に記した。
【００３３】
【表１】

	トレオレイン非添加(μg/mL)		トレオレイン添加(μg/mL)	
	ピロクトンオラミン	トリクロサン	ピロクトンオラミン	トリクロサン
比較例１	12	0	47	0
比較例２	0	12	0	750
実施例１	1.5	1.5	23.5	23.5

【００３４】
＜試験例２＞
供試菌として、腋臭の原因菌であるコリネバクテリウム　ミヌティシマム（Ｃｏｒｙｎｅｂａｃｔｅｒｉｕｍ　ｍｉｎｕｔｉｓｓｉｍｕｍ　ＡＴＣＣ２３３４８）を用い、試験例１と同様にして接種用菌液を調製した。
【００３５】
（希釈系列の調製）
イソプロピルメチルフェノール（比較例３）、レゾルシン（比較例４）、及びサリチル酸（比較例５）は、７０％エタノールを希釈溶媒として３０００μｇ／ｍＬに調製し、１１段階に倍倍希釈して希釈系列を調製した。
【００３６】
また、イソプロピルメチルフェノールとレゾルシンの等量混合物（実施例２）、及びイソプロピルメチルフェノールとサリチル酸の等量混合物（実施例３）についても、同様に希釈系列を調製した。
【００３７】
（最小殺菌濃度（ＭＢＣ）の測定）
上記各被験物質を試験例１と同様にして、ＭＢＣを求めた。結果を表２～３に示す。
【００３８】

【表２】

	トレオレイン非添加（μg/mL）		トレオレイン添加（μg/mL）	
	イソプロピルメチルフェノール	レゾルシン	イソプロピルメチルフェノール	レゾルシン
比較例３	188	0	375	0
比較例４	0	3000	0	3000
実施例２	94	94	188	188

【００３９】

【表３】

	トレオレイン非添加（μg/mL）		トレオレイン添加（μg/mL）	
	イソプロピルメチルフェノール	サリチル酸	イソプロピルメチルフェノール	サリチル酸
比較例３	188	0	375	0
比較例５	0	1500	0	1500
実施例３	94	94	188	188

【００４０】

尚、ＭＢＣによって、抗菌力を評価することができる。被験物質の濃度が薄いときには微生物への影響はないが、濃度を増していくと発育抑制が起こり、続いて発育は停止する。更に濃度が増すと、微生物は死滅していくことになる。このときの濃度がＭＢＣとして表される。

【００４１】

表１～３の結果から、本発明の抗菌性組成物は、腋臭原因菌に対して両者の抗菌活性が相乗的に増強されることが分かる。

【００４２】

＜試験例３＞

（試料の調製）

表４～５記した組成に従い、実施例４～６及び比較例６～１０の各デオドラント剤を常法により調製し、下記評価に供した。尚、表中の配合量は重量％を表す。

【００４３】

（試験例：デオドラント剤の評価）

腋臭が強いと判定された男子被験者１０名に、各試料を被験者の一方の腋下に塗布し、もう一方の腋下は比較対照として塗布しなかった。塗布前とその直後及び３時間後のそれぞれについて下記評価基準に従って臭いの判定をし、その平均値を採用した。結果を表３に示す。

【００４４】

＜評価基準＞

臭わない・・・・・・・・０点
かすかに臭う・・・・・・１点
やや臭うが弱い・・・・・２点
はっきりと臭う・・・・・３点
非常に強く臭う・・・・・４点

【００４５】

【表４】

	実施例４		比較例６		比較例７	
ピクトンオラミン	0.2		0.2		—	
トリクロサン	0.2		—		0.2	
エタノール	70.0		70.0		70.0	
精製水	残分		残分		残分	
	塗布部	対照部	塗布部	対照部	塗布部	対照部
塗布前	3.8	4.0	4.0	4.0	4.0	3.8
塗布直後	1.0	3.8	1.8	3.8	1.8	3.8
塗布３時間後	1.2	4.0	2.6	4.0	2.8	4.0

【００４６】

【表５】

	実施例５		実施例６		比較例８		比較例９		比較例10	
ｲｿﾌﾟﾛﾋﾟﾙﾒﾁﾙﾌｪﾉｰﾙ	0.2		0.2		0.2		—		—	
レゾルシン	0.2		—		—		0.2		—	
サリチル酸	—		0.2		—		—		0.2	
エタノール	70.0		70.0		70.0		70.0		70.0	
精製水	残分		残分		残分		残分		残分	
	塗布部	対照部	塗布部	対照部	塗布部	対照部	塗布部	対照部	塗布部	対照部
塗布前	4.0	4.0	3.8	3.8	3.8	4.0	4.0	3.8	3.8	4.0
塗布直後	1.2	4.0	1.4	4.0	2.0	4.0	2.6	4.0	2.6	4.0
塗布３時間後	1.8	3.8	1.8	3.8	2.6	3.8	3.4	4.0	3.2	3.8

【００４７】

表３～４の結果から、本発明のデオドラント剤は優れた防臭効果を有するとともに、その効果も長時間持続することが分かる。

【００４８】

以下、本発明にかかるデオドラント剤の処方例を示す。尚、配合量は重量％である。

【００４９】

（処方例１：デオドラントスプレー）

メチルフェニルポリシロキサン	１．３１
イソノナン酸イソノニル	２．２５
トリクロサン	０．０２
トリクロロカルバニリド	０．０２
メントール	０．１
クロルヒドロキシアルミニウム	１．５
シリカ	３．０
タルク	１．７
香料	０．１
液化石油ガス	残　分
合　　計	１００．０

【００５０】

（処方例２：デオドラントスプレー）

メチルフェニルポリシロキサン	０．５１
ミリスチン酸イソプロピル	２．８６
イソプロピルメチルフェノール	０．０１
トリクロロカルバニリド	０．０２
メントール	０．０５
酸化亜鉛	０．５
シリカ	４．３
タルク	１．６
香料	０．１
液化石油ガス	残　分
合　　計	１００．０

【００５１】
（処方例３：デオドラントスプレー）

エタノール	３７．９６
メントール	０．３
メチルフェニルポリシロキサン	１．０
トリクロサン	０．１
塩化ベンザルコニウム	０．０４
パラフェノールスルホン酸亜鉛	０．２
香料	０．４
液化石油ガス	残　分
合　　計	１００．０

【００５２】
（処方例４：デオドラントスプレー）

エタノール	３８．３６
イソノナン酸イソノニル	１．０
イソプロピルメチルフェノール	０．０５
塩化ベンザルコニウム	０．０４
パラフェノールスルホン酸亜鉛	０．１５
香料	０．４
液化石油ガス	残　分
合　　計	１００．０

【００５３】
（処方例５：デオドラントロールオン）

エタノール	６０．０
イソノナン酸イソノニル	１．０
トリクロサン	０．２
ジンクピリチオン	１．０
クロルヒドロキシアルミニウム	１０．０
メントール	０．３
香料	０．４
精製水	残　分
合　　計	１００．０

【００５４】
（処方例６：デオドラントロールオン）

エタノール	６０．０
イソノナン酸イソノニル	１．０
イソプロピルメチルフェノール	０．１
ジンクピリチオン	１．０
クロルヒドロキシアルミニウム	１０．０

メントール	０．１
香料	０．２
精製水	残　分
合　　計	１００．０

【００５５】
（処方例７：デオドラントローション）

エタノール	２０．０
ポリオキシエチレン硬化ヒマシ油	１．０
ジプロピレングリコール	２．０
イソプロピルメチルフェノール	０．１
ピロクトンオラミン	０．２
パラフェノールスルホン酸亜鉛	０．３
香料	０．３
精製水	残　分
合　　計	１００．０

【００５６】
（処方例８：デオドラントローション）

エタノール	３０．０
ポリオキシエチレンポリオキシプロピレンデシルテトラデシルエーテル	０．５
トリクロサン	０．２
ピロクトンオラミン	０．２
パラフェノールスルホン酸亜鉛	０．３
メントール	０．２
香料	０．２
精製水	残　分
合　　計	１００．０

【００５７】
（処方例９：デオドラントミスト）

エタノール	７０．０
塩化ベンザルコニウム	０．１
トリクロロカルバニリド	０．１
アラントインクロルヒドロキシアルミニウム	０．５
メントール	０．１
香料	０．５
精製水	残　分
合　　計	１００．０

【００５８】
（処方例１０：デオドラントミスト）

エタノール	３０．０
ポリオキシエチレン硬化ヒマシ油	０．５
塩化ベンザルコニウム	０．１
ピロクトンオラミン	０．２
硫酸アルミニウムカリウム	５．０
メントール	０．４
香料	０．５
精製水	残　分
合　　計	１００．０

【００５９】
（処方例１１：デオドラントスティック）

セタノール	５．０
ステアリン酸ナトリウム	１０．０
ミリスチン酸イソプロピル	１０．０
トリクロロカルバニリド	０．１
ジンクピリチオン	０．１
クロルヒドロキシアルミニウム	１５．０
メントール	０．４
香料	０．３
エタノール	残　分
合　　計	１００．０

【００６０】

（処方例１２：デオドラントスティック）

ステアリルアルコール	２５．０
ポリエチレングリコール	１０．０
トリクロカルバン	０．１
ピロクトンオラミン	０．２
クロルヒドロキシアルミニウム	２０．０
香料	０．３
デカメチルシクロペンタシロキサン	残　分
合　　計	１００．０

【００６１】

（処方例１３：デオドラントジェル）

エタノール	６０．０
カルボキシビニルポリマー	０．４
環状シリコーン	１．０
ポリエーテル変性シリコーン	０．５
トリクロサン	０．１
イソプロピルメチルフェノール	０．１
塩化ベンザルコニウム	０．１
トリエタノールアミン	０．４
シリコーンパウダー	１．０
香料	適　量
精製水	残　分
合　　計	１００．０

【００６２】

（処方例１４：パウダースプレー）

クロルヒドロキシアルミニウム	３．０
無水ケイ酸	１．５
シリコーン処理タルク	１．５
酸化亜鉛	０．５
トリクロサン	０．０５
イソプロピルメチルフェノール	０．１
ジンクピリチオン	０．０５
イソプロピルミリスチン酸エステル	２．０
ジメチルポリシロキサン	１．０
ソルビタン脂肪酸エステル	０．３
液化石油ガス	残　分
合　　計	１００．０

【００６３】

（処方例１５：液体防臭剤）

パラフェノールスルホン酸亜鉛	２．０
エタノール	３０．０
１，３－ブチレングリコール	３．０
レゾルシン	０．１
イソプロピルメチルフェノール	０．１
ポリオキシエチレン（50）硬化ヒマシ油	０．５
香料	適量
精製水	残　分
合　　計	１００．０

【００６４】

（処方例１６：消臭スプレー）

精製水	３０．０
エタノール	２０．０
サリチル酸	０．２
イソプロピルメチルフェノール	０．１
ジメチルエーテル	残　分
合　　計	１００．０

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Translate
Fulltext

Antibacterial composition and deodorant agent

Abstract

PROBLEM TO BE SOLVED: To provide an antibacterial composition which can synergistically reinforce respective antibacterial actions of specific compounds even in various skin states which differ in the amount and the ratio of sweat and sebum, and a deodorant agent comprising the antibacterial composition.
SOLUTION: The antibacterial composition suitable against Staphylococcus epidermidis comprises at least two compounds selected from the group consisting of benzalkonium chloride, benzethonium chloride, halocarban, trichlorocarbanilide, chlorhexidine hydrochloride, isopropylmethylphenol, triclosan, chlorhexidine gluconate, piroctone olamine, resorcin, lysozyme chloride, salicylic acid, hinokithiol, and thymol, and the deodorant agent comprises the composition.
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-JPH08325126 (A) [JP08325126] KAO CORP;
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Applicant citations
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-JP2003081801 (A) [JP2003081801];
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-JP2006096719 (A) [JP2006096719];

Claims

1. Benzalkonium chloride, benzethonium chloride, halo calvin, trichlorocarbanilide, chlorhexidine hydrochloride, isopropylmethylphenol, triclosan, chlorhexidine gluconate, zinc pyrithione, piroctone olamine, resorcin, lysozyme chloride, salicylic acid, and 2 is selected from the group consisting of thymol hinokitiol is one or more compounds characterized in containing the antibacterial composition.

2. 2 The method of one or more compounds, triclosan and trichlorocarbanilide, triclosan and benzalkonium chloride, triclosan and zinc pyrithione, triclosan and piroctone olamine, and trichlorocarbanilide isopropylmethylphenol, benzalkonium chloride isopropylmethylphenol, zinc isopropylmethylphenol, and isopropylmethylphenol piroctone olamine, and resorcin isopropylmethylphenol, salicylic acid isopropylmethylphenol, benzalkonium chloride trichlorocarbanilide, zinc trichlorocarbanilide, and piroctone olamine trichlorocarbanilide, benzalkonium chloride and piroctone olamine, and benzalkonium chloride and triclosan isopropylmethylphenol, triclosan and zinc or anti-microbial composition according to claim 1 and is isopropylmethylphenol.

3. Underarm odor according to claim 1 or 2 for anti-microbial composition is the causative agent.

4. Any one of claims 1-3 antimicrobial composition containing a deodorant agent.

Description

TECHNICAL FIELD

[0001] The present invention relates to anti-microbial composition, as well as the antibacterial composition containing deodorant agent.

BACKGROUND ART

[0002] Such as discomfort to the human body odor is underarm odor, in particular, sweat sebaceous and mixed, by which the causative underarm odor caused by decomposition thereof.

[0003] Therefore, conventionally, in order to suppress the occurrence of body odor, deodorant agent antimicrobial compounds have been used (for example, see Patent document 1-3).

[0004] However, the human skin or scalp sebum or sweat glands, sebum amount varies at any given sweat, sebum and sweat (moisture) is large in the high state, the antimicrobial compounds of antimicrobial activity is likely to be different. In the conventional deodorant agent, thus can not correspond to the change of the state of the skin, odor, effects on the problem may not be exhibited sufficiently. Further, the skin condition changes, odor, effects on persistence is the problem cannot be obtained.

[0005] On the other hand, by combining isopropylmethylphenol and triclosan, many skin perspiration secretion in addition to the state, even many skin sebum secretion, cause underarm odor deodorant agent can maintain an activity against gram have been reported (see Patent document 4).

[0006] However, in the technique described above, even in a state cause different skin can maintain the activity against underarm odor remains, the antimicrobial compounds included in each of the antimicrobial activity can be enhanced in a synergistic fashion but, even the skin condition of the various more effectively deodorant, deodorant effect can be produced deodorant are desired.

[0007] PATENT CITATIONS

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DISCLOSURE

TECHNICAL PROBLEM

[0008] The present invention relates, in view of the above prior art or, amount and ratio of sweat or sebum in a variety of different skin condition, each having a particular compound to synergistically enhance the antimicrobial activity can be anti-microbial composition, as well as the antibacterial composition containing an object to provide a deodorant agent.

TECHNICAL SOLUTION

[0009] That is, the present invention is, '1' benzalkonium chloride, benzethonium chloride, halo calvin, trichlorocarbanilide, chlorhexidine hydrochloride, and isopropylmethylphenol, triclosan, chlorhexidine gluconate, and zinc, piroctonolamine, resorcin, lysozyme chloride, salicylic acid, 2 is selected from the group consisting of normal thymol and one or more compounds containing the

antibacterial composition, '2' for the causative agent wherein the anti-underarm odor '1' the antimicrobial compositions, and '3' the '1' or '2' to containing the microbial composition of deodorant agent.

ADVANTAGEOUS EFFECTS

[0010] The anti-microbial composition according to the present invention, the amount and ratio of sweat or sebum in a variety of different skin condition, each of the specific compound originally has to synergistically enhance the antimicrobial activity can be achieved.

[0011] In addition, underarm odor causing bacteria exhibiting excellent anti-microbial activity, anti-underarm odor causative effect is useful as a deodorant.

BEST MODE

[0012] Anti-microbial composition according to the present invention, as essential components benzalkonium chloride, benzethonium chloride, halo calvin, trichlorocarbanilide, chlorhexidine hydrochloride, and isopropylmethylphenol, triclosan, chlorhexidine gluconate, and zinc, piroctonolamine, resorcin, lysozyme chloride, salicylic acid, 2 is selected from the group consisting of normal thymol and one or more particular compound. By having such a configuration, such as an anhydrous deodorant underarm odor is excellent, and deodorant effect, the effect can be maintained for a long time.

[0013] More specifically, underarm odor causative synergistic excellent antimicrobial activity from the viewpoint of, the following description of specific 2 3 one compound or two or preferably in combination.

[0014] Triclosan and trichlorocarbanilide, triclosan and benzalkonium chloride, triclosan and zinc, and triclosan piroctonolamine, and isopropylmethylphenol trichlorocarbanilide, benzalkonium chloride isopropylmethylphenol, zinc isopropylmethylphenol, and isopropylmethylphenol piroctonolamine, and resorcin isopropylmethylphenol, salicylic acid isopropylmethylphenol, benzalkonium chloride trichlorocarbanilide, trichlorocarbanilide and zinc, and trichlorocarbanilide piroctonolamine, benzalkonium chloride and piroctonolamine, benzalkonium chloride and triclosan isopropylmethylphenol and, or zinc isopropylmethylphenol and triclosan.

[0015] In particular, triclosan and piroctonolamine, and resorcin isopropylmethylphenol, salicylic acid isopropylmethylphenol or in combination with a particular one of preferably 2.

[0016] In addition, the piroctonolamine, common name, 1 - methyl -6 - - hydroxy - -4 - - (2, 4, 4 - trimethylpentyl) -2 (1H) of the pyridone monoethanolamine salt compound. As a commercially available product, trade name, available as (clariant, japan k.k.) climbasole is possible.

[0017] Anti-microbial composition according to the present invention, the content of each compound is not particularly limited, for the first compound 1 parts by weight, after each of the second compound is preferably 0.1-10 parts by weight, more preferably 0.2-5 parts by weight, more preferably 0.5-2 parts by weight are mixed. The second and subsequent to the first compound 10 parts by weight of the compound is blended exceeds, in addition, in the case of 0.1 parts by weight or less, antimicrobial activity synergistic enhancement effect cannot be expected not preferred.

[0018] For example, for the first compound triclosan is selected, when the user selects the first trichlorocarbanilide nido, triclosan 1 parts by weight, preferably 0.1-10 parts by weight trichlorocarbanilide nido. In addition, the first compound benzalkonium chloride, of a second compound triclosan, the third compound selects isopropylmethylphenol, benzalkonium chloride 1 parts by weight, triclosan 0.1-10 parts by weight, preferably 0.1-10 parts by weight isopropylmethylphenol.

[0019] In addition, the microbial composition of the present invention, other compounds described above, the scope in effect of the present invention, solvents, surfactants, antioxidants, sequestering agents and the like can be formulated as appropriate.

[0020] The microbial composition of the present invention, deodorants and formulated into a can be used. The blending amount is, but not limited to, 0.001-3 weight % deodorant agent, preferably 0.01-2 weight %, more preferably 0.1-1 weight % may be made. When the amount is less than 0.001 weight %, to the antimicrobial effect to measure, in addition, 3 weight % blended beyond, such as skin irritation to a problem in safety because, in any case is not preferable.

[0021] In the present invention, can be further antiperspirant component. The antiperspirant component used in the present invention, by converging the skin perspiration and agents that, for example, aluminum chloride, aluminum potassium sulfate, aluminum sulfate, aluminum acetate, chlorohydroxy, allantoin chlorohydroxy aluminum, zinc or the like para-phenol sulfonic acid can be exemplified.

[0022] The amount of the antiperspirant is, if the effect of the present invention is not particularly limited, and 0.1-20 weight % deodorant agent and may be, 0 .3-15 weight % and more preferably. 0.1 Weight % with a mixing amount of less than, deodorant, deodorant of sustained effect to the effect of the ineffective, in addition, 20 weight % blended beyond, such as skin irritation to a problem in safety because, in any case is not preferable.

[0023] According to the present invention deodorant agents include, does not impair the effect of the present invention is in the range, in addition to the components described above, odor component can be formulated. Odor

component, odor react, absorb, or mask the smell, the smell extinguishing effect and which, for example, a metal oxide such as zinc oxide, alkyl diethanolamide, hydroxyapatite, tea extract, perfume, antioxidants and the like can be exemplified.

[0024] The amount of the odor is, if the effect of the present invention is not particularly limited, and may be 0.01-5 weight % deodorant agent, 0 .1-2 weight % more preferred. 0.01 Weight % with a mixing amount of less than, the ineffective odor masking effect. in addition, 5 weight % blended exceeds, or is too strong scent, skin coloring may occur and, further, such as skin irritation to a problem in safety because. in any case is not preferable.

[0025] The present invention deodorant agents include, within the scope in effect of the present invention, in addition to the components described above, ethanol, lower alcohols such as isopropyl alcohol; lauryl alcohol, higher alcohols such as stearyl alcohol; glycerin, diglycerin, dipropylene glycol, 1, 3 - butanediol, 1, 2 - pentanediol, polyhydric alcohols such as 1, 2 - octanediol; bentonite, carrageenan, carboxyvinyl polymer, hydroxyethyl cellulose thickeners, tocopherol and derivatives thereof, of antioxidants such as ascorbic acid and derivatives thereof; edetate salt, phosphate, sequestering agent such as sodium polyphosphate; animal and plant extracts, citric acid, tartaric acid pH of the agent; silicones. humectants. surfactants. preservatives, powder, coloring agent. water or the like may be blended as appropriate.

[0026] The present invention deodorant agent, lotion, aerosol, stick, powder, roll-on, creams, gels, emulsions. sheet and the like can be used in a variety of forms, formulations is. generally known may be manufactured by the method.

MODE FOR INVENTION

[0027] ≪ test example 1> fungus as provided, the cause of the underarm odor staphylococcus bacteria using s. (Staphylococcus epidermidis IAM1296).

[0028] For vaccination (inoculum preparation of) as in s. staphylococcus scytalidhim 35 °C agar medium after incubation, further broth medium and incubated at 35 °C is implanted. The resulting culture broth medium 10 about^{7} /ml diluted to one for vaccination and bacteria.

[0029] (Preparation of dilution series) (Comparative Example 1) piroctonolamine, is (Comparative Example 2) and triclosan, diluted with ethanol as a solvent prepared 1500μg/ml 70%, were diluted 10 - fold dilution series stage was prepared

[0030] In addition, a mixture of equal parts and triclosan piroctonolamine (embodiment 1) also, a dilution series prepared in the same manner

[0031] (Measurement of minimum bactericidal concentration (MBC)) the each test material including 9 ml to 1 ml dilution series each agar medium placed in a petri dish, for each, for about 1 cm inoculated bacteria liquid and the length of each line. Culture, 35 °C 5 performed after determining the presence or absence of bacterial growth. At this time, growth was observed in the concentration, the printed image taken from bacteria inoculated, fresh agar medium were further implanted 3. At this time, growth was observed is determined as the minimum concentration MBC, the concentration of each component at that time are shown in Table 1 (non-addition torre oleic) noted above.

[0032] (Investigation of the influence of sebum) due to sebum to investigate the sterilization effect, added as sebum torre oleic following test was performed. That is, the dilution series for each test material including 1 ml, 8.9 ml and 0.1 weight % of the agar medium placed in a petri dish triolen, respectively, as described above for the vaccination of bacteria liquid and about 1 cm of the length of each line. Culture, 35 °C 5 performed after determining the presence or absence of bacterial growth. At this time, growth was observed in the concentration, the printed image taken from the inoculated bacteria, fresh agar medium were further implanted 3. At this time, growth was observed is determined as the minimum concentration MBC, the concentration of each component are shown in table 1 at that time (added torre oleic) noted above.

TABLE 1

[0034] ≪ test example 2> fungus as provided, the cause of the underarm odor (Corynebacterium minutissimum ATCC23348) corynebacterium minutishimamu using bacteria, in the same manner as in test example 1 was prepared for the inoculated bacteria.

[0035] (Preparation of dilution series) (Comparative Example 3) isopropylmethylphenol, resorcinol (Comparative Example 4), and salicylic acid (Comparative Example 5) is, ethanol as a solvent were diluted 3000μg/ml 70%, were diluted 11 - fold dilution series stage was prepared.

[0036] In addition, a mixture of equal parts and resorcin isopropylmethylphenol (example 2), and salicylic acid isopropylmethylphenol mixture of equal parts (example 3) also, a dilution series prepared in the same manner.

[0037] (Measurement of minimum bactericidal concentration (MBC)) each test material in the same manner as in test example 1, was determined MBC. Results are shown in table 2-3 shown.

TABLE 2

TABLE 3

[0040] In addition, by MBC, antimicrobial force can be evaluated. When a low concentration of the test substance to a microbe is strongly influenced by, for increasing the concentration and suppress development occurs, followed by

development is stopped. Further increasing concentration, kills microorganisms will now be described below. At this time expressed as concentration MBC.

[0041] From the results of table 1-3, the microbial composition of the present invention, both relative to the causative agent of the underarm odor to synergistically enhance the antimicrobial activity can be seen.

[0042] ≪ test example 3> table 4-5 (sample preparation) in accordance with the above composition, example 4-6 and Comparative example 6-10 prepared by a conventional method of deodorant agent, was subjected to evaluation. Note that, in the table represents the amount of the % by weight.

[0043] (Test example: evaluation of deodorant agent) which underarm odor is strong name from a male subject 10, each sample of a subject by applying the one beneath the armpit, beneath the armpit of the other for comparison is not applied. 3 Immediately after the coating before and after the following evaluation criteria for each of the odor of judgment, the average value was adopted. Results are shown in table 3 shown.

[0044] ≪ criteria> no smell, 0 point faintly smells, 1 point slightly smells is weak, 2 point clearly smells, 3 point very strongly smells, 4 point

TABLE 4

TABLE 5

[0047] From the results of table 3-4, the present invention deodorant agent and has excellent deodorizing effect, long-lasting effect as can be seen.

[0048] Hereinafter, according to the present invention showing an example in formulating deodorant. In addition, the blending amount in weight %.

[0049] (Example 1 formulation: deodorant spray) 2.25 0.02 0.02 1.31 methylphenyl polysiloxane isononanoic isononyl silica 3.0 1.5 0.1 aluminum chlorohydrate tricolsan 1.7 talc trichlorocarbanilide perfume 0. 1 menthol liquefied petroleum gas residue100. 0 with meter

[0050] (Example 2 formulation: deodorant spray) 2.85 0.01 0.02 0.51 methylphenyl polysiloxane isopropyl myristate isopropylmethylphenol menthol 0.05 zinc oxide 0.5 4.3 silica talc trichlorocarbanilide 1.6 0. 1 perfume liquefied petroleum gas residue100. 0 with meter

[0051] (Example 3 formulation: deodorant spray) ethanol 37.96 1.0 0.1 0.04 0.3 menthol para-phenol sulfonic acid zinc chloride methylphenyl polysiloxane tricolsan 0.2 0. 4 perfume benzalkontum liquefied petroleum gas residue100. 0 with meter

[0052] (Example 4 formulation: deodorant spray) ethanol 38.36 0.05 0.04 1.0 isononyl isononanoic isopropylmethylphenol para-phenol sulfonic acid zinc 0.15 perfume 0. 4 benzalkonium chloride liquefied petroleum gas residue100. 0 with meter

[0053] (Example 5 formulation: deodorant roll-on) ethanol 60.0 1.0 0.2 1.0 10.0 and zinc aluminum chlorohydrate isononanoic isononyl tricolsan perfume 0. 4 0.3 menthol purified water residue100. 0 with meter

[0054] (Example 6 formulation: deodorant roll-on) ethanol 60.0 1.0 0.1 1.0 10.0 and zinc aluminum chlorohydrate isononanoic isononyl isopropylmethylphenol 0.1 perfume 0. 2 menthol purified water residue100. 0 with meter

[0055] (Example 7 formulation: deodorant lotion) ethanol 20.0 polyoxyethylene hydrogenated castor oil 1.0 2.0 0.1 0.2 0.3 zinc para-phenol sulfonic dipropylene isopropylmethylphenol 0. 3 perfumes piroctonolamine purified water residue100. 0 with meter

[0056] (Example 8 formulation: deodorant lotion) 0.5 0.2 30.0 polyoxyethylene polyoxypropylene decyltetradecyl ether ethanol tricolsan 0.2 0.3 0.2 0. 2 zinc para-phenol sulfonic menthol fragrance piroctonolamine purified water residue100. 0 with meter

[0057] (Example 9 formulation: deodorant mist) ethanol 70.0 0.1 0.5 0.1 0.1 benzalkonium chloride menthol perfume 0. 5 allantoin aluminum chlorohydrate trichlorocarbanilide purified water residue100. 0 with meter

[0058] (Example 10 formulation: deodorant mist) ethanol 30.0 polyoxyethylene hydrogenated castor oil 0.5 0.1 0.2 aluminum potassium sulfate 5.0 benzalkonium chloride 0. 5 0.4 menthol fragrance piroctonolamine purified water residue100. 0 with meter

[0059] (Example 11 formulation: deodorant stick) cetanol 5.0 10.0 0.1 10.0 sodium stearate and zinc isopropyl myristate 0.1 15.0 0.4 0. 3 aluminum chlorohydrate trichlorocarbanilide menthol fragrance ethanol residue100. 0 with meter

[0060] (Example 12 formulation: deodorant stick) polyethylene glycol 10.0 0.1 0.2 20.0 25.0 stearyl perfume 0. 3 aluminum chlorohydrate triclocarban piroctonolamine decamethylcyclopentasiloxane residue100. 0 with meter

[0061] (Example 13 formulation: deodorant gel) ethanol 50.0 0.4 1.0 0.5 vinyl cyclic silicone polyether-modified silicone 0.1 0.1 0.1 0.4 triethanolamine tricolsan isopropylmethylphenol perfume suitable silicone powder 1.0 benzalkonium chloride amount purified water residue100. 0 with meter

[0062] (Example 14 formulation: spray) aluminum chlorohydrate anhydrous silicic acid 1.5 3.0 0.5 0.05 1.5 talc Silicone-treated zinc oxide and zinc 0.05 2.0 0.1 isopropyl myristate tricolsan isopropylmethylphenol sorbitan fatty acid ester 0. 3 1.0 dimethyl ester liquefied petroleum gas residue100. 0 with meter

[0063] (Example 15 formulation: liquid deodorant) ethanol 30. 0 1, 3-2.0 3.0 0.1 butylene para-phenol sulfonic acid zinc isopropylmethylphenol resorcin 0.1 polyoxyethylene hydrogenated castor oil (50) 0.5 an appropriate amount of fragrance purified water residue100. 0 with meter

[0064] (Example 16 formulation: air fresheners) salicylic acid 0.2 0. 1 20.0 30.0 purified water and ethanol isopropylmethylphenol dimethyl ether residue100. 0 with meter

Description

[0001] 本発明は抗菌性組成物、並びに該抗菌性組成物を含有したデオドラント剤に関する。

[0002] 人に不快感を与える腋臭などの体臭は、特に、汗が皮脂と混ざり、それが腋臭原因菌により分解されることにより生じるとされている。

[0003] そこで、従来から、体臭の発生を抑制するために、抗菌性化合物がデオドラント剤に用いられてきた(例えば、特許文献1～3を参照)。

[0004] しかし、人の肌や頭皮には汗腺や皮脂腺が存在し、その箇々で汗と皮脂の量が異なり、汗(水分)が多い状態と皮脂が多い状態とでは、抗菌性化合物の抗菌活性が異なる可能性があった。従来のデオドラント剤では、このような肌状態の変化に対応できず、防臭・消臭効果を十分に発揮できないといった問題があった。さらには、肌状態の変化により、防臭・消臭効果が持続して得られないといった問題もあった。

[0005] 一方、トリクロサン及びイソプロピルメチルフェノールを併用することにより、汗が多く分泌した肌状態に加え、皮脂が多く分泌した肌状態でも、腋臭原因菌に対する抗菌活性を維持できるデオドラント剤が報告されている(特許文献4を参照)。

[0006] しかし、上記技術では、異なった肌状態でも腋臭原因菌に対する抗菌活性を維持できることに留まり、抗菌性化合物のそれぞれが有する抗菌活性を相乗的に増強できるものではなく、種々の肌状態でもより効果的に防臭・デオドラント効果を発揮できるデオドラント剤が望まれる。

[0007] 特開2002-60335号公報

特開2003-81801号公報

特開2004-189633号公報

特開2006-96719号公報

[0008] 本発明は、上記従来技術に鑑みてなされたものであって、汗や皮脂の量や比率が異なる様々な肌状態でも、特定の化合物がそれぞれ有する抗菌活性を相乗的に増強できる抗菌性組成物、並びに該抗菌性組成物を含有するデオドラント剤を提供することを課題とする。

[0009] すなわち、本発明は、
〔1〕塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる2種以上の化合物を含有することを特徴とする抗菌性組成物、
〔2〕抗腋臭原因菌用である前記〔1〕に記載の抗菌性組成物、並びに
〔3〕前記〔1〕又は〔2〕に記載の抗菌性組成物を含有してなるデオドラント剤
に関する。

[0010] 本発明にかかる抗菌性組成物は、汗や皮脂の量や比率が異なる様々な肌状態でも、特定の化合物のそれぞれが本来有する抗菌活性を相乗的に増強することができるという効果を奏する。

[0011] しかも、腋臭原因菌に対して優れた抗菌活性を発揮することから、抗腋臭原因菌効果を発揮するデオドラント剤として有用である。

[0012] 本発明にかかる抗菌性組成物は、必須成分として塩化ベンザルコニウム、塩化ベンゼトニウム、ハロカルバン、トリクロロカルバニリド、塩酸クロルヘキシジン、イソプロピルメチルフェノール、トリクロサン、グルコン酸クロルヘキシジン、ジンクピリチオン、ピロクトンオラミン、レゾルシン、塩化リゾチーム、サリチル酸、ヒノキチオール及びチモールからなる群から選ばれる2種以上の特定の化合物を含有する。かかる構成を有することで、腋臭などの体臭に優れた防臭・デオドラント効果を発揮するとともに、その効果を長時間持続することができる。

[0013] 具体的には、腋臭原因菌に対して相乗的な優れた抗菌活性を発揮する観点から、以下の特定の2種又は3種の化合物を組み合わせて含有するのが好ましい。

[0014] トリクロサンとトリクロロカルバニリド、
トリクロサンと塩化ベンザルコニウム、
トリクロサンとジンクピリチオン、
トリクロサンとピロクトンオラミン、
イソプロピルメチルフェノールとトリクロロカルバニリド、
イソプロピルメチルフェノールと塩化ベンザルコニウム、
イソプロピルメチルフェノールとジンクピリチオン、
イソプロピルメチルフェノールとピロクトンオラミン、
イソプロピルメチルフェノールとレゾルシン、
イソプロピルメチルフェノールとサリチル酸、
トリクロロカルバニリドと塩化ベンザルコニウム、
トリクロロカルバニリドとジンクピリチオン、
トリクロロカルバニリドとピロクトンオラミン、
塩化ベンザルコニウムとピロクトンオラミン、
塩化ベンザルコニウムとトリクロサンとイソプロピルメチルフェノール、又は
ジンクピリチオンとトリクロサンとイソプロピルメチルフェノール。

[0015] 特に、トリクロサンとピロクトンオラミン、イソプロピルメチルフェノールとレゾルシン、又はイソプロピルメチルフェノールとサリチル酸の特定の2種を組み合わせて含有するのが好ましい。

[0016] 尚、ピロクトンオラミンとは、一般名称、1-ヒドロキシ-4-メチル-6-(2,4,4-トリメチルペンチル)-2(1H)-ピリドン モノエタノールアミン塩の化合物である。市販品としては、商品名、オクトピロックス(クラリアント・ジャパン社製)として入手が可能である。

[0017] 本発明にかかる抗菌性組成物は、各化合物の含有量は特に限定されないが、第一の化合物1重量部に対し、第二以降の化合物のそれぞれを0.1～10重量部とするのが好ましく、より好ましくは0.2～5重量部、更に好ましくは0.5～2重量部となるように配合する。第一の化合物に対し第二以降の化合物が10重量部を超えて配合すると、また、0.1重量部未満の場合、抗菌活性の相乗的な増強効果が期待できないために好ましくない。

[0018] 例えば、第一の化合物にトリクロサンを選択し、第二のトリクロロカルバニドを選択した場合、トリクロサン1重量部に対し、トリクロロカルバニド0.1～10重量部とするのが好ましい。また、第一の化合物に塩化ベンザルコニウム、第二の化合物にトリクロサン、第三の化合物にイソプロピルメチルフェノールを選択した場合、塩化ベンザルコニウム1重量部に対し、トリクロサン0.1～10重量部、イソプロピルメチルフェノール0.1～10重量部とするのが好ましい。

[0019] 尚、本発明の抗菌性組成物には、上記した化合物の他、本発明の効果を阻害しない範囲で、溶剤、界面活性剤、酸化防止剤、金属イオン封鎖剤等を適宜配合することができる。

[0020] 本発明の抗菌性組成物は、デオドラント剤に配合して使用することができる。その配合量は、特に限定されないが、デオドラント剤中0.001～3重量%、好ましくは0.01～2重量%、より好ましくは0.1～1重量%とするとよい。配合量が0.001重量%未満の場合、抗菌効果に劣るために、また、3重量%を超えて配合すると、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

[0021] 本発明では、更に制汗成分を含有することができる。本発明で用いられる制汗成分は、皮膚を収斂することにより汗の発生を抑制する薬剤であり、例えば、塩化アルミニウム、硫酸アルミニウムカリウム、硫酸アルミニウム、酢酸アルミニウム、クロロヒドロキシアルミニウム、アラントインクロロヒドロキシアルミニウム、パラフェノールスルホン酸亜鉛などを例示することができる。

[0022] 制汗成分の配合量は、本発明の効果を発揮すれば特に限定されず、デオドラント剤中0.1～20重量%とするとよく、0.3～15重量%とすることがより好ましい。0.1重量%未満の配合量では、防臭・デオドラント効果の持続性の効果に劣るために、また、20重量%を超えて配合すると、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

[0023] 本発明にかかるデオドラント剤には、本発明の効果を損なわない範囲であれば、上記した成分の他に、消臭成分を配合することができる。消臭成分とは、臭いを発する物質と反応・吸着したり、臭いをマスクしたりして、臭いを消す効果を有する薬剤であり、例えば、酸化亜鉛などの金属酸化物、アルキルジエタノールアミド、ヒドロキシアパタイト、茶抽出物、香料、酸化防止剤などを例示することができる。

[0024] 消臭成分の配合量は、本発明の効果を発揮すれば特に限定されず、デオドラント剤中0.01～5重量%とするとよく、0.1～2重量%とすることがより好ましい。0.01重量%未満の配合量では、臭いのマスキングの効果に劣るために、また、5重量%を超えて配合すると、香りが強くなりすぎたり、肌への着色が生ずる場合があり、また、皮膚刺激性など安全性に問題が生じる場合があるために、いずれの場合も好ましくない。

[0025] 本発明のデオドラント剤には、本発明の効果を阻害しない範囲で、上記した成分の他、エタノール、イソプロピルアルコール等の低級アルコール;ラウリルアルコール、ステアリルアルコール等の高級アルコール;グリセリン、ジグリセリン、ジプロピレングリコール、1,3-ブタンジオール、1,2-ペンタンジオール、1,2-オクタンジオール等の多価アルコール;ベントナイト、カラギーナン、カルボキシビニルポリマー、ヒドロキシエチルセルロース等の増粘剤;トコフェロール及びその誘導体、アスコルビン酸及びその誘導体等の酸化防止剤;エデト酸塩、リン酸、ポリリン酸ナトリウム等の金属イオン封鎖剤;動物及び植物エキス、クエン酸、酒石酸等のpH調整剤;シリコーン類、保湿剤、界面活性剤、防腐剤、粉体、着色剤、水等を適宜配合することもできる。

[0026] 本発明のデオドラント剤は、ローション、エアゾール、スティック、パウダー、ロールオン、クリーム、ジェル、乳液、シート剤などの種々の形態に用いることができ、製剤化については、一般に知られている方法により製造すればよい。

[0027] <試験例1>
供試菌として、腋臭の原因菌であるスタフィロコッカス エピデルミディス(Staphylococcus epidermidis IAM1296)を用いた。

[0028] (接種菌液の調製)
接種用菌液としてスタフィロコッカス エピデルミディスを寒天培地で35℃で培養後、更にブイヨン培地に移植して35℃で培養した。得られた培養液をブイヨン培地で約10^7個/mLに希釈したものを接種用菌液とした。

[0029] (希釈系列の調製)
ピロクトンオラミン(比較例1)、及びトリクロサン(比較例2)は、70%エタノールを希釈溶媒として1500μg/mLに調製し、10段階に倍倍希釈して希釈系列を調製した。

[0030] また、ピロクトンオラミンとトリクロサンの等量混合物(実施例1)についても、同様に希釈系列を調製した。

[0031] (最小殺菌濃度(MBC)の測定)
上記各被験物質を含む希釈系列1mLに対して各寒天培地9mLをシャーレに入れ、それぞれについて、上記接種用菌液を約1cmの長さに画線した。培養は、35℃で行い5日後の菌の生育の有無を判定した。このとき、生育が認められなかった濃度では、接種した画線から菌を採取し、新鮮寒天培地に移植し更に3日培養した。このとき、

生育が認められなかった最小の濃度をMBCとして求め、そのときの各成分の濃度を表1(トレオレイン非添加)に記した。

[0032] (皮脂成分の影響の検討)
皮脂成分の影響による殺菌効果を検討するために、皮脂成分としてトレオレインを添加して下記試験を実施した。即ち、上記各被験物質を含む希釈系列1mLに対して、0.1重量%のトリオレインと寒天培地8.9mLをシャーレに入れ、それぞれについて、上記と同様に接種用菌液を約1cmの長さに画線した。培養は、35℃で行い5日後の菌の生育の有無を判定した。このとき、生育が認められなかった濃度では、接種した画線から菌を採取し、新鮮寒天培地に移植しさらに3日培養した。このとき、生育が認められなかった最小の濃度をMBCとして求め、そのときの各成分の濃度を表1(トレオレイン添加)に記した。

[0034] <試験例2>
供試菌として、腋臭の原因菌であるコリネバクテリウム ミヌティシマム (Corynebacterium minutissimum ATCC23348)を用い、試験例1と同様にして接種用菌液を調製した。

[0035] (希釈系列の調製)
イソプロピルメチルフェノール(比較例3)、レゾルシン(比較例4)、及びサリチル酸(比較例5)は、70%エタノールを希釈溶媒として3000μg/mLに調製し、11段階に倍倍希釈して希釈系列を調製した。

[0036] また、イソプロピルメチルフェノールとレゾルシンの等量混合物(実施例2)、及びイソプロピルメチルフェノールとサリチル酸の等量混合物(実施例3)についても、同様に希釈系列を調製した。

[0037] (最小殺菌濃度(MBC)の測定)
上記各被験物質を試験例1と同様にして、MBCを求めた。結果を表2～3に示す。

[0039] 尚、MBCによって、抗菌力を評価することができる。被験物質の濃度が薄いときには微生物への影響はないが、濃度を増していくと発育抑制が起こり、続いて発育は停止する。更に濃度が増すと、微生物は死滅していくことになる。このときの濃度がMBCとして表される。

[0041] 表1～3の結果から、本発明の抗菌性組成物は、腋臭原因菌に対して両者の抗菌活性が相乗的に増強されることが分かる。

[0042] <試験例3>
試料の調製)
表4～5記した組成に従い、実施例4～6及び比較例6～10の各デオドラント剤を常法により調製し、下記評価に供した。尚、表中の配合量は重量%を表す。

[0043] (試験例:デオドラント剤の評価)
腋臭が強いと判定された男子被験者10名に、各試料を被験者の一方の腋下に塗布し、もう一方の腋下は比較対照として塗布しなかった。塗布前とその直後及び3時間後のそれぞれについて下記評価基準に従って臭いの判定をし、その平均値を採用した。結果を表3に示す。

[0044] <評価基準>
臭わない・・・・・・・・0点
かすかに臭う・・・・・・1点
やや臭うが弱い・・・・・2点
はっきりと臭う・・・・・3点
非常に強く臭う・・・・・4点

[0047] 表3～4の結果から、本発明のデオドラント剤は優れた防臭効果を有するとともに、その効果も長時間持続することが分かる。

[0048] 以下、本発明にかかるデオドラント剤の処方例を示す。尚、配合量は重量%である。

[0049] (処方例1:デオドラントスプレー)
メチルフェニルポリシロキサン 1.31
イソノナン酸イソノニル 2.25
トリクロサン 0.02
トリクロロカルバニリド 0.02
メントール 0.1
クロルヒドロキシアルミニウム 1.5
シリカ 3.0
タルク 1.7
香料 0.1
液化石油ガス 残分
合計 100.0

[0050] (処方例2:デオドラントスプレー)
メチルフェニルポリシロキサン 0.51
ミリスチン酸イソプロピル 2.88
イソプロピルメチルフェノール 0.01
トリクロロカルバニリド 0.02
メントール 0.05
酸化亜鉛 0.5
シリカ 4.3
タルク 1.8
香料 0.1
液化石油ガス 残分
合計 100.0

[0051] (処方例3:デオドラントスプレー)
エタノール 37.96
メントール 0.3
メチルフェニルポリシロキサン 1.0

トリクロサン 0.1
塩化ベンザルコニウム 0.04
パラフェノールスルホン酸亜鉛 0.2
香料 0.4
液化石油ガス 残分
合計 100.0

[0052] (処方例4:デオドラントスプレー)
エタノール 38.36
イソノナン酸イソノニル 1.0
イソプロピルメチルフェノール 0.05
塩化ベンザルコニウム 0.04
パラフェノールスルホン酸亜鉛 0.15
香料 0.4
液化石油ガス 残分
合計 100.0

[0053] (処方例5:デオドラントロールオン)
エタノール 60.0
イソノナン酸イソノニル 1.0
トリクロサン 0.2
ジンクピリチオン 1.0
クロルヒドロキシアルミニウム 10.0
メントール 0.3
香料 0.4
精製水 残分
合計 100.0

[0054] (処方例6:デオドラントロールオン)
エタノール 60.0
イソノナン酸イソノニル 1.0
イソプロピルメチルフェノール 0.1
ジンクピリチオン 1.0
クロルヒドロキシアルミニウム 10.0
メントール 0.1
香料 0.2
精製水 残分
合計 100.0

[0055] (処方例7:デオドラントローション)
エタノール 20.0
ポリオキシエチレン硬化ヒマシ油 1.0
ジプロピレングリコール 2.0
イソプロピルメチルフェノール 0.1
ピロクトンオラミン 0.2
パラフェノールスルホン酸亜鉛 0.3
香料 0.3
精製水 残分
合計 100.0

[0056] (処方例8:デオドラントローション)
エタノール 30.0
ポリオキシエチレンポリオキシプロピレン
デシルテトラデシルエーテル 0.5
トリクロサン 0.2
ピロクトンオラミン 0.2
パラフェノールスルホン酸亜鉛 0.3
メントール 0.2
香料 0.2
精製水 残分
合計 100.0

[0057] (処方例9:デオドラントミスト)
エタノール 70.0
塩化ベンザルコニウム 0.1
トリクロロカルバニリド 0.1
アラントインクロルヒドロキシアルミニウム 0.5
メントール 0.1
香料 0.5
精製水 残分
合計 100.0

[0058] (処方例10:デオドラントミスト)
エタノール 30.0
ポリオキシエチレン硬化ヒマシ油 0.5
塩化ベンザルコニウム 0.1
ピロクトンオラミン 0.2
硫酸アルミニウムカリウム 5.0
メントール 0.4
香料 0.5
精製水 残分
合計 100.0

[0059] (処方例11:デオドラントスティック)
セタノール 5.0
ステアリン酸ナトリウム 10.0
ミリスチン酸イソプロピル 10.0
トリクロロカルバニリド 0.1

ジンクピリチオン 0.1
クロルヒドロキシアルミニウム 15.0
メントール 0.4
香料 0.3
エタノール 残分
合計 100.0

[0080] (処方例12:デオドラントスティック)
ステアリルアルコール 25.0
ポリエチレングリコール 10.0
トリクロカルバン 0.1
ピロクトンオラミン 0.2
クロルヒドロキシアルミニウム 20.0
香料 0.3
デカメチルシクロペンタシロキサン 残分
合計 100.0

[0081] (処方例13:デオドラントジェル)
エタノール 60.0
カルボキシビニルポリマー 0.4
環状シリコーン 1.0
ポリエーテル変性シリコーン 0.5
トリクロサン 0.1
イソプロピルメチルフェノール 0.1
塩化ベンザルコニウム 0.1
トリエタノールアミン 0.4
シリコーンパウダー 1.0
香料 適量
精製水 残分
合計 100.0

[0082] (処方例14:パウダースプレー)
クロルヒドロキシアルミニウム 3.0
無水ケイ酸 1.5
シリコーン処理タルク 1.5
酸化亜鉛 0.5
トリクロサン 0.05
イソプロピルメチルフェノール 0.1
ジンクピリチオン 0.05
イソプロピルミリスチン酸エステル 2.0
ジメチルポリシロキサン 1.0
ソルビタン脂肪酸エステル 0.3
液化石油ガス 残分
合計 100.0

[0083] (処方例15:液体防臭剤)
パラフェノールスルホン酸亜鉛 2.0
エタノール 30.0
1,3-ブチレングリコール 3.0
レゾルシン 0.1
イソプロピルメチルフェノール 0.1
ポリオキシエチレン(50)硬化ヒマシ油 0.5
香料 適量
精製水 残分
合計 100.0

[0084] (処方例16:消臭スプレー)
精製水 30.0
エタノール 20.0
サリチル酸 0.2
イソプロピルメチルフェノール 0.1
ジメチルエーテル 残分
合計 100.0

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(54) Title: DEODORANT ANTIBACTERIAL POWDER PREPARATION, PROCESS FOR PRODUCING THE SAME AND PRODUCT USING THE POWDER PREPARATION

(54) 発明の名称: 消臭・抗菌性粉末製剤、その製造方法及びこの粉末製剤を利用した製品

(57) Abstract: A deodorant antibacterial powder preparation of 1 to 40 μm average particle diameter produced by hydrating calcium oxide obtained by firing seashells; a deodorant antibacterial powder preparation comprising 5 to 95 mass% of the above powder and 95 to 5 mass% of tea powder or powder of used tea leaves having an average particle diameter of 1 to 40 μm; or a deodorant antibacterial powder preparation comprising 5 to 95 mass% of calcium hydroxide powder of 1 to 40 μm average particle diameter and 95 to 5 mass% of tea powder or powder of used tea leaves having an average particle diameter of 1 to 40 μm. These deodorant antibacterial powder preparations can exert striking deodorant antibacterial effects against various odors and bacteria and can find advantageous application in a wide spectrum of fields.

(57) 要約: 貝殻を焼成して得られた酸化カルシウムを水和してなる、平均粒径１～４０μｍの消臭・抗菌性粉末製剤；この粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤；及び平均粒径１～４０μｍの水酸化カルシウム粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤は、各種臭気、各種細菌に対して、高性能の消臭、抗菌効果を有し、広範囲の分野で好適に使用することができる。

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明 細 書

消臭・抗菌性粉末製剤、その製造方法及びこの粉末製剤を利用した製品

技術分野

本発明は、各種臭気、各種細菌に対して、高性能の消臭、抗菌効果を有する消臭・抗菌性粉末製剤、その製造方法及びこの粉末製剤を利用した製品に関する。

背景技術

近年、合成樹脂を用いた新建材、家具、壁紙、自動車用レザー、繊維製品、食品包装材、飼料袋及び家庭用品などが広く普及している。しかしながら、このような新建材や家具等において、新建材や家具等自体、あるいは接着剤や塗料から発生するホルムアルデヒド等の揮発性物質は、近年、シックハウス症候群の原因として大きな問題となっており、ホルムアルデヒド等の揮発性物質の低減が要望されている。また、快適性の観点から、病原性大腸菌Ｏ－１５７に代表される各種細菌に対して抗菌性を示し、かつ使用上安全な抗菌剤が望まれている。一方、食品包装材及び飼料袋の分野では、熱可塑性フィルムが広く使用されているが、内容物の保存性の観点から、安全で、かつ抗菌性及び抗カビ性を示す抗菌剤が強く求められている。また、トイレの臭いやタバコの臭い等の消臭に効果を発揮する家庭用消臭剤が求められている。

安全な消臭・抗菌剤として、貝殻を利用したものが提案されており、例えば、貝殻を粉砕して得た炭酸カルシウム粉末を含有する消臭剤（例えば、特開２００１－１４５６９３号公報参照）、ホタテ貝の貝殻粉末を含有する抗菌剤（例えば、特開２００１－１９９８２３号公報参照）、ホタテ貝

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殻の粉砕物を焼成してなる細菌抑制剤（例えば、特開２００２－２５５７１４号公報参照）が提案されている。しかながら、その消臭性能や抗菌性能は、ともに十分ではなかった。

発明の開示

本発明は、上記事情に鑑みなされたもので、各種臭気、各種細菌に対して、高性能の消臭、抗菌効果を有し、広範囲の分野で使用可能な消臭・抗菌粉末を提供することを目的とするものである。

本発明者らは、上記課題を解決するために鋭意研究を重ねた結果、貝殻を焼成して得られた酸化カルシウムを水和した水和物であって、特定の平均粒径を有する酸化カルシウム水和物粉末が、消臭・抗菌に有効であることを見出した。本発明はかかる知見に基づいて完成したものである。

すなわち、本発明の要旨は下記のとおりである。

（１）　貝殻を焼成して得られた酸化カルシウムを水和してなる、平均粒径１～４０μｍの消臭・抗菌性粉末製剤。

（２）　上記（１）の粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤。

（３）　平均粒径１～４０μｍの水酸化カルシウム粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤。

（４）　貝殻を８００～１３００℃で焼成して酸化カルシウムを得、該酸化カルシウムを水和して消臭・抗菌性粉末製剤を製造する方法において、酸化カルシウムの０．０５～０．１５質量％水溶液のｐＨが９～１３となるように水和することを特徴とする消臭・抗菌性粉末製剤の製造方法。

（５）　上記（４）の製造方法において、貝殻を焼成して得られた焼成物を１００μｍ以下の粒径となるように粉砕し、該粉砕物１ｋｇに対して水

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０．２～１リットルの割合で加えて酸化カルシウムを水和させることを特徴とする消臭・抗菌性粉末製剤の製造方法。

（６）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤を０．１～３０質量％配合してなる合成樹脂組成物。

（７）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤を含む表面処理剤により表面処理されてなる消臭・抗菌性レザーであって、消臭・抗菌性粉末製剤の配合量が、表面処理剤の乾燥質量基準で１～３０質量％である消臭・抗菌性レザー。

（８）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤を乾燥質量基準で０．１～３５質量％配合してなる塗料。

（９）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤を乾燥質量基準で１～８０質量％配合してなる繊維処理剤。

（１０）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤を含む処理剤により処理されてなる消臭・抗菌性繊維であって、消臭・抗菌性粉末製剤の配合量が、処理剤の乾燥質量基準で１～７０質量％である消臭・抗菌性繊維。

（１１）　上記（６）の合成樹脂組成物からなる消臭・抗菌性フィルム。

（１２）　上記（６）の合成樹脂組成物を発泡させてなる消臭・抗菌性発泡材。

（１３）　上記（６）の合成樹脂組成物からなる消臭・抗菌性防草シート。

（１４）　上記（１）～（３）のいずれかの消臭・抗菌性粉末製剤であって、平均粒径１～１０μｍの消臭・抗菌性粉末製剤を０．５～２０質量％配合してなる熱可塑性樹脂組成物製不織布。

（１５）　平均粒径３～２０μｍの酸化カルシウム水和物粉末を、繊維に対する乾燥質量１～５０ｇ／ｍ^2 で用いて繊維を裏処理してなる消臭・抗菌性付与繊維。

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（１６） 上記（１０）又は（１５）に記載の消臭・抗菌性付与繊維により形成されてなるカーシート又は自動車天井材。

（１７） 上記（１）～（３）のいずれかに記載の消臭・抗菌性粉末製剤及び平均粒径１～４０μｍの酸化カルシウム水和物粉末からなる消臭・抗菌性混合粉末製剤を含有してなる合板。

発明を実施するための最良の形態

本発明の消臭・抗菌性粉末製剤において用いる貝殻としては、ほたて貝、あさり、はまぐり、牡蠣、さざえ、あわび及びムール貝等の貝殻が挙げられる。このうち、工業的に処理されている割合が高いほたて貝及び牡蠣の貝殻が、入手しやすい点から好適であり、さらに、不純物の含有量が少なく、かつ大量の廃棄物を有効利用する観点から、ほたて貝の貝殻が好適である。貝殻を焼成して得られる酸化カルシウムには、亜鉛、鉄、マグネシウム等の微量金属が含まれているので、抗菌性付与の点で好適である。

貝殻の焼成は、貝殻を水で洗浄した後、８００～１３００℃で、３～６時間行う。焼成後、焼成物から細かい砂等を除去し、水を加えて酸化カルシウムを水和させる。水和後、５～４０℃程度に冷却して沈殿物を得、この沈殿物を遠心分離機又はろ過機により分離し、得られた分離物を乾燥させ、分級することにより、所定の平均粒径を有する酸化カルシウム水和物が得られる。

上記焼成は、得られる酸化カルシウムの消臭・抗菌性能を向上させる点から、高温で行うことが重要であり、焼成温度が８００℃未満であると、焼成が十分に行われないため、消臭・抗菌性能が低下するおそれがある。１３００℃を超える温度で焼成しても、消臭・抗菌性能がより向上するものでもなく、エネルギーが無駄に消費されるだけである。焼成条件は、好

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ましくは９００～１２００℃で３～５時間である。焼成物は、砂等を取り除いた後に粒径１００μｍ以下に粉砕することが好ましい。粉砕には、ボールミル及びジェットミル等を用いることができる。また、上記水和の際に加える水の量は、焼成物１ｋｇに対して０．２～１リットルの割合とすることが好ましい。

上記焼成及び水和により得られる酸化カルシウム水和物は、その０．０５～０．１５質量％水溶液のｐＨが９～１３、好ましくは１０～１３であることが、高い消臭・抗菌性能を示す上で重要である。例えばほたての貝殻を用いる場合、温度８００～１３００℃で焼成して得られた焼成物を粒径１００μｍ以下となるように粉砕し、この粉砕物１ｋｇに対して水を０．２～１リットルの割合で加えて水和させ、水和後の冷却温度を５～４０℃とすることにより、上記ｐＨを有する酸化カルシウム水和物を得ることができる。

本発明において、酸化カルシウム水和物粉末の平均粒径は１～４０μｍであることを要する。用途によっても異なるが、通常は、好ましくは２～３０μｍ、より好ましくは３～２０μｍ、さらに好ましくは５～１５μｍである。この平均粒径が１μｍ未満であると、酸化カルシウム水和物粉末が凝集しやすくなるため、取り扱い難くなり、また１μｍ未満に微粉化するのにも困難を伴う。この平均粒径が４０μｍを超えると、酸化カルシウム水和物粉末の表面積が小さくなるため、消臭・抗菌性能が発現されず、また、粒径が４０μｍを大幅に超えるものも含まれることとなるので、配合時に異物となる。

酸化カルシウム水和物粉末の平均粒径は、ボールミルの粉砕時間又はジェットミルによる粉砕圧力及び粉砕時間の調整により、調整することができる。

本発明においては、酸化カルシウム水和物粉末に、茶粉末又は茶殻粉末

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を混合することが、消臭・抗菌の対象となる物質の範囲が拡大する点から好ましい。具体的には、いわゆるシックハウスの原因となるホルムアルデヒドはもちろんのこと、アンモニア，酢酸臭等の生活臭、タバコの臭いも消臭対象となる。

茶としては、例えば、緑茶、ウーロン茶、紅茶、麦茶及びプーアール茶等の各種茶が挙げられ、消臭・抗菌性能に優れる点から、緑茶、紅茶及びウーロン茶が好適である。茶は一種を単独で用いてもよく、二種以上を混合して用いてもよい。また、茶粉末と茶殻粉末とを混合して用いてもよい。

茶粉末又は茶殻粉末は、茶又は茶殻を乾燥させた後、ボウルミルやジェットミルで粉砕することにより得ることができる。これらの粉末は、平均粒径が１～４０μｍであることが好ましく、分散性及び消臭・抗菌性能向上の点から、５～３５μｍが好ましい。茶粉末又は茶殻粉末の平均粒径の調整は、上記酸化カルシウム水和物粉末における平均粒径の調整と同様に行うことができる。

上記酸化カルシウム水和物粉末と茶粉末又は茶殻粉末の使用割合は、酸化カルシウム水和物粉末５～９５質量%、茶粉末又は茶殻粉末９５～５質量%が好ましい。この使用割合は、消臭の対象となる臭気、抗菌の対象となる菌の種類により調整する。例えば、ホルムアルデヒド、酢酸、硫化水素等の酸性臭気に対しては、酸化カルシウム水和物粉末の使用割合を増やすのが効果的であり、その他の臭気の場合は逆である。また、着色を避けたい用途の場合には、茶粉末又は茶殻粉末の使用割合を減少させることが好ましいが、５質量%未満であると、アンモニア臭やタバコ臭に対する消臭効果が十分ではない。また、茶粉末又は茶殻粉末の使用割合が９５質量%を超えると、ホルムアルデヒドや酢酸に対する消臭効果が低下する。抗菌性能については、茶粉末又は茶殻粉末は、枯草菌、黄色ブドウ球菌及び大腸菌等の広範囲の菌に対して抗菌性能を示すが、黄色ブドウ球菌及び大

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腸菌に対しては、酸化カルシウム水和物粉末の使用割合を増やすのが効果的である。

上記茶粉末又は茶殻粉末は、上記酸化カルシウム水和物粉末と混合する代わりに、工業的に製造される水酸化カルシウム粉末と混合することもできる。水酸化カルシウム粉末の平均粒径は、上記酸化カルシウム水和物粉末の平均粒径と同様に１～４０μｍであることを要する。用途によっても異なるが、通常は、好ましくは２～３０μｍ、より好ましくは３～２０μｍ、さらに好ましくは５～１５μｍである。水酸化カルシウム粉末と茶粉末又は茶殻粉末の使用割合は、上記酸化カルシウム水和物粉末と茶粉末又は茶殻粉末の使用割合と同様であり、対象とする臭気や菌により、上記と同様に調整することができる。

本発明の消臭・抗菌性粉末製剤、すなわち、酸化カルシウム水和物粉末、酸化カルシウム水和物粉末と茶粉末又は茶殻粉末との混合粉末、及び水酸化カルシウムと茶粉末又は茶殻粉末との混合粉末は、合成樹脂に配合することにより、合成樹脂で形成する各種製品における消臭・抗菌に役立てることができる。この場合、消臭・抗菌性粉末製剤の配合割合は、０．１～３０質量％が好ましく、０．２～２０質量％がより好ましく、０．５～１０質量％がさらに好ましい。この配合割合が０．１質量％未満では、消臭・殺菌効果が期待できず、３０質量％を超えると、皮膚に悪影響を及ぼすおそれがある。

合成樹脂としては、耐アルカリ性に劣るものでなければよい。食品用包装袋や飼料袋等に使用する場合は、ポリエチレン、ポリプロピレン、ＰＶＣ（ポリ塩化ビニル）及びポリ塩化ビニリデン等が挙げられる。壁紙には、ポリ塩化ビニル及び軟質ポリオレフィンが好適に使用される。食品用包装袋、飼料袋及び壁紙の場合は、フィルム状のものを用い、その厚さは用途により異なるが、１５～１００μｍが好適である。

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射出成形品には、ポリエチレンやポリプロピレン等のポリオレフィン系樹脂はもちろんのこと、ＡＢＳ（アクリロニトリルーブタジエンースチレン）樹脂やポリスチレン樹脂等のスチレン系樹脂、ポリアクリロニトリル樹脂やポリフェニレンスルフィド樹脂等の熱可塑性樹脂が好適に使用される。射出成形法以外の成形法の場合、フェノール樹脂，メラミン樹脂，エポキシ樹脂及び尿素樹脂等の熱可塑性硬化樹脂が使用される。さらに、エチレンープロピレン共重合体やスチレンーブタジエン共重合体等の熱可塑性エラストマーも使用することができる。

本発明の消臭・抗菌性粉末製剤を、ポリエステル繊維、レーヨン繊維、アクリル繊維及びポリプロピレン繊維等の繊維製品の、消臭・抗菌性付与繊維処理剤として使用する場合、裏処理剤に配合することが、使用の容易性の点から好ましく、特に、自動車用シート、家具用シート、絨毯の消臭・抗菌性付与繊維処理剤として好適に使用される。処理剤の基剤としては、アクリル系樹脂や合成ゴムラテックス等を使用することができる。処理剤は、この基剤に消臭・抗菌性粉末製剤を配合したものである。

処理剤における消臭・抗菌性粉末製剤の配合量は、処理剤の乾燥質量基準で１～８０質量％が好ましく、５～６５質量％がさらに好ましい。この配合量が１質量％未満であると、消臭・抗菌効果が乏しく、８０質量％を超えると消臭・抗菌性粉末製剤を配合する際に処理剤が硬くなり、繊維製品の風合いが損なわれることとなる。なお、消臭・抗菌性粉末製剤の平均粒径は１～４０μｍであることを要し、好ましくは２～３０μｍ、より好ましくは３～２０μｍ、さらに好ましくは５～１５μｍである。

裏処理剤としての使用量は、要求特性にもよるが、乾燥質量で１～５０ｇ／m^2が好ましく、５～３０ｇ／m^2がさらに好ましい。この使用量が１ｇ／m^2未満では、消臭性能が十分ではなく、５０ｇ／m^2を超えると、繊維が硬くなるおそれがあり、コストも上昇する。

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なお、本処理剤は、ポリエチレン、ポリプロピレン、レーヨン、ナイロン、ビニロン及びポリエステル等を含む熱可塑性樹脂組成物からなる不織布の処理剤としても広く使用することができる。

本発明の消臭・抗菌性粉末製剤をレザーの消臭・抗菌剤として使用する場合、消臭・抗菌性粉末製剤をウレタン樹脂等に配合して表面処理剤とし、これをレザー表面に、バーコーター及びロールコーター等により塗布すればよい。レザーとしては、ＰＶＣレザー、ウレタンレザー、オレフィン系熱可塑性エラストマーレザー、及び天然皮革などが挙げられる。天然皮革以外のものは、基材層に表皮層が形成されたものであり、基材層を形成する素材としては、ＰＶＣ、ウレタン及びオレフィン系熱可塑性エラストマー等が挙げられる。消臭・抗菌性効果を向上させるために、基材層にも消臭・抗菌性粉末製剤を含有させることができる。表面処理剤における消臭・抗菌性粉末製剤の配合量は、表面処理剤の乾燥質量基準で１～３０質量％が好ましく、５～２０質量％がさらに好ましい。この配合量が１質量％未満であると、消臭・抗菌効果が乏しく、３０質量％を超えると表皮層の物性が低下する。

本発明の消臭・抗菌性粉末製剤を、各種椅子、ソファー及び自動車シートのクッション材に使用される軟質ウレタンフォームに配合したものは、消臭・抗菌クッション材として好適に使用される。また、上記軟質ウレタンフォームの他、ポリエチレンやポリプロピレン等の熱可塑性ポリオレフィン等のポリオレフィン樹脂、ポリスチレン樹脂及び硬質ウレタン等を発泡させた発泡材は、家庭用品及び建築物用の断熱材として使用される。これらのクッション材や発泡材における消臭・抗菌性粉末製剤の配合量は、クッション材や発泡材の乾燥質量基準で１～３０質量％が好ましく、５～２０質量％がさらに好ましい。この配合量が１質量％未満であると、消臭・抗菌効果が乏しく、３０質量％を超えるとクッション性や断熱性が低下

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する。なお、発泡材は公知の方法で製造することができる。

本発明の消臭・抗菌性粉末製剤は、防草シートに配合することもできる。消臭・抗菌防草シートは、上述した食品包装用袋等と同様の素材からなり、消臭・抗菌性粉末製剤が配合されたフィルム（厚さは限定されないが、好ましくは２０～５０μｍ程度）をヤーン状に切断したものを編み込んだものであり、公知の方法で製造することができる。消臭・抗菌防草シートは、農地の畔、露地、鉢物の下敷き等の用途に好適である。また、消臭・抗菌防草シートは、ポリプロピレン等からなる不織布に消臭・抗菌性粉末製剤を配合したものであってもよい。

本発明の消臭・抗菌性粉末製剤の用途としては、建材用途、自動車関連用途、家庭用品用途も挙げられ、具体的には、建材用途として、畳用張地、畳用芯材、防水防湿フィルム、ガラス保護フィルム、合板、化粧合板、パーティクルボード、ファイバーボード、チップボード、集成材、パネル、ふすま及び障子紙等に配合し、あるいはフローリング用建築仕上げ材として配合して使用することができる。自動車関連用途としては、天井材、内装用塗料及びエアコン用フィルター等が挙げられる。家庭用品用途としては、クッションカバー、クッション材、カーテン、テーブルクロス及び各種敷き材等が挙げられる。

また、本発明の消臭・抗菌性粉末製剤を、フローリング用ワックスに配合したものは、消臭・抗菌性ワックスとして好適に使用することができる。

さらに、本発明の消臭・抗菌性粉末製剤は、消臭・抗菌塗料、特に抗菌ＯＡ機器用塗料、家電製品用塗料及び家具用塗料等に配合して好適に使用することができる。

塗料は、溶剤系樹脂及び水系樹脂に、本発明の消臭・抗菌性粉末製剤を、乾燥質量基準で０．１～３５質量％配合することにより得ることができる。この場合、消臭・抗菌性粉末製剤は、平均粒径１～４０μｍの範囲であれ

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ば使用可能であるが、好ましくは平均粒径１～２０μｍであり、３～１５μｍがより好ましい。この平均粒径が１μｍ未満であると粉末が凝集しやすく、４０μｍを超えると、塗膜に凹凸が発生しやすくなる。さらに、最大粒径が５０μｍ以下であることが、平滑な表面を得る上で好ましい。

溶剤系樹脂としては、アクリル樹脂、ウレタン樹脂、アルキド樹脂及びアミノ系樹脂等が挙げられ、これらは一種を単独で用いてもよく、二種以上を混合して用いてもよい。水系樹脂としては、酢酸ビニル系エマルジョン、アクリル系エマルジョン、ウレタン系エマルジョン、水溶性アクリル樹脂、水溶性ウレタン樹脂、水溶性エポキシ樹脂及び水溶性アルキド樹脂等が挙げられ、これらは一種を単独で用いてもよく、二種以上を混合して用いてもよい。

これらの塗料の塗装対象物としては、金属、ガラス、樹脂、紙、セラミックス及び木材等が挙げられ、任意に選定することができる。

塗装方法としては、はけ塗り、吹き付け塗装、静電塗装及び粉体塗装等があり、適宜選定できる。これらの塗料は、通常の塗料の物性を損なうことなく、抗菌性や消臭性が付与されたものである。

消臭・抗菌性不織布は、上述のように、繊維処理材による方法の他に、不織布の原料繊維を紡糸する段階で、常法により消臭・抗菌性粉末製剤を配合することにより得ることができる。消臭・抗菌性粉末製剤の配合量は、０．５～２０質量％とすることが、消臭・抗菌性効果及び成形性の点から適切である。不織布の製造方法は、特に限定されるものではなく、乾式法や湿式法等の公知の方法により製造することができる。具体的な製造方法としては、例えば、ニードルパンチ機を用いて繊維同士を絡ませたり、糸を用いて縫い止める等の機械的接合方法、熱、圧力又はバインダーによる接着方法等が挙げられる。

この場合、微細な繊維として押し出すため、消臭・抗菌性粉末製剤は、

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平均粒径１～１０μｍが好ましく、２～５μｍがより好ましい。この平均粒径が１μｍ未満であると粉末が凝集しやすく、加工上問題がある。平均粒径が１０μｍを超えると、紡糸時に糸が切断しやすくなり、細い繊維を得ることが困難となる。

不織布の原料繊維としては、ポリエチレン、ポリプロピレン及びナイロン等が好ましく使用されるが、消臭・抗菌性粉末製剤を原料に配合できるものであればよく、特に限定されるものではない。

実施例

次に、本発明を実施例及び比較例により更に具体的に説明するが、本発明はこれらの例によってなんら限定されるものではない。

実施例１

ほたての貝殻を水で洗浄した後、１１００℃にて４時間焼成し、メッシュサイズ５ｍｍの篩を用いて細かい砂を除去し、焼成物１ｋｇを得た。これに水０．５リットルを加えて酸化カルシウムを水和させ、２５℃に冷却し、沈殿物を吸引ろ過器を用いて分離し、分離物を１２０℃にて５時間かけて乾燥させた。この乾燥物をエアセパレーターにて分級し、平均粒径９μｍの酸化カルシウム水和物粉末（消臭・抗菌性粉末製剤）を得た。

内容積３００ミリリットルの三角フラスコに１質量％ホルムアルデヒド水溶液を１．５マイクロリットル入れ、上記酸化カルシウム水和物粉末０．０５ｇ（１ｇ／リットル水溶液でのｐＨが１２．５）を入れ、２４℃にて１２０分間放置した後、北川式検知器によりフラスコ内のホルムアルデヒド濃度を測定した。また、後述する方法により、抗カビ試験を行った。結果を表１に示す。

実施例２

緑茶葉を、水分量が０．５質量％となるように乾燥させ、シュレッダー

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にて粗粉砕し、ボールミル及びジェットミルにてさらに粉砕し、平均粒径３０μｍの茶粉末を得た。この茶粉末５０質量％と、実施例１で得られた酸化カルシウム水和物粉末５０質量％とを混合した消臭・抗菌性混合粉末製剤（１ｇ／リットル水溶液でのｐＨが１１）０．０５ｇを使用して、実施例１と同様にホルムアルデヒド濃度を測定した。また、抗カビ試験を行った。結果を表１に示す。

実施例３

酸化カルシウム水和物粉末の代わりに平均粒径４μｍの水酸化カルシウム（１ｇ／リットル水溶液でのｐＨが１１）を用いた以外は、実施例２と同様にして消臭・抗菌性混合粉末製剤を得、同様にホルムアルデヒド濃度を測定した。また、抗カビ試験を行った。結果を表１に示す。

実施例４

実施例２で得られた消臭・抗菌性混合粉末製剤をポリカーボネート系ウレタン樹脂（大日精化工業社製，レザーミンＤ６０２５）に配合し、消臭・抗菌表面処理剤を作製した。混合粉末製剤の配合量は、表面処理剤の乾燥質量基準で２０質量％とした。ポリエステル繊維６５質量％とレーヨン繊維３５質量％との混紡繊維の平織物からなる基布上に、軟質ポリ塩化ビニル層が形成されたＰＶＣレザーの表面に、バーコーターにて上記表面処理剤を塗布し、乾燥させて厚さが約１０μｍの表面処理層を有する消臭・抗菌処理レザーを得た。このレザーから５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

実施例５

実施例３で得られた消臭・抗菌性混合粉末製剤をアクリルエマルジョン（ガンツ化成（株）製，クロスレン）に配合し、繊維処理剤を作製した。混合粉末製剤の配合量は、繊維処理剤の乾燥質量基準で１５質量％とした。

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ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、乾燥させて厚さが約１００μｍの繊維処理剤層（裏材）を有するシートを得た。このシートから５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

実施例６

実施例４で作製した消臭・抗菌処理レザーから切り出した５ｃｍ×５ｃｍのサンプルと、４質量％アンモニア水溶液５マイクロリットルを内容積３００ミリリットルの三角フラスコに入れ、２５℃にて１２０分間放置した後、北川式検知器によりフラスコ内のアンモニア濃度を測定したところ、１４５質量ｐｐｍであった。

実施例７

実施例１で得られた酸化カルシウム水和物粉末１質量部と、直鎖低密度ポリエチレン（出光石油化学（株）製，ＩＤＥＭＩＴＳＵ　ＬＬ　０１３４，メルトフローレート：１．２ｇ／１０分）９９質量部とをドライブレンドし、その後、混練用二軸押出機（混練部＝４０ｍｍφ）にて２００℃において混練し、樹脂組成物を得た。この樹脂組成物を、押出機（押出部＝５０ｍｍφ）にてインフレーション成形し、幅６０ｍｍ、厚さ４０μｍのインフレーションフィルムを作製した。このフィルムから５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。

実施例８

実施例１で得られた酸化カルシウム水和物粉末を溶剤系ウレタン樹脂溶液中に、乾燥質量基準で５質量％配合し、塗料を調製した。次に、この塗料をスプレーガン（岩田塗装機工業（株）製）により、厚さ２ｍｍのＡＢＳ（アクリロニトリルーブタジエンースチレン）板に対して吹き付け塗装を行った。このＡＢＳ板から５ｃｍ×５ｃｍのサンプルを切り出し、実施

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例１と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

実施例９

実施例１で得られた酸化カルシウム水和物粉末、ポリオール成分、イソシアネート成分、発泡剤、触媒及び整泡剤を下記に示す割合で配合して、常法により、酸化カルシウム水和物粉末を５質量％含む消臭・抗菌性ウレタンフォームを作製した。このウレタンフォームから５ｃｍ×５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

＜配合成分＞

ポリオール（ダウポリウレタン社製，＃３０００）	１００質量部
イソシアネート（ダウポリウレタン社製，Ｔ－８０）	４０質量部
発泡剤（水）	２．９質量部
アミン系触媒（エアプロダクツ社製，３３ＬＶ）	０．３質量部
アミン系触媒（エアプロダクツ社製，ＡＴ３３）	０．３質量部
スズ系触媒（日東化成社製，Ｔ－９）	０．３質量部
整泡剤（日本ユニカー社製，Ｌ６２０２）	０．３質量部
酸化カルシウム水和物粉末	８質量部

実施例１０

実施例１で得られた酸化カルシウム水和物粉末を分級して平均粒径３μｍの粉末を得、これをポリプロピレン樹脂（ＭＩ＝５０ｇ／１０分）に配合して、酸化カルシウム水和物粉末を４０質量％含むマスターバッチを得た。このマスターバッチ５質量部をポリプロピレン樹脂（ＭＩ＝５０ｇ／１０分）９５質量部に配合し、酸化カルシウム水和物粉末を２質量％含む組成物を調製した。この組成物を、押出機に投入し、樹脂温度２２０℃で溶融混練した後、混練物をダイ（２００ホ

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ール）から押し出し、高速エアーで延伸し、細化することにより、連続した長繊維フィラメントを作製した。なお、ＭＩ（メルトインデックス）は、ＪＩＳ　Ｋ　６９２１に準拠し、２３０℃、荷重２．１６ｋｇｆ（２１．１８Ｎ）で測定した。

このフィラメントをランダムに集積してシートとし、このシートをエンボスロールと平滑ロールとの間に通し、１４０℃にて熱エンボスして、不織布を作製した。

得られた不織布は、平均繊維径２２．４μｍ、平均目付５０ｇ／ｍ^{2}であった。この不織布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

比較例１

実施例１において、酸化カルシウム水和物粉末を使用しないでホルムアルデヒド濃度を測定した。また、抗カビ試験を行った。結果を表１に示す。

比較例２

実施例１において、酸化カルシウム水和物粉末を平均粒径１μｍの水酸化マグネシウム（１ｇ／リットル水溶液でのｐＨが７）に変えた以外は、実施例１と同様にホルムアルデヒド濃度を測定した。また、抗カビ試験を行った。結果を表１に示す。

比較例３

実施例１において、焼成温度を６００℃とし、焼成後の水和工程を経ないこと以外は実施例１と同様にして得られた平均粒径３０μｍの粉末（１ｇ／リットル水溶液でのｐＨが８）について、実施例１と同様にホルムアルデヒド濃度を測定した。また、抗カビ試験を行った。結果を表１に示す。

比較例４

実施例５において、消臭・抗菌性混合粉末製剤を使用しない以外は、実

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施例５と同様にホルムアルデヒド濃度を測定した。結果を表１に示す。

比較例５

実施例６において、消臭・抗菌処理レザーの代わりに、実施例４で用いたものと同様で消臭・抗菌性混合粉末製剤を配合しないＰＶＣレザーを使用した以外は、実施例５と同様にアンモニア濃度を測定したところ、２３０質量ｐｐｍであった。

[抗カビ試験]

１．供試菌株：Ｍｕｃｏｒ属糸状菌

２．使用培地：

①培地１：グルコース無機塩培地

グルコース	３０．０ｇ
硝酸アンモニウム	３．０ｇ
燐酸一カリウム	１．０ｇ
硫酸マグネシウム	０．５ｇ
塩化カリウム	０．２５ｇ
硫酸第一鉄	０．００２ｇ
脱イオン水	１０００ミリリットル

②培地２：グルコース無機塩寒天培地

グルコース	３０．０ｇ
硝酸アンモニウム	３．０ｇ
燐酸一カリウム	１．０ｇ
硫酸マグネシウム	０．５ｇ
塩化カリウム	０．２５ｇ
硫酸第一鉄	０．００２ｇ
脱イオン水	１０００ミリリットル
寒天	２５．０ｇ

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３．胞子液の調製：

Ｍｕｃｏｒ属糸状菌をＰＤＡ（ポテトデキストロース寒天培地）プレート上で胞子形成（２８℃で一週間培養）させたものに対し、滅菌水１０ミリリットルを用いて胞子／菌糸懸濁液を調製した。

次いで、これを滅菌脱脂綿で濾過した後、遠心分離により胞子を沈殿させ、上記培地１（寒天抜き、グルコース無機塩培地）２０ミリリットルに再懸濁することによって胞子液を調製した。

４．試験片の設置及び植菌

上記培地２（グルコース無機塩寒天培地）上に、実施例１～３及び比較例１～３で使用した消臭・抗菌性粉末製剤の５ｇ／リットル水溶液（０．５質量%）１ミリリットルをプレート上に塗布した。

これに上記胞子液を試験片上で微細水滴が形成される程度に噴霧することにより植菌し、プレートをパラフィンフィルムでシールした後、恒温恒室槽で培養した。噴霧は噴霧器と２連球を用いて行い、培養条件は、培養温度３０℃、湿度９５%、培養期間は４週間とした。

５．糸状菌生育観察：

植菌してから２週間後に、試験プレート表面上の菌糸の生育度合いを目視観察した。

６．結果の判定：

以下に示す判定基準により、菌糸の生育状況の経時変化を判定した。

[生育判定基準]

－：肉眼において、菌糸（カビ）の生育が認められない

＋／－：菌糸の生育が僅かに認められる

＋：菌糸の生育が認められる

＋／＋：菌糸の顕著な生育及び胞子形成が認められる

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表1

	ホルムアルデヒド(質量ppm)	2週間後のカビ発生状況
実施例1	1	−
実施例2	3	−
実施例3	3	+/−
実施例4	1	
実施例5	ND	
実施例7	5	
実施例8	5	
実施例9	6	
実施例10	5	
比較例1	28	+/+
比較例2	25	+/+
比較例3	25	+
比較例4	15	

ND;検知せず。

実施例１１

平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で２４ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、０．５質量ｐｐｍ未満であった。

実施例１２

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平均粒径５μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で２６ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、０．５質量ｐｐｍ未満であった。

実施例１３

平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で５ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、０．５質量ｐｐｍ未満であった。

実施例１４

平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、酸化カルシウム水和物粉末濃度が２質量％の分散液を作製した。この分散液を、３０ｃｍ×３０ｃｍ×１２ｍｍの合板上にスプレーを用いて塗布し、酸化カルシウム水和物粉末が２ｇ／９００ｃｍ^{2}で塗布された、ホルムア

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ルデヒド発生低減合板を製造した。この合板のホルムアルデヒド放散量をＪＩＳ　Ａ　１９０１に規定される方法で測定したところ、０．２ｍｇ／リットルであった。一方、未処理の同一材質合板のホルムアルデヒド放散量は０．５ｍｇ／リットルであった。

比較例６

水９０質量％、平均粒径８μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）１０質量％の割合で混合した分散液を、湿式ビーズミル（アシザワファインテック（株）製、ＬＭＺ－２）にて湿式粉砕し、平均粒径０．６μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を得た。この消臭・抗菌粉末製剤をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で２７ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布は、消臭・抗菌粉末製剤が凝集し、実使用に耐え得るものではなかった。

比較例７

平均粒径８μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を分級して得られた平均粒径４５μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を、アクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層しようとしたところ、繊維処理剤が固液分離し、積層することができなかった。

比較例８

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平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で０．５ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、２質量ｐｐｍであった。

比較例９

平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジョン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量％とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で５８ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍ×５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。この濃度は０．５質量ｐｐｍ未満であったが、得られた消臭・抗菌性付与ポリエステル基布の風合いを確認したところ、非常に硬く、実使用に耐え得るものではなかった。

産業上の利用可能性

本発明の消臭・抗菌性粉末は、各種臭気、各種細菌に対して、高性能の消臭、抗菌効果を有し、広範囲の分野で使用可能なものである。

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請求の範囲

1．貝殻を焼成して得られた酸化カルシウムを水和してなる、平均粒径１～４０μｍの消臭・抗菌性粉末製剤。

2．請求の範囲第１項に記載の粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤。

3．平均粒径１～４０μｍの水酸化カルシウム粉末５～９５質量％と、平均粒径１～４０μｍの茶粉末又は茶殻粉末９５～５質量％とからなる消臭・抗菌性粉末製剤。

4．貝殻を８００～１３００℃で焼成して酸化カルシウムを得、該酸化カルシウムを水和して消臭・抗菌性粉末製剤を製造する方法において、酸化カルシウムの０．０５～０．１５質量％水溶液のｐＨが９～１３となるように水和することを特徴とする消臭・抗菌性粉末製剤の製造方法。

5．請求の範囲第４項に記載の製造方法において、貝殻を焼成して得られた焼成物を１００μｍ以下の粒径となるように粉砕し、該粉砕物１ｋｇに対して水０．２～１リットルの割合で加えて酸化カルシウムを水和させることを特徴とする消臭・抗菌性粉末製剤の製造方法。

6．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤を０．１～３０質量％配合してなる合成樹脂組成物。

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７．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤を含む表面処理剤により表面処理されてなる消臭・抗菌性レザーであって、消臭・抗菌性粉末製剤の配合量が、表面処理剤の乾燥質量基準で１～３０質量％である消臭・抗菌性レザー。

８．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤を乾燥質量基準で０．１～３５質量％配合してなる塗料。

９．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤を乾燥質量基準で１～８０質量％配合してなる繊維処理剤。

１０．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤を含む処理剤により処理されてなる消臭・抗菌性繊維であって、消臭・抗菌性粉末製剤の配合量が、処理剤の乾燥質量基準で１～７０質量％である消臭・抗菌性繊維。

１１．請求の範囲第６項に記載の合成樹脂組成物からなる消臭・抗菌性フィルム。

１２．請求の範囲第６項に記載の合成樹脂組成物を発泡させてなる消臭・抗菌性発泡材。

１３．請求の範囲第６項に記載の合成樹脂組成物からなる消臭・抗菌性防草シート。

１４．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製

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剤であって、平均粒径１～１０μｍの消臭・抗菌性粉末製剤０．５～２０質量%配合してなる熱可塑性樹脂組成物製不織布。

１５．平均粒径３～２０μｍの酸化カルシウム水和物粉末を、繊維に対する乾燥質量１～５０ｇ／ｍ^2 で用いて繊維を裏処理してなる消臭・抗菌性付与繊維。

１６．請求の範囲第１０項又は第１５項に記載の消臭・抗菌性付与繊維により形成されてなるカーシート又は自動車天井材。

１７．請求の範囲第１項～第３項のいずれかに記載の消臭・抗菌性粉末製剤及び平均粒径１～４０μｍの酸化カルシウム水和物粉末からなる消臭・抗菌性混合粉末製剤を含有してなる合板。

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2004/004593

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl[7] A01N59/06, 25/08, 25/12, 65/00, A61L9/01, C09D5/14, D06M11/44

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)
Int.Cl[7] A01N59/06, 25/08, 25/12, 65/00, A61L9/01, C09D5/14, D06M11/44

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	JP 2003-26525 A (Yugen Kaisha Tone Seisakusho), 29 January, 2003 (29.01.03), Full text (Family: none)	1,4-17 2,3,6-17
Y	JP 2000-86417 A (ELP Corp.), 28 March, 2000 (28.03.00), Full text (Family: none)	2,3,6-17
Y	JP 2002-114617 A (Yugen Kaisha Yamaso Micron), 16 April, 2002 (16.04.02), Full text (Family: none)	2,3,6-17

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* 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
07 June, 2004 (07.06.04)	29 June, 2004 (29.06.04)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (January 2004)

国際調査報告

国際出願番号 PCT／JP2004／004593

A. 発明の属する分野の分類（国際特許分類（IPC））
Int. Cl⁷ A01N59／06, 25／08, 25／12, 65／00, A61L9／01,
C09D5／14, D06M11／44

B. 調査を行った分野

調査を行った最小限資料（国際特許分類（IPC））
Int. Cl⁷ A01N59／06, 25／08, 25／12, 65／00, A61L9／01,
C09D5／14, D06M11／44

最小限資料以外の資料で調査を行った分野に含まれるもの

国際調査で使用した電子データベース（データベースの名称、調査に使用した用語）

C. 関連すると認められる文献

引用文献のカテゴリー＊	引用文献名 及び一部の箇所が関連するときは、その関連する箇所の表示	関連する請求の範囲の番号
X Y	JP 2003－26525 A（有限会社東根製作所） 2003.01.29, 全文（ファミリーなし）	1, 4－17 2, 3, 6－17
Y	JP 2000－86417 A（株式会社エルプ） 2000.03.28, 全文（ファミリーなし）	2, 3, 6－17
Y	JP 2002－114617 A（有限会社 山曹ミクロン） 2002.04.16, 全文（ファミリーなし）	2, 3, 6－17

☐ C欄の続きにも文献が列挙されている。

☐ パテントファミリーに関する別紙を参照。

＊ 引用文献のカテゴリー
「A」特に関連のある文献ではなく、一般的技術水準を示すもの
「E」国際出願日前の出願または特許であるが、国際出願日以後に公表されたもの
「L」優先権主張に疑義を提起する文献又は他の文献の発行日若しくは他の特別な理由を確立するために引用する文献（理由を付す）
「O」口頭による開示、使用、展示等に言及する文献
「P」国際出願日前で、かつ優先権の主張の基礎となる出願の日の後に公表された文献
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「X」特に関連のある文献であって、当該文献のみで発明の新規性又は進歩性がないと考えられるもの
「Y」特に関連のある文献であって、当該文献と他の1以上の文献との、当業者にとって自明である組合せによって進歩性がないと考えられるもの
「&」同一パテントファミリー文献

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Translate
Fulltext

Deodorant antibacterial powder preparation, process for producing the same and product using the powder preparation

Abstract

A deodorant antibacterial powder preparation of 1 to 40 mum average particle diameter produced by hydrating calcium oxide obtained by firing seashells; a deodorant antibacterial powder preparation comprising 5 to 95 mass% of the above powder and 95 to 5 mass% of tea powder or powder of used tea leaves having an average particle diameter of 1 to 40 mum; or a deodorant antibacterial powder preparation comprising 5 to 95 mass% of calcium hydroxide powder of 1 to 40 mum average particle diameter and 95 to 5 mass% of tea powder or powder of used tea leaves having an average particle diameter of 1 to 40 mum. These deodorant antibacterial powder preparations can exert striking deodorant antibacterial effects against various odors and bacteria and can find advantageous application in a wide spectrum of fields.

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Claims

The scope of the claims

1. Shellfish enclosure shell obtained by calcining calcium oxide becomes hydrated, having an average particle diameter of 1-4 0 m · deodorant antimicrobial powder formulation.

1 Apparatus as claimed in claim 2. wherein the 5-9 5 mass % and a powder, a powder or a powder having an average particle diameter of 1-4 0 μ π ι mass % tea tea leaves 9 5-5 · deodorant and antibacterial powder composed made agent. 3.1-4 0 μ π ι 5-9 5 of calcium hydroxide powder having an average particle diameter of the mass^{2}X"and, an average particle size of the powder mass 9 5-5 1-4 0 μ π ι tea powder or tea leaves. $^{/0}$And a deodorant consisting of

The anti-microbial powder formulation•.

4. A baked at a temperature of 8 0 0-1 3 0 0 °C Clamshell to obtain calcium oxide, the oxide rushum force hydration · deodorant antimicrobial powder formulation in a method of fabricating a, 0.0 5-0.1 5 of the calcium oxide mass^{2}/$_{0}$ρ H is 9-1 3 of the aqueous solution so as to become hydrated · deodorant antimicrobial characterized in that the manufacturing method of the powder formulation.

Wherein the first range of 5. to 4 in a method of manufacturing, obtained by calcining Clamshell baked product so as to have a particle size of less than or equal to 1 0 0 μ m and ground, the pulverized product with a flow rate of water in a ratio of 0.2-1 1 k g liters in addition to hydrate the calcium oxide characterized in that the manufacturing method of the powder formulation · deodorant antimicrobial.

Wherein the first range of 6. - 3 1 wherein said first recited in any one of the anti-microbial powder formulation for removal of odor · 0.1-3 0 mass % is the ratio of the synthetic resin composition.

Wherein the first range of 7. - 3 1 wherein said first recited in any one of the anti-microbial powder formulation for removal of odor · have been surface treated by a surface treatment agent including a deodorant antimicrobial · becomes even HELASTIC, deodorant antimicrobial · when the amount of the powder formulation, in a dry weight basis of the surface treating agent 1-3 0% by mass in the deodorant. The anti-microbial HELASTIC.

Wherein the first range of 1-8. wherein the first recited in any of claims 3 wherein the anti-microbial powder formulation for removal of odor · 0.1-3 5 mass % based on the dry weight ratio of the paint.

Wherein the first range of 1-3 9. wherein said first recited in any one of the anti-microbial powder formulation for removal of odor · 1-8 0 mass % based on the dry weight ratio of the fiber treatment agent.

Wherein the range of 1 0. - 3 1 wherein said first recited in any one of the first of the anti-microbial powder for removal of odor · anesthesics made by the second treating agent being treated to · deodorant antimicrobial fibers, deodorant antimicrobial · when the blending amount of the powder formulation, 1-7 0 mass % in dry weight basis of the processing agent in the antimicrobial fiber · deodorant or PFREQ_VALID.

Wherein the range of 6 to 1 1. wherein the first synthetic resin composition that contains a foldback · deodorant antimicrobial lim

Wherein the first range of 6 to 1 2. wherein the synthetic resin composition is foamed · deodorant antimicrobial foam with the active hydrogen group.

6 Apparatus as claimed in claim 1 3. wherein the synthetic resin composition made from antibacterial anti-tsaoko · deodorant sheet.

Wherein the first range of 1 4 - 3 1 wherein said first recited in any one of the anti-microbial powder for removal of odor · made agent, a deodorant having an average particle diameter of 1-1 0 m'0.5-2 0 mass % of the anti-microbial powder formulation is the ratio of the tungsten nonwoven fabric made of a thermoplastic composition.

1 5. Hydrate calcium oxide having an average particle diameter of 3-2 0 m to a powder, a linkage fibers 1-5 0 g/m dry mass^{2}fibers in back-treating the resultant deodorant ' imparting antimicrobial fiber.

Wherein the range of 1 6. to 1 0 wherein the first or second 1 5 · deodorant antimicrobial property-imparting fibers which are formed with a car seat or more automobile headliners.

1 7. Apparatus as claimed in claim 3 wherein said first 1 - described in any one of the anti-microbial powder for removal of odor · fluicon made of calcium oxide hydrate powder having an average particle diameter from 1-4 0 μ m · deodorant antimicrobial mixed powder formulation containing a plywood.

Description

[0001] · Deodorant antimicrobial powder formulation is written in the light of the pores, and a method of manufacturing the product in the art using the powder formulation

[0002] The present invention, various kinds of odor, various kinds of bacteria, deodorizing performance, odor present in the antimicrobial efficacy of the antibacterial powder formulation ·, and a method of manufacturing the article to be manufactured by using a powder formulation. Background

[0003] In recent years, new building materials employing the synthetic resin, furniture, wall paper, automotive leather, textile products, food packaging, food bags and household goods is widely used. However a flask, such as furniture in

such a new building materials, furniture per se new building materials, adhesive and paint that is generated from the drain of the volatile materials such as formaldehyde, in recent years, as the cause of the sick house syndrome has become a major problem, such as the formaldehyde-arsenide Saeng the reduction of the volatile material is demanded In addition, from the viewpoint of comfort, which is typically a pathogenic E. coli O- 1 5 7 shows various antimicrobial to the bacteria, one of the antimicrobial agents or safe for use is demanded On the other hand, the amount of the food packaging and fodder bag in the field, the thermoplastic film is widely used, from the point of view of storage stability of the contents, safe, antibacterial and antifungal properties and the antimicrobial agent is strongly demanded. In addition, such as odor and cigarette odor in toilet deodorizing effect of improving the deodorant is required for home use.

[0004] · Safe odor as an antibacterial agent, include the use of a shell enclosure shell have been proposed, a switching, calcium carbonate powder obtained by grinding the shell containing the agent (for example see Japanese Laid-Open 2 0 0 1- 1 4 5 6 9 3), the amount of shell scallops shell powder containing the antimicrobial agent (for example, see Japanese Laid-Open 2 0 0 1- 1 9 9 5 2 3), shell scallops and firing the pulverized material of a shell and a bacteriostatic agent (for example, see Japanese Patent Laid-open 2002 25 5 7 1 4) is proposed. While only, the deodorant performance and antimicrobial capability, is not sufficient in both. Disclosure

[0005] The present invention, is made in view of the above circumstances, the various odor, various bacteria on the other hand, the performance of the deodorant, antibacterial effect, which can be used in the field of a wide range of a deodorant, anti-bacteria for the purpose of provide a powder of the present invention.

[0006] The present inventors, in order to solve the above problems as a result of earnest studies, the shell is obtained by firing the hydrated calcium oxide or a hydrate, an average particle size of a particular calcium oxide hydrate powder, deodorant and antimicrobial · a in the found effective. The present invention is completed based on the findings of the present invention.

[0007] That is, the gist of the invention are as follows.

[0008] (1) Obtained by firing the clamshell calcium oxide becomes hydrated, an average particle size of deodorant · antimicrobial powder formulation 1-40 μ m.

[0009] (2) Mass % and a powder of the (1) 5-9 5, average particle diameter of 1-40 μ m mass % of tea powder and a powder or tea leaves 95-5 · deodorant antimicrobial powder formulation.

[0010] (3) Calcium hydroxide powder having an average particle diameter of 1-40 m and 5-9 5 mass %, average particle diameter of 1-40 μ m mass % of tea powder and a powder or tea leaves 9 5-5 · deodorant antimicrobial powder formulation.

[0011] (4) 800 1 3 00 °C surrounded shell is fired at the obtained calcium oxide, calcium oxide hydration deodorant antimicrobial · method for manufacturing a powder formulation, 0 0 5-0 1 5 parts by mass of calcium oxide^{0} /$_{0}$ p H of the aqueous solution to be hydrated and becomes 9-1 3 is characterized in that the method of manufacturing the deodorizing · antimicrobial powder formulation.

[0012] (5) In the above manufacturing method (4), a shell is fired at 1 0 0 μ m the baked product thus obtained was ground to a particle size of less than or equal to, the rate of water relative to the ground at a rate of l k g 0.2-1 torr to hydrate the calcium oxide in addition to the group consisting of a deodorant and antimicrobial · method of manufacturing the powder formulation.

[0013] (6) - (3) Odor of any of the (1) antibacterial powder formulation mass % · 0.1-30 is the ratio of the synthetic resin composition.

[0014] (7) - (3) Odor of any of the (1) antibacterial powder formulation · a table containing the surface-treated with a surface treatment agent made of a leather odor antimicrobial ·, · odor when the amount of the antibacterial powder formulation, a dry weight basis of the surface treating agent is 1-3 0 mass % in the deodorant antimicrobial · razor.

[0015] (8) - (3) Odor of any of the (1) antibacterial powder formulation · non mass % on the basis of the quality of the dried 0.1-3 5 is the ratio of the coating.

[0016] (9) - (3) Odor of any of the (1) antibacterial powder formulation · 1-8 0 mass % on the basis of dry quality is the ratio of the fiber treatment agent.

[0017] - (3) (1 0) Of any of the above (1) deodorant antimicrobial powder formulation · being treated by a treatment agent containing a deodorant antimicrobial fiber ·, · deodorant antimicrobial powder when the amount of the formulation, 1-70 mass % based on the dry weight of the treatment agent in the deodorant antimicrobial fibrous ·.

[0018] (1 1) Made of synthetic resin composition of the above (6) deodorant antimicrobial film.. (1 2) The aforementioned synthetic (6) resin composition that is on the calling · deodorant antimicrobial foam.

[0019] (1 3) Made of synthetic resin composition of the above (6) deodorant antimicrobial anti-grass sheet ·$_{c}$ (1 4) of any of the above (1) deodorant antimicrobial · - (3) powder formulation and in this dog, average particle size of deodorant 1-1 0 μ π ι · 0.5-20 mass % of the anti-microbial powder formulation is the ratio of the nonwoven fabric made of thermoplastic resin composition.

[0020] 3-20 μ m (1 5) Having an average particle diameter of calcium oxide hydrate powder, the dry fiber with respect to a 1-5 0 g/m^2 fibers in the back-treating the resultant deodorant · anti-bacteria-providing fiber. (1 0) Or (1 8) (1 5) above is provided with such a deodorant antimicrobial · fibers or automotive car seats which are formed with the ceiling material.

[0021] (1) - (3) Serial loading any of the above (1) deodorant antimicrobial powder formulation of · 1-4 0 μ m hydrate of calcium oxide and average particle size of a powder deodorant mixture powder formulation of an anti-fungal · containing the plywood. Detailed Description of the Invention

[0022] Deodorant antimicrobial powder formulation of the present invention as used in the shell, the firefly oysters, clams, is the bore-in, oyster shell, turban, mussels and abalone shell and the like are found. Among these, the proportion of the industrial processing of the oyster shell and the shell is high if the shell of the firefly, and easily available from the preferred and, further, the content of the impurities maltopectin be low, and a large amount of waste from the viewpoint of effective use, it is preferable that the shell of the shell of the firefly. And to calcium oxide obtained by burning the shell is, zinc, iron, magnesium ©-time since minor amounts of metals, is preferred from the viewpoints of imparting antimicrobial properties.

[0023] Surrounded and the firing of the shell, a shell was washed with water and then, in 8 0 0-1 3 0 0 °C, at time 3-6. After firing, and the like are removed from the sintering fine sand, and water was added to hydrate the cull shum oxide. After hydration, a precipitate of the solution was cooled to about 5-4 0 °C, a precipitate of a centrifuge or filtration subcatalyst separated, the resulting separated were dried, followed by classifying, having a predetermined average particle size calcium oxide hydrate is obtained.

[0024] The sintering may, deodorant antimicrobial · calcium oxide obtained from the viewpoint of improving the performance, and it is important to perform at a high temperature, the baking temperature is less than 8 0 0 °C, for the firing is not sufficiently performed, thereby deteriorating the deodorant is · antimicrobial properties. May be fired at a temperature greater than 1 3 0 0 °C, a deodorant antimicrobial efficiency is more improved and also · but also, energy is only consumed in vain. The firing condition, preferably has a 3-5 at time 900-1 200 °0 aru. The baked product, such as sand having a particle diameter of 1 00 μ m or less has been removed is taken is preferably ground. Is pulverized, the poppet ball mill and jet mill or the like can be used. In addition, the amount of water is added for hydration, to the baked product 1 k g where the position of the percentage of 0.2-1 liters.

[0025] Obtained by calcining and hydrated calcium oxide hydrate, 0.0 5-0.1 5 mass % aqueous solution thereof is 9-1 3 p H, preferably the 1 0-1 3 version, the antimicrobial performance of high deodorizing · is important. Firefly for example in the case of using the shell of the shell, the temperature of the baked product thus obtained was baked at 800-1 300 °C for grain size is ground to 1 00 Atm or less, the resulting powder savage added at a ratio of 1 k g to 0.2-1 with water and hydrated liters, after hydration by a cooling temperature of 5-40 °C, p H hydrate with the calcium oxide can be obtained.

[0026] In the present invention, the average particle diameter of calcium oxide hydrate powder required to be 1-40 m. May vary depending on the applications, usually, preferably 2-30 m, more preferably 3-20 zm, more preferably at 5-1 5 μ m. This average particle diameter is less than 1 m, calcium oxide hydrate powder is liable to be aggregated, it becomes difficult to handle, less than 1 zm to that of the finely powdered also only with difficulty. This average particle diameter is more than 40 μ m, the surface area of the calcu oxide hydrate powder is small and a prism, a deodorant · antimicrobial properties and are not expressed, in addition, the particle diameter is much greater than 40 μ π ι is also included so that, upon engagement with the distribution becomes a foreign matter.

[0027] Average particle diameter of the calcium oxide hydrate powder, the powder in a ball mill into powder by a frame time or di ettomill by adjusting the pressure and time, in the adjustment of the parent.

[0028] In the present invention, calcium oxide hydrate powder, the powder may be mixed as a powder or tea in tea leaves, the antimicrobial substances to be subjected to deodorizing · preferable from the viewpoint of a wider range. Specifically, a so-called sick house and become a cause of not only formaldehyde Saeng arsenide, ammonia, such as acetic acid smell of life of the odor, tobacco odor deodorant target.

[0029] Tea include, for example, green tea, oolong tea, black tea, such as barley tea and tea and tea pu-erh, from the viewpoint of excellent antimicrobial properties and deodorant ·, green tea, oolong tea and loop is preferable. Tea may be used alone, or two or more types may be used with the kneaded. In addition, a mixture of powder and the tea leaves and tea powder may be used for tea leaves powder is a powder or tea, tea or tea leaves after drying, ya tumbling gesture can be produced by pulverizing a bowl can be obtained. These powder, average particle diameter is preferably 1-4 0 μ π ι, and loop-dispersible antimicrobial performance · from a point on a deodorant, preferably 5-3 5 μ π ι. Tea powder or tea leaves the adjustment of the average particle diameter, the average particle diameter in the above-mentioned calcium oxide hydrate powder in the same manner as the adjustment can be performed.

[0030] The calcium oxide hydrate powder content of the tea leaves and tea powder or a powder, 5-9 5 mass % of calcium acid monohydrate powder, the

powder or tea powder is preferably 9 5-5% amount of the quality of the tea leaves. The ratio of use, the odor is the target of the deodorant, antibacterial target by adjusting the associated bacteria. For example, drain formaldehyde, acetic acid, an acidic odor with water such as sulfide, calcium oxide hydrate powder or to increase the usage ratio of the effective and, in the case of the reverse the odor of the other. In addition, in the case of the application wants to avoid coloration, tea powder or tea leaves to reduce the rate of use of the powder preferably, less than 5 mass %, with respect to the ammonia odor and tobacco odor deodorization effects are not sufficient. In addition, the rate of use of tea powder or tea leaves powder exceeds 9 5 mass %, with respect to the formaldehyde odor Saeng ya acetic acid effect is reduced. Performance is an anti-bacteria, tea powder is a powder or tea leaves, Bacillus subtilis, E. coli and S. pudou yellow the antimicrobial performance of a wide range of bacteria is, the large intestinal bacteria is Staphylococcus aureus and loop, and the ratio of calcium oxide hydrate powder is effective.

[0031] The tea powder or tea leaves the powder, the calcium oxide hydrate is mixed with a powder instead, karushiumu hydroxide produced industrially at the same be mixed with the powder. Average particle size of the calcium hydroxide powder, the calcium oxide hydrate powder in average particle diameter of 1-4 0 μ m in the same manner as is required. May vary depending on the application, normally, preferably 2-3 0 μ m, more preferably 3-2 0 μ m, more preferably at 5-1 5 μ m. Karushiumu hydroxide powder and tea powder content of the tea leaves or powder, the calcium oxide hydrate powder and tea powder for a percentage of the powder is also similar to tea leaves, ya target odor by bacteria, can be adjusted in the same manner as described above.

[0032] · Deodorant antimicrobial powder formulation of the present invention, that is, calcium oxide hydrate powder, a powder or tea powder karushiumu oxide hydrate powder mixture of tea leaves, calcium acid and water as a powder or powder mixture of tea and tea leaves is, by mixing a synthetic resin, synthetic resin in the various products can be used for antimicrobial · odor. In this case, deodorant antimicrobial · the compounding ratio of the powder formulation, more preferably 0.1-3 0 mass %, more preferably 0.2-2 0 mass %, more preferably 0.5-1 0 mass %. When this blending proportion is less than 0.1 mass %, the sterilization effect is not expected to be a deodorant•, 3 0 mass % is exceeded, which may adversely affect the skin.

[0033] As a synthetic resin, may be inferior in alkali resistance. Is placed in a bag such as bag or feed for food wrapping the case of the use, polyethylene, polypropylene, polyvinylidene chloride (polyvinyl chloride) and P V C and the like. Wall paper materials for the soft polyvinyl chloride and - is preferably used. Food packaging bag, the bag in the case of the wallpaper and loop , using a film-shaped, and its thickness is different depending on the application, 1 5-1 0 0; u m is suitable. Is the injection molded article, a polyolefin resin such as polyethylene and polypropylene as well, or a polystyrene resin (an acrylonitrile one butadiene-styre) A B S of a resin such as styrene-based resin, polyacrylonitrile resin or poly hue Saeng rensulfi the second thermoplastic resin of the resin or the like is preferably used in the middle row. In the case of a molding method other than injection molding, a foldback nord resin, melamine resin, urea resin and epoxy resin such as e thermoplastic resin is used. Further, the propylene copolymer or styrene chillen e one of a thermoplastic elastomer copolymer such as butadiene or one can be used.

[0034] Deodorant of the present invention the antibacterial powder formulation, polyester fibers, rayon fibers, polypropylene fibers and atariru fiber product, deodorant antimicrobial · when used as a fiber treating agent is applied, the back treatment agent can be formulated, from the viewpoints of the ease of use, in particular, a vehicle seat, the seat for furniture, the decline of the deodorant antimicrobial•of suitable for use as a fiber treating agent applied. As the base of the processing agent, an acrylic resin or a synthetic rubber latex and the golfer can be used. Treatment agent, a deodorant agent · this group was used as the antimicrobial powder formulation of the present invention.

[0035] · In the deodorant antimicrobial agent is blended in an amount of the powder formulation, a dry mass of the treating agent is preferably 1-8 0 mass % of the reference voltage in, more preferably 5-6 5 mass %. This amount is less than 1 mass %, poor · deodorant antimicrobial effect, 8 0 mass % · deodorant antimicrobial powder formulation exceeds in formulating a treatment agent is hardened, the texture of the fibrous article becomes lost. Incidentally, a deodorant antimicrobial · average particle size of the powder formulation were required to 1-4 0 m, preferably 2-3 0 m, more preferably has 3-2 0 μ m, more preferably at 5-1 5 μ m.

[0036] And a processing agent is used in an amount of the back, depending on the required properties, 1-5 0 g Zm dry mass^2 preferably, 5-3 0 g/m^2 is still more preferable. 1 g/m If the amount used^2 is less than, the deodorizing performance is not sufficient, 5 0 g/m^2 is exceeded, there is a possibility that the fiber is hard, costs increase. In addition, the treating agent may, polyethylene, polypropylene, rayon, Naimark an opening, such as bi - containing thermoplastic polyester and the conduction from the resin composition of the nonwoven fabric can be also widely used as a treatment.

[0037] · Deodorant antimicrobial powder formulation of the present invention the odor of the razor " is used as an antibacterial agent, deodorant antimicrobial powder formulation · urethane resin or the like and may be incorporated into the

surface treating agent, leather surface of this, an x-bar code may be applied by a roll coater and loop. As leather, a leather P V C, urethane leather, a leather orefin-based thermoplastic elastomers, and natural leather and the like. Other than the natural leather, the skin layer is formed on the substrate layer and, as a material which forms a base layer. P V C, urethanes and orefin horse-based thermoplastic elastomer and the like. Deodorant in order to improve the antimicrobial effect ·, · also to a substrate layer containing a deodorant antimicrobial powder formulation can be made. A deodorant in the surface treatment agent

[0038] *the mixing amount of the antibacterial powder formulation, the surface treatment agent is preferably 1-3 0 mass % based on the dry mass, more preferably 5-2 0 mass %. This amount is less than 1 mass %, poor · deodorant antimicrobial effect, 3 0 mass % is greater than the decrease in the properties of the skin layers.

[0039] · Deodorant of the present invention the antibacterial powder formulation, various chair, a sofa and the vehicle is used as a cushion member of the sheet according to the soft polyurethane foam was blended, deodorant antimicrobial · as a cushion material are preferably used. In addition, the soft diacrylate down form other, such as polyethylene or polypropylene materials for the thermoplastic resin such as a reflex fin poliovirus, such as a polystyrene resin and a rigid urethane foam or a foamed material is calling, household goods and used as a heat insulating material for building. Ya of these groups in the foam cushion deodorant antimicrobial · is used in an amount of the powder formulation, ya of the cushion material of the foam is preferably 1-3 0 mass % based on the dry mass, more preferably 5-2 0 mass %. This amount is less than 1 mass %, poor-deodorant antimicrobial effect, 3 0 mass % is greater than the reduction in cushioning ya insulating properties. Incidentally, the foam may be produced by a known method.

[0040] · Deodorant of the present invention the antibacterial powder formulation, can be compounded as the anti-grass sheet-c · deodorant antimicrobial sheet grass, such as a food packaging bag in the same manner as described above and the material of the odor -. Anti-microbial film formulated powder formulation (thickness but are not limited to, preferably has about 2 0-5 0/i m) was cut to form a yarn and that of braided, can be manufactured by a known method. · Deodorant antimicrobial anti-grass sheet, banks of the farmland, bare ground, suitable for applications such as an underlay potted plant. In addition, antimicrobial anti-grass sheet · deodorant, deodorant nonwoven fabric made of polypropylene and the like to be contained in the anti-microbial powder formulation · may be used.

[0041] · Deodorant antimicrobial powder formulation of the present invention as other applications, for building materials, automobile-related applications, and household applications, in particular, for use as building material, in place of the clad for the convolution, the convolution core material for the, water-proof film, the glass protective film, plywood, decorative plywood, a tickle board pattern, flipper board, chip board, assemblies, panel, a Japanese paper or the like is blended into the bran and, or the finish and construction for the ring to be used by blending. Car-related applications include, a ceiling material, the air conditioner and interior paint and the filter for the horse. Household applications, kano maltopectin one cushion, the cushion material, a curtain, and the like and each tape nose cross SEEBS species.

[0042] In addition, a deodorant of the present invention the antibacterial powder formulation ·, the outer ring is arranged in a ring to the wax, the wax preferably used as a deodorant antimicrobial · be$_e$ further, a deodorant of the present invention the antibacterial powder formulation ·, · deodorant antimicrobial coating, in particular antimicrobial

[0043] O A coating equipment, such as paint of the paint for home electric appliances and furniture can be preferably used in the formulation.

[0044] May be a paint, solvent-based tungsten and an aqueous resin, a deodorant of the present invention the antibacterial powder formulation ·, 0.1-3 5 mass % based on the dry mass can be obtained by compounding$_e$ in this case, deodorant antimicrobial powder formulation ·, average particle diameter in the range of 1-4 0 μ π ι can be used, and the average particle size is preferably 1-2 0 μ π ι, more preferably 3-1 5 m. This average particle diameter is less than 1 m agglomerated powder and base resin, the content is more than 4 0/i m, unevenness in the coating film tends to occur. Furthermore, 5 0 m or less is the most large particle size, it is desirable to obtain smooth surfaces.

[0045] Solvent-based resin may be, the golfer tungsten methacrylate, urethane resin, an alkyl amino-based resins and loop Saeng tungsten and the like, and these may be used alone, a mixture of two or more of may be used. Water-based resin, an ethylene-vinyl acetate erjo, acrylic-based essentialgeon, essentialgeon urethane, water-soluble acrylic resin, a water-soluble urethane resin, water-soluble epoxy resin and a water-soluble alkyd resins and the like, and these may be used alone, a mixture of two or more types may be used..

[0046] The coating of the paint of the object may include, metal, glass, resin, paper, wood and the like and a ceramic and loop rows, can be arbitrarily selected.

[0047] As the coating method, brush painting, spray coating, electrostatic coating and powder coating and the like, can be appropriately selected. These coating compositions, without impairing the physical properties of the coating at the normal Iodine, a deodorant and antimicrobial properties are imparted by the present invention.

[0048] · Deodorant antimicrobial nonwoven fabric, as described above, in addition to the fiber treatment method according to the material, the material of the nonwoven fabric at the stage of spinning fibers, odor by a conventional method by mixing the antimicrobial powder formulation · can be obtained. Deodorant antimicrobial · is used in an amount of the powder formulation, and 0.5-2 0 mass %, the antimicrobial effect · odor from the viewpoint of formability and appropriate. The method of manufacturing a nonwoven fabric, is not particularly limited, a dry method or wet method or the like can be manufactured by a known method. Concrete manufacturing method include, for example, using a needle-punched fibers together or each pass, such as sewing yarn is stopped at a mechanical joining methods, heat, pressure or an adhesion method using an adhesive binder and the like.

[0049] In this case, in order to push the fine fibers, deodorant antimicrobial powder formulation, the average particle diameter is preferably 1-1 0 μ m, more preferably 2-5 μm. This average particle diameter is less than 1 μ π ι powder tends to agglomerate, there is a problem in the processing. Average particle diameter exceeds 1 0 m, the yarn becomes apt to be cut upon spinning, and it is difficult to obtain a thin fiber

[0050] As the raw fibers of the nonwoven fabric, polyethylene, polypropylene and loop such as an opening is preferably used a nylatch, · deodorant antimicrobial powder formulation can be formulated as a raw material may be any, is not particularly limited. Example

[0051] Then, according to the comparative example and loop embodiment of the present invention to further specifically be described, by way of example these the invention and is not limited in any way.

[0052] Example 1

[0053] Firefly after the shell was washed with water, and calcined at 1 1 00 °C 4, the mesh size of 5 mm and sieved to remove fine sand, I k g the baked product was obtained. Liters 0.5 does this by the addition of water to the re-hydrated calcium oxide, and cooled to 2 5 °C, the precipitate was separated using a suction filter, isolate 1 2 0 °C was dried by application of time or the 5. The dried product was classified by an air separator, a hydrate of calcium oxide powder having an average particle diameter 9/m (deodorant, anti-microbial powder formulation) was obtained.

[0054] Torr within the interior volume 3 00 of mass 1 in a conical flask and evaluated _FUB_ADR_EXTEND millounces^{0} /$_{0}$ Saeng Imicrolitre 1 5 is placed in an aqueous solution of formaldehyde, the calcium oxide hydrate powder 0. 05 g (1 gZ - liter aqueous solution of ρ H is 1 2.5) was placed, after having been left for 1 2 0 at .24 ¾, the drain of the flask is detected by the detector by Kitagawa type formaldehyde concentration was measured. In addition, a method described below, ' antifungal test was performed. The effects of imaging shown in Table 1.

[0055] Example 2

[0056] Green tea leaves, 0.5 mass % and the amount of moisture is dried, coarsely pulverized by a shredder, a ball mill and a jet mill and further pulverized, the average particle diameter

[0057] 3 0 m Tea powder was obtained. 5 0 Mass % and the tea powder, obtained in Example 1 5 0 mass % calcium oxide hydrate powder obtained by mixing deodorant antimicrobial mixed powder formulation · (1 g / - liter aqueous solution of the 1 1 p H) 0.0 5 g is used, similar to the embodiment of the invention the drain 1 formaldehyde concentration was measured. in addition, one row of the test dust. Shown in Table 1.

[0058] Example 3

[0059] Calcium oxide hydrate powder instead of the 4 μ m proteasome calcu hydroxide having an average particle diameter (1 g Z 1 1 torr-containing aqueous solution of the ρ H _FUB_ADR_EXTEND) except for using, in the same manner as in Example 2 is mixed to obtain a powder formulation of a deodorant antimicrobial · , formaldehyde concentration was measured in the same manner as the drain. In addition, antifungal test was performed. Shown in Table 1.

[0060] Example 4

[0061] Obtained in Example 2 a mixed powder formulation to a deodorant antimicrobial · may be made of the polycarbonate-based © (manufactured by Neo Rez. rhodamine D 6 0 2 5 as a laser) may be incorporated into, a deodorant antimicrobial - surface treating agent was prepared. The powder mixture is blended in an amount of the formulation, a dry surface treatment agent and on the basis of 2 0 mass % fishy. 5 5 Mass % and 3 5 parts by mass of polyester fibers and rayon fibers % blend of the base fabric on a flat fabric, a soft vinyl chloride with nil layer is formed on the surface of the razor P V C, an x-bar code by means of coating with the surface treatment were, and drying the surface treatment layer having a thickness of about 1 0 μ m · having a deodorant antimicrobial treatment of leather. 5 c m X 5 c m Cut out from a sample of the leather, formaldehyde in the same manner as in Example 1 to determine the concentration of the drain. Shown in Table 1.

[0062] Example 5

[0063] Obtained in Example 3 a mixed powder formulation to a deodorant antimicrobial · ____ karler ____ ____ 0 (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. The powder mixture is blended in an amount of the formulation, based on the dry weight of the

fiber treating agent was 1 5 mass %. Polyester backing, the fiber treating agent is laminated by a roll coater, and the dry fine particles having a thickness of about 1 00 μμι (backing) of the fiber treating agent layer having a sheet was obtained. From the sheet specimen 5 c mX 5 c m, 1 in the same way as the formaldehyde concentration was measured in the drain. Shown in Table 1.

[0064] Example 6

[0065] Example 4 was produced in the antimicrobial treatment of leather cut out from deodorizing · the sample of 5 cmX 5 c m, 5 to 4 mass % aqueous ammonia solution is the product of the contents of the micro rate torr

[0066] 300 Milli-liters of the placed in a conical flask, after allowing to stand for 25 °C at 1 20, Kitagawa type is detected by the detector was measured in the concentration of ammonia in the flask,

[0067] 145 P p m was the mass.

[0068] Example 7

[0069] Karushiumu oxide obtained in Example 1 1 parts by mass of hydrate powder, linear low density polyethylene (Idemitsu (strain), I DEM I TSU LL 0 1 3 4. methacrylate manufactured by a melt flow rate: 1.2 gZI O min) and 99 parts by mass of a command to the drive recorder, after that, the kneaded by a twin-screw extruder (kneading=40 mm φ) were kneaded in a 200 °C, tungsten composition was obtained. This resin composition, the extruder (extrusion=50 mm φ) formed by the opening and inflation $, width 60 mm, thickness 40 μ m of the inflation film was produced. 5 cmX 5 c m Cutting out a sample of this film, formaldehyde in the same manner as Example 1 to determine the concentration of the drain.

[0070] Example 8

[0071] Example 1 calcium oxide hydrate powder obtained in the solvent system is soluble in the liquid urethane tungsten, 5 mass % is blended in a dry weight basis, to prepare a paint Then, the coating material spray gun (Iwata (strain) coating machine industry Ltd.) by, a thickness of 2 mm (acrylate-butadiene-styrene first ioni one tolyl) AB S plates were spray coating. Cutting out a sample of this AB S plate 5 c mX 5 c m, the drain W 200 in the same manner as example 1 in the formaldehyde concentration was measured. Shown in Table 1.

[0072] Example 9

[0073] Example 1 calcium oxide hydrate powder obtained, the polyol component, i seo synate component, a foaming agent, a catalyst and a foam stabilizer by blending in the proportions shown in the following, a conventional method, including 5 mass % calcium oxide hydrate powder deodorant antimicrobial ule · Tan foam was produced. 5 c m X 5 c m X 5 c m of the urethane foam sample is cut off, in the same manner as in Example 1 with the constant measuring formaldehyde concentration of the drain. Shown in Table 1. &Gt constituents of the formulation ingredients;

[0074] Polyol (Dow poly urethane Co., #3 0 0 0) 1 00 parts by mass (poly urethane manufactured by Dow Chemical, T- 8 0) isoshianeto 40 parts by mass of Wednesday (ZK) 2.9 parts by weight of a blowing agent and an amine catalyst 3 parts by mass (Nourybond Co., 3 3 LV) an amine-based catalyst (manufactured by Nourybond, AT 3 3) 3 parts by mass of a tin-based catalyst (manufactured by Nitto Kasei, one T 9) 3 parts by mass of a foam control agent (manufactured by Japan one yuka, L 6 20 2) 3 parts by mass of 8 parts by mass of calcium oxide hydrate powder Example 1 0

[0075] Obtained in Example 1 calcium oxide hydrate powder was classified to obtain a powder having an average particle diameter of 3 μ m, a polypropylene resin which one (M I=5 0 g// 1 0) is blended in, 40 calcium oxide hydrate powder mass^{0} /$_{0}$ comprising a master batch was obtained. The master patch 5 parts by mass of a polypropylene resin (M I=5 0 gZ 1 0 min) is the ratio of 9 5 parts by mass, the hydration of calcium oxide powder composition comprising 2 mass % was prepared. The composition, is projected into an extruder, melted and kneaded at a resin temperature of 2 2 0 °C and then, the kneaded material extruded from the die (2 0 0 holes are alcohols), and stretched in the air at a high speed, to be recycled, a long fiber filament was made to continuously modulate. Incidentally, M I is (melt inde' slawx), based on J I S K 6 9 2 1, 2 3 0 °C, measured load 2. 1 6 k g f (2 1. 1 8 N).

[0076] Random filament is integrated into a sheet, the sheet was passed through between the emboss roll and a smooth roll, and a thermal embossing at 1 4 0 °C, the non-woven fabric was produced.

[0077] The resulting non-woven fabric, the average fiber diameter 2 2.4 ^ m, 5 0 g having a basis weight in the average amino m^{2} was. Cutting out a sample of the nonwoven fabric 5 c m X 5 c m from the same manner as in Example 1. Saeng formaldehyde concentration was measured. Results are shown in table 1 to be preserved.

[0078] Comparative Example 1

[0079] In Example 1, without the use of calcium oxide hydrate powder rudehi golfer formnitrogen Saeng concentration was measured . In addition, antifungal test was performed. Results are shown in table 1 c shown in Comparative Example 2

[0080] In Example 1, calcium oxide hydrate powder magnesium hydroxyl 1 μ m i having an average particle diameter (1 gZ - liter aqueous solution of the p H 7) except for the changes, in the same manner as in Example 1 of the drain

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formaldehyde concentration was measured. In addition, antifungal test was performed. Shown in Table 1.

[0081] Comparative Example 3

[0082] In Example 1, 600 °C and the baking temperature, such as a hydration step after firing through the same manner as in Example 1 except that the solder and the obtained powder of average particle diameter 3 0 μ m ; (1 g/ - liter aqueous solution of the p H 8) will be, in the same manner as in Example 1 the golfer formnitrogen rudehi Saeng concentration was measured. In addition, antifungal test was performed. Shown in Table 1。Comparative Example 4

[0083] In Example 5, a deodorant antimicrobial · but without the use of the mixed powder formulation, similar to the embodiment of the invention the drain 5 formaldehyde concentration was measured. Shown in Table 1. Comparative Example 5

[0084] In Example 6, instead of the deodorant antimicrobial treatment of leather -, the same as in Example 4 was used as a deodorant ' does not incorporate an antimicrobial mixed powder formulation except that the used razor P V C, the ammonia concentration in the same manner as Example 5 was measured, the mass 2 3 0 was p p m.

[0085] [Antifungal test]

[0086] Test strain 1.: filamentous fungi of the genus M u c o r

[0087] Mediums 2.:

[0088] -1 1 Medium: glucose in a mineral salts medium

[0089] Gunorekosu 3 0 0 g

[0090] Ammoniumu nitrate 3 0 g

[0091] Helium 0 g the force of the phosphoric acid

[0092] Maguneshiumu sulfate 0 5 g

[0093] Helium force chloride 0 2 5 g

[0094] Ferrous sulfate ' 0 0 0 2 g

[0095] Milliliter of deionized water 0 0 0

[0096] -2 2 Medium: glucose inorganic salt agar medium

[0097] Gunorekosu 3 0.0 g

[0098] Ammoniumu nitrate 3 0 g

[0099] 1.0 g Rum the force of the phosphoric acid

[0100] Maguneshiumu sulfate 0.5 g

[0101] Helium force chloride 0.2 5 g

[0102] 0.0 0 2 g Ferrous sulfate

[0103] Milliliters of deionized water 1 0 0 0

[0104] For the preparation of the agar 2 5.0 g 3. spores:

[0105] P D A filamentous fungus genus M u c o r to a plate (potatoid agar kiss troost de) on (2 8 °C for one week culture) spore formation and with respect to those, lyricist micellar/spores using sterile water 1 0 mycelial suspension was prepared.

[0106] Then, after filtering through sterile cotton wool, spores was precipitated by centrifugation, the above media (agar vent, a mineral salts medium glucose) 1 2 0 spores by the re-suspended miriitsutoru prepare a solution.

[0107] The installation of the test piece 4. was inoculated and loop

[0108] The above media (glucose inorganic salt agar medium) on the 2, and Comparative Example 1-3 Example 1-3 was used in the deodorant antimicrobial powder formulation liters · 5 g Z aqueous solution (0.5 mass^{9} /$_{0}$) 1milliliter was applied to the plate.

[0109] Spores on the test piece in the liquid enough to form a fine water droplets in the spray inoculated iodine, after the plates were sealed with paraffin, and cultured in a constant temperature bath chamber. Sprayer 2 and sprayed by means of the communication is performed, the culture conditions, culture temperature 3 0 °C, humidity 9 5%, and 4 weeks culture period.

[0110] 5. The growth of fungi was observed:

[0111] 2 Weeks after the inoculation and then, the degree of growth of the mycelia on the surface of the test plate was observed visually.

[0112] 6. Determination of a result of:

[0113] According to the criterion shown below, the time course of the growing condition of the mycelia were determined.

[0114] [Growth criteria]

[0115] One: in the naked eye, the mycelial growth was observed (force PVC)

[0116] + Z -. slightly observed in the growth of the mycelia

[0117] +. Growth of the mycelia were found in the

[0118] : Hyphae and spores were found in the significant growth in Table 1

[0119]

[0120] ND, not detected. Example 1 1

[0121] 1 0 μ m of calcium oxide hydrate powder having an average particle diameter (deodorant antimicrobial powder formulation ·) Foremargion to actin

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(Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. · Deodorant antimicrobial amount of the powder formulation, the dry fiber treating agent 5 0 mass % on the basis the fishy. Polyester backing, laminated fiber treatment agent with the above-mentioned one with a roll coater, and dried at 1 0 0 °C for 3 0, 2 4 g by dry mass of the fiber treatment agent[2] · deodorant antimicrobial applied to the coated fabric to produce a poly ester group. 5 c m X 5 c m Switched for a sample of this backing out from, in the same manner as in Example 1 was measured by the formaldehyde concentration of the drain, p p m less than 0.5 parts by weight.

[0122] Example 1 2 calcium oxide hydrate powder having an average particle diameter 5 μ π ι (deodorant antimicrobial powder formulation ·) acrylic gelon a (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber is made of a treatment agent. Deodorant ' mixing amount of the antibacterial powder formulation, a dry weight basis of the mass 50 in the fiber treating agent. /. And. Polyester backing, the fiber treating agent is laminated by a roll coater, and dried at 1 0 0 °C for 3 0, fiber is a processing agent 2 6 g/m by dry mass[2] deodorant antimicrobial coating applied to the base cloth. stell police were prepared. 5 c m X 5 c m Cutting out a sample of the fabric, the concentration of formaldehyde in the same manner as in Example 1 was measured and the drain, p p m less than 0 5 parts by weight.

[0123] Example 1 3

[0124] Calcium oxide hydrate powder having an average particle diameter 1 0 (deodorant antimicrobial powder formulation ·) Foremargion to actin (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. · Deodorant antimicrobial amount of the powder formulation, on the basis of dry fiber treating agent and 50 mass % fishy. Polyester backing, laminated fiber treatment agent with the above-mentioned one with a roll coater, and dried at 1 0 0 °C for 3 0, 5 g/m by dry mass of the fiber treatment agent[2] applied to the coated deodorant antimicrobial police stell · base fabric was prepared. 5 c m X 5 c m Cutting out a sample of the fabric, the concentration of formaldehyde in the same manner as in Example 1 was measured and the drain, p p m less than 0.5 parts by weight.

[0125] Example 14

[0126] 1 0 m Average particle diameter of calcium oxide hydrate powder to acrylic gelon (deodorant antimicrobial powder formulation ·) (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, a concentration of 2 mass % calcium oxide hydrate powder to prepare a dispersion. This dispersion, 30 c mX 3 0 c mX 1 2 mm were applied using a spray on the plywood, 2 g Z 9 0 0 c m calcium oxide hydrate powder[2] applied in, the golfer formnitrogen Saeng rudehi plywood generation reduction was manufactured. J I S A 1 9 0 1 Saeng formaldehyde emission of the plywood in the method as defined in the was measured, was 0.2 m g/Torr-containing lysine. On the other hand, the processing of the same material of the amount released formaldehyde plywood Saeng de arsenate was 0.5 m g Z Torr-containing lysine.

[0127] Comparative Example 6

[0128] 9 0 Mass % of water, 8 μ m of calcium oxide hydrate powder having an average particle diameter (deodorant antimicrobial powder formulation.) were mixed at ratios of 1 0 mass % in a liquid, wet-type bead mill (scissor fine tech Ltd. (strain), L M Z - 2) wet-pulverized by a, a hydrate of calcium oxide powder having an average particle diameter 0 6 μ π ι (deodorant antimicrobial powder formulation ·) was obtained. The deodorant 'acrylic gelon antimicrobial powder formulation (Ganz Chemical Co. Ltd. (strain), the cross-xylene) is blended into the, ' The fiber treating agent was prepared. · Deodorant antimicrobial amount of the powder formulation, based on the dry weight of the fiber treating agent and 5 0 mass %. Poly ester phenolic backing, the fiber treatment agent is laminated by a roll coater, and dried at 1 0 0 °C for 3 0, 2 7 g Zm by dry mass and the fiber treatment agent[2] deodorant applied to ' the polyester fabric provided with antimicrobial properties. The base cloth, deodorant antimicrobial powder formulation · aggregation, was not practically usable.

[0129] Comparative Example 7

[0130] 8 μ π ι of calcium oxide hydrate powder having an average particle diameter (deodorant antimicrobial powder formulation ·) obtained were classified to 4 5 μ π ι average particle diameter of calcium oxide hydrate powder (deodorant antimicrobial powder formulation) to, acrylic gelon (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. · Deodorant antimicrobial powder formulation is used in an amount of, based on the dry weight of the fiber treating agent was 5 0 mass %. Polyester backing, said fiber by a roll coater was stacked on the treating agent, solid-liquid separation of the fiber treatment agent, which could not be laminated.

[0131] Comparative Example 8 1 0 μ π ι of calcium oxide hydrate powder having an average particle diameter (deodorant antimicrobial powder formulation ·) acrylic gelon a (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. · Deodorant antimicrobial amount of the powder formulation, the dry fiber treating agent 5 0 mass % on the basis the fishy. Polyester backing, laminated fiber treatment agent with the above-mentioned one with a roll coater, and dried at 1 0 0 °C for 3 0, 0.5 g Z m by dry mass of the fiber treatment agent[2] applied to the coated deodorant antimicrobial, to prepare a crosslinked base fabric. 5 c m X 5 c m Cutting out a

sample of the fabric, the concentration of formaldehyde in the same manner as in Example 1 was measured and the drain, p p m in the mass 2.

[0132] Comparative Example 9

[0133] Average particle diameter of 1 0 μ m of calcium oxide hydrate powder to acrylic gelon (deodorant antimicrobial powder formulation ·) (Ganz Chemical Co. Ltd. (strain), the cross-xylene) may be incorporated into, fiber treating agent was prepared. · Deodorant antimicrobial amount of the powder formulation, the dry fiber treating agent 5 0 mass % on the basis the fishy. Polyester backing, laminated fiber treatment agent with the above-mentioned one with a roll coater, and dried at 1 0 0 °C for 3 0, 5 8 g/m by dry mass and the fiber treatment agent[2] · deodorant antimicrobial applied to the coated fabric to produce a poly ester group. 5 c m X 5 c m Switched for a sample of this backing out from, in the same manner as in Example 1 of the drain formaldehyde concentration was measured. 0.5 P p m by this degree is less than was the mass, the obtained deodorant ' applied to the texture of the crosslinked anti-microbial fabric was checked, a very hard, a practically usable was not obtained. The availability of the industrial

[0134] · Deodorant antimicrobial powder of the present invention, various kinds of odor, various bacteria, as the performance of the deodorant, antibacterial effect, are capable of being used in the field of a wide range.

Description

[0001] 明 細 書 消臭 ·抗菌性粉末製剤、 その製造方法及びこの粉末製剤を利用した製品 技術分野

[0002] 本発明は、 各種臭気、 各種細菌に対して、 高性能の消臭、 抗菌効果を有 する消臭 ·抗菌性粉末製剤、 その製造方法及びこの粉末製剤を利用した製 品に関する。 背景技術

[0003] 近年、 合成樹脂を用いた新建材、 家具、 壁紙、 自動車用レザー、 繊維製 品、 食品包装材、 飼料袋及び家庭用品などが広く普及している。 しかしな がら、 このような新建材や家具等において、 新建材や家具等自体、 あるい は接着剤や塗料から発生するホルムアルデヒド等の揮発性物質は、 近年、 シックハウス症候群の原因として大きな問題となっており、 ホルムアルデ ヒ ド等の揮発性物質の低減が要望されている。 また、 快適性の観点から、 病原性大腸菌 O— 1 5 7に代表される各種細菌に対して抗菌性を示し、 か つ使用上安全な抗菌剤が望まれている。 一方、 食品包装材及び飼料袋の分 野では、 熱可塑性フィルムが広く使用されているが、 内容物の保存性の観 点から、 安全で、 かつ抗菌性及び抗カビ性を示す抗菌剤が強く求められて いる。 また、 トイレの臭いやタバコの臭い等の消臭に効果を発揮する家庭 用消臭剤が求められている。

[0004] 安全な消臭 ·抗菌剤として、 貝殻を利用したものが提案されており、 例 えば、 貝殻を粉砕して得た炭酸カルシウム粉末を含有する消臭剤 (例えば、特開 2 0 0 1— 1 4 5 6 9 3号公報参照) 、 ホタテ貝の貝殻粉末を含有 する抗菌剤 (例えば、 特開 2 0 0 1— 1 9 9 8 2 3号公報参照) 、 ホタテ貝 殻の粉砕物を焼成してなる細菌抑制剤 (例えば、 特開 2002— 25 5 7 1 4号公報参照) が提案されている。 しかながら、 その消臭性能や抗菌性 能は、 ともに十分ではなかった。 発明の開示

[0005] 本発明は、 上記事情に鑑みなされたもので、 各種臭気、 各種細菌に対して、 高性能の消臭、 抗菌効果を有し、 広範囲の分野で使用可能な消臭,抗 菌粉末を提供することを目的とするものである。

[0006] 本発明者らは、 上記課題を解決するために鋭意研究を重ねた結果、 貝殻を焼成して得られた酸化カルシウムを水和した水和物であって、 特定の平 均粒径を有する酸化カルシウム水和物粉末が、 消臭 ·抗菌に有効であるこ とを見出した。 本発明はかかる知見に基づいて完成したものである。

[0007] すなわち、 本発明の要旨は下記のとおりである。

[0008] (1) 貝殻を焼成して得られた酸化カルシウムを水和してなる、 平均粒 径 1～40 μ mの消臭 ·抗菌性粉末製剤。

[0009] (2) 上記 (1) の粉末 5～95質量%と、 平均粒径 1～40 μ mの茶 粉末又は茶殻粉末 95～5質量%とからなる消臭 ·抗菌性粉末製剤。

[0010] (3) 平均粒径 1～40 mの水酸化カルシウム粉末 5～95質量%と, 平均粒径 1～40 μ mの茶粉末又は茶殻粉末 95～5質量%とからなる消 臭 ·抗菌性粉末製剤。

[0011] (4) 貝殻を 800～1300°Cで焼成して酸化カルシウムを得、 該酸 化カルシウムを水和して消臭 ·抗菌性粉末製剤を製造する方法において、 酸化カルシウムの 0 0 5～0. 1 5質量[9] /o水溶液の p Hが 9～13とな るように水和することを特徴とする消臭 ·抗菌性粉末製剤の製造方法。

[0012] (5) 上記 (4) の製造方法において、 貝殻を焼成して得られた焼成物 を 1 0 0 μ m以下の粒径となるように粉砕し、 該粉砕物 1 kgに対して水 0. 2～1リットルの割合で加えて酸化カルシウムを水和させることを特 徴とする消臭 ·抗菌性粉末製剤の製造方法。

[0013] (6) 上記 (1) ～ (3) のいずれかの消臭 ·抗菌性粉末製剤を 0. 1 ～30質量%配合してなる合成樹脂組成物。

[0014] (7) 上記 (1) ～ (3) のいずれかの消臭 ·抗菌性粉末製剤を含む表 面処理剤により表面処理されてなる消臭 ·抗菌性レザーであって、 消臭 · 抗菌性粉末製剤の配合量が、 表面処理剤の乾燥質量基準で 1～30質量% である消臭 ·抗菌性レザー。

[0015] (8) 上記 (1) ～ (3) のいずれかの消臭 ·抗菌性粉末製剤を乾燥質 量基準で 0. 1～35質量%配合してなる塗料。

[0016] (9) 上記 (1) ～ (3) のいずれかの消臭 ·抗菌性粉末製剤を乾燥質 量基準で 1～80質量%配合してなる繊維処理剤。

[0017] (10) 上記 (1) ～ (3) のいずれかの消臭 抗菌性粉末製剤を含む 処理剤により処理されてなる消臭 抗菌性繊維であって、 消臭 抗菌性粉 末製剤の配合量が、 処理剤の乾燥質量基準で1～70質量%である消臭・抗菌性繊維。

[0018] (11) 上記 (6) の合成樹脂組成物からなる消臭 抗菌性フィルム。 (12) 上記 (6) の合成樹脂組成物を発泡させてなる消臭 抗菌性発 泡材。

[0019] (13) 上記 (6) の合成樹脂組成物からなる消臭 抗菌性防草シート。 (14) 上記 (1) ～ (3) のいずれかの消臭 抗菌性粉末製剤であつ て、 平均粒径 1～10 μmの消臭 抗菌性粉末製剤を 0.5～20質量% 配合してなる熱可塑性樹脂組成物製不織布。

[0020] (15) 平均粒径 3～20 μmの酸化カルシウム水和物粉末を、 繊維に 対する乾燥質量 1～50 g/m^2 で用いて繊維を表処理してなる消臭 抗 菌性付与繊維。 (16) 上記 (10) 又は (15) に記載の消臭 抗菌性付与繊維により形成されてなるカ---シート又は床敷材天井材。

[0021] (17) 上記 (1) ～ (3) のいずれかに記載の消臭 抗菌性粉末製剤 及び平均粒径 1～40 μmの酸化カルシウム水和物粉末からなる消臭 抗 菌性混合粉末製剤を含有してなる台板。 発明を実施するための最良の形態

[0022] 本発明の消臭 抗菌性粉末製剤において用いる貝殻としては、 ほたて貝、あさり、 はまぐり、 牡蠣、 さざえ、 あわび及びムール貝等の貝殻が挙げられる。 このうち、 工業的に処理されている割合が高いほたて貝及び牡蠣の 貝殻が、 入手しやすい点から好適であり、 さらに、 不純物の含有量が少な く、 かつ大量の廃棄物を有効利用する観点から、 ほたて貝の貝殻が好適である。 貝殻を焼成して得られる酸化カルシウムには、 亜鉛、 鉄、 マグネシ ウム等の微量金属が含まれているので、 抗菌性付与の点で好適である。

[0023] 貝殻の焼成は、 貝殻を水で洗浄した後、 600～1300 °Cで、 3～6 時間行う。 焼成後、 焼成物から細かい砂等を除去し、 水を加えて酸化カル シウムを水和させる。 水和後、 5～40 °C程度に冷却して沈殿物を得、 この沈殿物を遠心分離機又はろ過機により分離し、 得られた分離物を乾燥させ、 分級することにより、 所定の平均粒径を有する酸化カルシウム水和物 が得られる。

[0024] 上記焼成は、 得られる酸化カルシウムの消臭 抗菌性能を向上させる点 から、 高温で行うことが重要であり、 焼成温度が 800 °C未満であると、 焼成が十分に行われないため、 消臭 抗菌性能が低下するおそれがある。 1300 °Cを超える温度で焼成しても、 消臭 抗菌性能がより向上するも のでもなく、 エネルギ---が無駄に消費されるだけである。 焼成条件は、 好 ましくは 900～1200°0で3～5時間である。 焼成物は、 砂等を取り 除いた後に粒径 100 μm以下に粉砕することが好ましい。 粉砕には、 ボ---ルミル及びジェットミル等を用いることができる。 また、 上記水和の際 に加える水の量は、 焼成物 1 kgに対して 0.2～1 リットルの割合とす ることが好ましい。

[0025] 上記焼成及び水和により得られる酸化カルシウム水和物は、 その 0.05～0.15質量%水溶液の pHが 9～13、 好ましくは 10～13であることが、 高い消臭 抗菌性能を示す上で重要である。 例えばほたての貝 殻を用いる場合、 温度 800～1300°Cで焼成して得られた焼成物を粒 径 100 Atm以下となるように粉砕し、この粉砕物 1 kgに対して水を 0.2～1 リットルの割合で加えて水和させ、 水和後の冷却温度を 5～40°C とすることにより、 上記 pHを有する酸化カルシウム水和物を得ることが できる。

[0026] 本発明において、 酸化カルシウム水和物粉末の平均粒径は 1～40 m であることを要する。 用途によっても異なるが、 通常は、 好ましくは 2～30 m, より好ましくは 3～20 zm、 さらに好ましくは 5～15 μm である。 この平均粒径が 1 m未満であると、 酸化カルシウム水和物粉末 が凝集しやすくなるため、 取り扱い難くなり、また 1 zm未満に微粉化す るのにも困難を伴う。 この平均粒径が 40 μmを超えると、 酸化カルシウ ム水和物粉末の表面積が小さくなるため、 消臭 抗菌性能が発現されず、 また、 粒径が 40 μmを大幅に超えるものも含まれることとなるので、 配 合時に異物となる。

[0027] 酸化カルシウム水和物粉末の平均粒径は、 ボールミルの粉砕時間又はジエツトミルによる粉砕圧力及び粉砕時間の調整により、 調整することができる。

[0028] 本発明においては、 酸化カルシウム水和物粉末に、 茶粉末又は茶殻粉末を混合することが、 消臭 抗菌の対象となる物質の範囲が拡大する点から好ましい。 具体的には、 いわゆるシックハウスの原因となるホルムアルデ ヒドはもちろんのこと、 アンモニア, 酢酸臭等の生活臭、 タバコの臭いも 消臭対象となる。

[0029] 茶としては、 例えば、 緑茶、 ウーロン茶、 紅茶、 麦茶及びプーアール茶等の各種茶が挙げられ、 消臭 抗菌性能に優れる点から、 緑茶、 紅茶及び ウ--ロン茶が好適である。 茶は一種を単独で用いてもよく、 二種以上を混 合して用いてもよい。 また、 茶粉末と茶殻粉末とを混合して用いてもよい 茶粉末又は茶殻粉末は、 茶又は茶殻を乾燥させた後、 ボウルミルやジェ ットミルで粉砕することにより得ることができる。 これらの粉末は、 平均 粒径が 1～40 μmであることが好ましく、 分散性及び消臭 抗菌性能向 上の点から、 5～35 μmが好ましい。 茶粉末又は茶殻粉末の平均粒径の 調整は、 上記酸化カルシウム水和物粉末における平均粒径の調整と同様に 行うことができる。

[0030] 上記酸化カルシウム水和物粉末と茶粉末又は茶殻粉末の使用割合は、 酸化カルシウム水和物粉末 5～95質量%、 茶粉末又は茶殻粉末 95～5質 量%が好ましい。 この使用割合は、 消臭の対象となる臭気、 抗菌の対象と なる菌の種類により調整する。 例えば、 ホルムアルデヒド、 酢酸、 硫化水 素等の酸性臭気に対しては、 酸化カルシウム水和物粉末の使用割合を増や すのが効果的であり、 その他の臭気の場合は逆である。 また、 着色を避け たい用途の場合には、 茶粉末又は茶殻粉末の使用割合を減少させることが 好ましいが、 5質量%未満であると、 アンモニア臭やタバコ臭に対する消 臭効果が十分ではない。 また、 茶粉末又は茶殻粉末の使用割合が 95質量 %を超えると、 ホルムアルデヒドや酢酸に対する消臭効果が低下する。 抗 菌性能については、 茶粉末又は茶殻粉末は、 枯草菌、 黄色ブドウ球菌及び 大腸菌等の広範囲の菌に対して抗菌性能を示すが、 黄色ブドウ球菌及び

大 腸菌に対しては、 酸化カルシウム水和物粉末の使用割合を増やすのが効果 的である。

[0031] 上記茶粉末又は茶殻粉末は、 上記酸化カルシウム水和物粉末と混合する代わりに、 工業的に製造される水酸化カルシウム粉末と混合することもで きる。 水酸化カルシウム粉末の平均粒径は、 上記酸化カルシウム水和物粉 末の平均粒径と同様に１～４０μｍであることを要する。 用途によっても 異なるが、 通常は、 好ましくは２～３０μｍ、 より好ましくは３～２０μｍ、 さらに好ましくは５～１５μｍである。 水酸化カルシウム粉末と茶粉 末又は茶殻粉末の使用割合は、 上記酸化カルシウム水和物粉末と茶粉末又 は茶殻粉末の使用割合と同様であり、 対象とする臭気や菌により、 上記と 同様に調整することができる。

[0032] 本発明の消臭 ･抗菌性粉末製剤、 すなわち、 酸化カルシウム水和物粉末、 酸化カルシウム水和物粉末と茶粉末又は茶殻粉末との混合粉末、 及び水酸 化カルシウムと茶粉末又は茶殻粉末との混合粉末は、 合成樹脂に配合する ことにより、 合成樹脂で形成する各種製品における消臭 ･抗菌に役立てる ことができる。 この場合、 消臭 ･抗菌性粉末製剤の配合割合は、 ０．１～３０質量％が好ましく、 ０．２～２０質量％がより好ましく、 ０．５～１０質量％がさらに好ましい。 この配合割合が ０．１質量％未満では、 消臭 ･殺菌効果が期待できず、 ３０質量％を超えると、 反 対に悪影響を及ぼす おそれがある。

[0033] 合成樹脂としては、 耐アルカリ性に劣るものでなければよい。 食品用包 装袋や飼料袋等に使用する場合は、 ポリエチレン、 ポリプロピレン、 ＰＶＣ (ポリ塩化ビニル) 及びポリ塩化ビニリデン等が挙げられる。 壁紙には･ ポリ塩化ビニル及び軟質ポリオレフィンが好適に使用される。 食品用包装 袋、 飼料袋及び壁紙の場合は、 フィルム状のものを用い、 その厚さは用途 により異なるが、 １５～１００μｍが好適である。 射出成形品には、 ポリエチレンやポリプロピレン等のポリオレフィン系樹脂はもちろんのこと、 ＡＢＳ (アクリロニトリル---ブタジエンースチレン) 樹脂やポリスチレン樹脂等のスチレン系樹脂、 ポリアクリロニトリル 樹脂やポリフエニレンスルフィド樹脂等の熱可塑性樹脂が好適に使用され る。 射出成形法以外の成形法の場合、 フ ノール樹脂、メラミン樹脂、ェポキシ樹脂及び尿素樹脂等の熱可塑性硬化樹脂が使用される。 さらに、 ェ チレン一プロピレン共重合体やスチレン一ブタジエン共重合体等の熱可塑 性エラストマーも使用することができる。

[0034] 本発明の消臭 ･抗菌性粉末製剤を、 ポリエステル繊維、 レーヨン繊維、 アタリル繊維及びポリプロピレン繊維等の繊維製品の、 消臭 ･抗菌性付与 繊維処理剤として使用する場合、 裏処理剤に配合することが、 使用の容易 性の点から好ましく、 特に、 自動車用シート、 家具用シート、 絨毯の消臭 ･抗菌性付与繊維処理剤として好適に使用される。 処理剤の基剤としては、 アクリル系樹脂や合成ゴムラテックス等を使用することができる。 処理剤 は、 この基剤に消臭 ･抗菌性粉末製剤を配合したものである。

[0035] 処理剤における消臭 ･抗菌性粉末製剤の配合量は、 処理剤の乾燥質量基準で１～８０質量％が好ましく、 ５～６５質量％がさらに好ましい。 この 配合量が１質量％未満であると、 消臭 ･抗菌効果が乏しく、 ８０質量％を 超えると消臭 ･抗菌性粉末製剤を配合する際に処理剤が硬くなり、 繊維製 品の風合いが損なわれることとなる。 なお、 消臭 ･抗菌性粉末製剤の平均 粒径は １～４０ｍであることを要し、 好ましくは ２～３０ｍ、 より好 ましくは ３～２０μｍ、 さらに好ましくは ５～１５μｍである。

[0036] 裏処理剤としての使用量は、 要求特性にもよるが、 乾燥質量で １～５０ｇ Ｚｍ^{2}が好ましく、 ５～３０ｇ /ｍ^{2}がさらに好ましい。 この使用量が １ｇ /ｍ^{2}未満では、 消臭性能が十分ではなく、 ５０ｇ /ｍ^{2}を超えると、 繊 維が硬くなるおそれがあり、 コストも上昇する。 なお、 本処理剤は、 ポリエチレン、 ポリプロピレン、 レーヨン、 ナイ口ン、 ビ-ロン及びポリエステル等を含む熱可塑性樹脂組成物からなる不織 布の処理剤としても広く使用することができる。

[0037] 本発明の消臭 ･抗菌性粉末製剤をレザ--の消臭《抗菌剤として使用する 場合、 消臭 ･抗菌性粉末製剤をウレタン樹脂等に配合して表面処理剤とし、 これをレザー表面に、 バーコ--ター及びロールコーター等により塗布すれ ばよい。 レザーとしては、 ＰＶＣレザ--、 ウレタンレザ--、 ォレフィン系 熱可塑性エラストマ--レザ--、 及び天然皮革などが挙げられる。 天然皮革 以外のものは、 基材層に表皮層が形成されたものであり、 基材層を形成す る素材としては、 ＰＶＣ、 ウレタン及びォレフィン系熱可塑性エラストマ 一等が挙げられる。 消臭 ･抗菌性効果を向上させるために、 基材層にも消 臭 ･抗菌性粉末製剤を含有させることができる。 表面処理剤における消臭

[0038] ･抗菌性粉末製剤の配合量は、 表面処理剤の乾燥質量基準で １～３０質量％が好ましく、 ５～２０質量％がさらに好ましい。 この配合量が １質量％ 未満であると、 消臭 抗菌効果が乏しく、 ３０質量％を超えると表皮層の 物性が低下する。

[0039] 本発明の消臭 抗菌性粉末製剤を、 各種椅子、 ソファ--及び自動車シ--トのクッション材に使用される軟質ウレタンフォームに配合したものは、 消臭 ･抗菌クッション材として好適に使用される。 また、 上記軟質ウレタ ンフォームの他、 ポリエチレンやポリプロピレン等の熱可塑性ポリオレフ ィン等のポリオレフィン樹脂、 ポリスチレン樹脂及び硬質ウレタン等を発 泡させた発泡材は、 家庭用品及び建築物用の断熱材として使用される。 こ れらのクッション材や発泡材における消臭 ･抗菌性粉末製剤の配合量は、 クッション材や発泡材の乾燥質量基準で １～３０質量％が好ましく、 ５～２０質量％がさらに好ましい。 この配合量が １質量％未満であると、 消臭 ･抗菌効果が乏しく、 ３０質量％を超えるとクッション性や断熱性が低下 する。 なお、 発泡材は公知の方法で製造することができる。

[0040] 本発明の消臭 ･抗菌性粉末製剤は、 防草シ--トに配合することもできるｃ消臭 ･抗菌防草シートは、 上述した食品包装用袋等と同様の素材からなり ･ 消臭、抗菌性粉末製剤が配合されたフィルム (厚さは限定されないが、 好 ましくは ２０～５０μｍ程度) をヤーン状に切断したものを編み込んだ ものであり、 公知の方法で製造することができる。 消臭 ･抗菌防草シートは、 農地の畔、 路地、 鉢物の下敷き等の用途に好適である。 また、 消臭 抗菌 防草シートは、 ポリプロピレン等からなる不織布に消臭 ･抗菌性粉末製剤 を配合したものであってもよい。

[0041] 本発明の消臭 抗菌性粉末製剤の用途としては、 建材用途、 自動車関連用途、 家庭用品用途も挙げられ、 具体的には、 建材用途として、 畳用張地、 畳用芯材、 防水防湿フィルム、 ガラス保護フィルム、 合板、 化粧合板、 パーティクルボード、 ファイバーボード、 チップボード、 集成材、 パネル、 ふすま及び障子紙等に配合し、 あるいはフローリング用建築仕上げ材として配合して使用することができる。 自動車関連用途としては、 天井材、 内 装用塗料及びエアコン用フィルター等が挙げられる。 家庭用品用途として は、 クッションカバー、 クッション材、 カーテン、 テーブルクロス及び各 種敷き材等が挙げられる。

[0042] また、 本発明の消臭 抗菌性粉末製剤を、 フローリング用ワックスに配 合したものは、 消臭 抗菌性ワックスとして好適に使用することができる。さらに、 本発明の消臭 抗菌性粉末製剤は、 消臭 抗菌塗料、 特に抗菌

[0043] ＯＡ機器用塗料、 家電製品用塗料及び家具用塗料等に配合して好適に使用することができる。

[0044] 塗料は、 溶剤系樹脂及び水系樹脂に、 本発明の消臭 抗菌性粉末製剤を、 乾燥質量基準で０．１～３５質量％配合することにより得ることができる。この場合、 消臭 抗菌性粉末製剤は、 平均粒径１～４０μｍの範囲であれば使用可能であるが、 好ましくは平均粒径１～２０μｍであり、３～１５ｍがより好ましい。 この平均粒径が１ｍ未満であると粉末が凝集しやすく、４０ﾊmを超えると、塗膜に凹凸が発生しやすくなる。 さらに、 最 大粒径が５０ｍ以下であることが、 平滑な表面を得る上で好ましい。

[0045] 溶剤系樹脂としては、 アクリル樹脂、 ウレタン樹脂、 アルキド樹脂及びアミノ系樹脂等が挙げられ、 これらは一種を単独で用いてもよく、 二種以上を混合して用いてもよい。 水系樹脂としては、 酢酸ビニル系エマルジョン、 アクリル系エマルジョン、 ウレタン系エマルジョン、 水溶性アクリル 樹脂、 水溶性ウレタン樹脂、 水溶性エポキシ樹脂及び水溶性アルキド樹脂 等が挙げられ、 これらは一種を単独で用いてもよく、 二種以上を混合して 用いてもよい。

[0046] これらの塗料の塗装対象物としては、 金属、 ガラス、 樹脂、 紙、 セラミックス及び木材等が挙げられ、 任意に選定することができる。

[0047] 塗装方法としては、 はけ塗り、 吹き付け塗装、 静電塗装及び粉体塗装等があり、 適宜選定できる。 これらの塗料は、 通常の塗料の物性を損なうこ となく、 抗菌性や消臭性が付与されたものである。

[0048] 消臭 抗菌性不織布は、 上述のように、 繊維処理材による方法の他に、 不織布の原料繊維を紡糸する段階で、 常法により消臭 抗菌性粉末製剤を 配合することにより得ることができる。 消臭 抗菌性粉末製剤の配合量は、 ０．５～２０質量％とすることが、 消臭 抗菌性効果及び成形性の点から 適切である。 不織布の製造方法は、 特に限定されるものではなく、 乾式法 や湿式法等の公知の方法により製造することができる。 具体的な製造方法 としては、 例えば、 ニードルパンチ機を用いて繊維同士を絡ませたり、 糸 を用いて縫い止める等の機械的接合方法、 熱、 圧力又はバインダーによる 接着方法等が挙げられる。

[0049] この場合、 微細な繊維として押し出すため、 消臭 抗菌性粉末製剤は、 平均粒径１～１０μｍが好ましく、２～５μｍがより好ましい。 この平均 粒径が１μｍ未満であると粉末が凝集しやすく、 加工上問題がある。 平均 粒径が１０ｍを超えると、 紡糸時に糸が切断しやすくなり、 細い繊維を 得ることが困難となる。

[0050] 不織布の原料繊維としては、 ポリエチレン、 ポリプロピレン及びナイロン等が好ましく使用されるが、 消臭 抗菌性粉末製剤を原料に配合できるものであればよく、 特に限定されるものではない。 実施例

[0051] 次に、 本発明を実施例及び比較例により更に具体的に説明するが、 本発明はこれらの例によってなんら限定されるものではない。

[0052] 実施例１

[0053] ほたての貝殻を水で洗浄した後、１１００℃にて４時間焼成し、メッシュサイズ５ｍｍの篩を用いて細かい砂を除去し、焼成物１ｋｇを得た。これに水０．５リットルを加えて酸化カルシウムを水和させ、２５℃に冷却し、沈殿物を吸引ろ過器を用いて分離し、分離物を１２０℃にて５時間かけて乾燥させた。この乾燥物をエアセパレーターにて分級し、平均粒径９ﾊmの酸化カルシウム水和物粉末（消臭 抗菌性粉末製剤）を得た。

[0054] 内容積３００ミリリットルの三角フラスコに１質量％ホルムアルデヒド水溶液を１．５マイクロリットル入れ、上記酸化カルシウム水和物粉末０．０５ｇ（１ｇ/リットル水溶液での ｐＨが１２．５）を入れ、２４℃にて１２０分間放置した後、北川式検知器によりフラスコ内のホルムアルデヒド濃度を測定した。 また、 後述する方法により、抗カビ試験を行った。結 果を表１に示す。

[0055] 実施例２

[0056] 緑茶葉を、水分量が０．５質量％となるように乾燥させ、シュレッダーにて粗粉砕し、ボールミル及びジェットミルにてさらに粉砕し、平均粒径

[0057] ３０ｍの茶粉末を得た。この茶粉末５０質量％と、実施例１で得られた酸化カルシウム水和物粉末５０質量％とを混合した消臭 抗菌性混合粉末 製剤（１ｇ/リットル水溶液での ｐＨが１１）０．０５ｇを使用して、実 施例１と同様にホルムアルデヒド濃度を測定した。また、抗カビ試験を行 った。結果を表１に示す。

[0058] 実施例３

[0059] 酸化カルシウム水和物粉末の代わりに平均粒径４μｍの水酸化カルシウム（１ｇ/リットル水溶液での ｐＨが１１）を用いた以外は、実施例２と 同様にして消臭 抗菌性混合粉末製剤を得、同様にホルムアルデヒド濃度 を測定した。また、抗カビ試験を行った。結果を表１に示す。

[0060] 実施例４

[0061] 実施例２で得られた消臭 抗菌性混合粉末製剤をポリカーボネート系ウレタン樹脂（大日精化工業社製、レザーミン Ｄ６６２５）に配合し、消臭 抗菌表面処理剤を作製した。混合粉末製剤の配合量は、表面処理剤の乾 燥質量基準で２０質量％と

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した。 ポリエステル繊維 6 5質量%とレーヨン 繊維 3 5質量%との混紡繊維の平織物からなる基布上に、 軟質ポリ塩化ビ ニル層が形成された P V Cレザーの表面に、 バーコーターにて上記表面処 理剤を塗布し、 乾燥させて厚さが約 1 0 μ mの表面処理層を有する消臭・抗菌処理レザーを得た。 このレザーから 5 c m X 5 c mのサンプルを切り 出し、 実施例 1と同様にホルムアルデヒド濃度を測定した。 結果を表 1に示す。

[0062] 実施例 5

[0063] 実施例 3で得られた消臭・抗菌性混合粉末製剤をアクリルェマルジヨン (ガンツ化成 (株) 製, クロスレン) に配合し、 繊維処理剤を作製した。 混合粉末製剤の配合量は、 繊維処理剤の乾燥質量基準で 1 5質量%とした。 ポリエステル系基布に、 ロールコーターにて上記繊維処理剤を積層し、 乾 燥させて厚さが約 1 00 μmの繊維処理剤層 (繊材) を有するシートを得 た。 このシートから 5 c mX 5 c mのサンプルを切り出し、 実施例 1と同 様にホルムアルデヒ ド濃度を測定した。 結果を表 1に示す。

[0064] 実施例 6

[0065] 実施例 4で作製した消臭・抗菌処理レザーから切り出した 5 cmX 5 c mのサンプルと、 4質量%アンモニア水溶液 5マイクロリットルを内容積

[0066] 300ミリ リットルの三角フラスコに入れ、 25°Cにて 1 20分間放置した後、 北川式検知器によりフラスコ内のアンモニア濃度を測定したところ、

[0067] 145質量 p p mであった。

[0068] 実施例 7

[0069] 実施例 1で得られた酸化カルシウム水和物粉末 1質量部と、 直鎖低密度 ポリエチレン (出光石油化学 (株) 製, I DEM I TSU LL 0 1 3 4, メルトフロー レー ト : 1 2 gZI O分) 99質量部とをドライブレ ンドし、 その後、 混練用二軸押出機 (混練部 =40 mm φ) にて 200°C において混練し、 樹脂組成物を得た。 この樹脂組成物を、 押出機 (押出部 =50 mm φ) にてインフレーション成汗 SL、 幅 60 mm、 厚さ 40 μ m のインフレーションフィルムを作製した。 このフィルムから 5 cmX 5 c mのサンプルを切り出し、 実施例 1と同様にホルムアルデヒ ド濃度を測定 した。

[0070] 実施例 8

[0071] 実施例 1で得られた酸化カルシウム水和物粉末を溶剤系ウレタン樹脂溶 液中に、 乾燥質量基準で 5質量%配合し、 塗料を調製した。 次に、 この塗 料をスプレーガン (岩田塗装機工業 (株) 製) により、 厚さ 2mmの AB S (アクリ ロニトリル―ブタジエン―スチレン) 板に対して吹き付け塗装 を行った。 この AB S板から 5 c mX 5 c mのサンプルを切り出し、 実施 W 200 例 1と同様にホルムアルデヒ ド濃度を測定した。 結果を表 1に示す。

[0072] 実施例 9

[0073] 実施例 1で得られた酸化カルシウム水和物粉末、 ポリオール成分、 イソシァネート成分、 発泡剤、 触媒及び整泡剤を下記に示す割合で配合して、 常法により、 酸化カルシウム水和物粉末を 5質量%含む消臭・抗菌性ウレ タンフォームを作製した。 このウレタンフォームから 5 c m X 5 c m X 5 c mのサンプルを切り出し、 実施例 1と同様にホルムアルデヒド濃度を測 定した。 結果を表 1に示す。 <配合成分 >

[0074] ポリオール (ダウポリ ウレタン社製, # 3 0 0 0) 1 00質量部 イソシァネート (ダウポリ ウレタン社製, T― 8 0) 40質量部 発泡剤 (水) 2. 9質量部 アミン系触媒 (エアプロダクツ社製, 3 3 LV) 3質量部 アミン系触媒 (エアプロダクツ社製, AT 3 3) 3質量部 スズ系触媒 (日東化成社製, T― 9) 3質量部 整泡剤 (日本ュユカー社製, L 6 20 2) 3質量部 酸化カルシウム水和物粉末 8質量部 実施例 1 0

[0075] 実施例 1で得られた酸化カルシウム水和物粉末を分級して平均粒径 3 μ mの粉末を得、 これをポリプロピレン樹脂 (M I = 5 0 g/ 1 0 分) に配合して、 酸化カルシウム水和物粉末を 40質量% 含むマスタ ーバッチを得た。 このマスターバッチ 5質量部をポリプロピレン樹脂 (M I = 5 0 gZ 1 0分) 9 5質量部に配合し、 酸化カルシウム水和 物粉末を 2質量%含む組成物を調製した。 この組成物を、 押出機に投 入し、 樹脂温度 2 2 0 °Cで溶融混練した後、 混練物をダイ (2 0 0ホ ール) から押し出し、 高速エアーで延伸し、 細化することにより、 連 続した長繊維フィラメントを作製した。 なお、 M I (メルトインデックス) は、 J I S K 6 9 2 1に準拠し、 2 3 0 °C、 荷重 2 1 6 k g f (2 1 . 1 8 N) で測定した。

[0076] このフィラメントをランダムに集積してシートとし、 このシートを エンボスロールと平滑ロールとの間に通し、 1 4 0 °Cにて熱エンボス して、 不織布を作製した。

[0077] 得られた不織布は、 平均繊維径 2 2. 4 ^ m、 平均目付 5 0 gノ m^{2}であった。 この不織布から 5 c m X 5 c mのサンプルを切り出し. 実施例 1 と同様にホルムアルデヒド濃度を測定した。 結果を表 1に示す。

[0078] 比較例 1

[0079] 実施例 1において、 酸化カルシウム水和物粉末を使用しないでホルムア ルデヒド濃度を測定した。 また、 抗カビ試験を行った。 結果を表 1に示すc比較例 2

[0080] 実施例 1において、 酸化カルシウム水和物粉末を平均粒径 1 μmの水酸 化マグネシウム (1 gZリットル水溶液での p Hが 7) に変えた以外は、 実施例 1と同様にホルムアルデヒド濃度を測定した。 また、 抗カビ試験を 行った。 結果を表 1に示す。

[0081] 比較例 3

[0082] 実施例 1において、 焼成温度を 600°Cとし、 焼成後の水和工程を経ないこと以外は実施例 1と同様にして得られた平均粒径 3 0 μmの粉末 (1 g/リットル水溶液での p Hが 8) について、 実施例 1と同様にホルムア ルデヒド濃度を測定した。 また、 抗カビ試験を行った。 結果を表 1に示す。比較例 4

[0083] 実施例 5において、 消臭・抗菌性混合粉末製剤を使用しない以外は、 実 施例 5と同様にホルムアルデヒド濃度を測定した。 結果を表 1に示す。 比較例 5

[0084] 実施例 6において、 消臭 ·抗菌処理レザ---の代わりに、 実施例 4で用いたものと同様で消臭 ·抗菌性混合粉末製剤を配合しない ＰＶＣレザ---を使 用した以外は、 実施例 5と同様にアンモニア濃度を測定したところ、 ２３０質量ｐｐｍであった。

[0085] [抗カビ試験]

[0086] １．供試菌株: Ｍｕｃｏｒ属糸状菌

[0087] ２．使用培地

[0088] ①培地１：グルコース無機塩培地

[0089] グノレコース３００ｇ

[0090] 硝酸アンモニウム３０ｇ

[0091] 燐酸---力リウム０ｇ

[0092] 硫酸マグネシゥム０５ｇ

[0093] 塩化力リウム０２５ｇ

[0094] 硫酸第---鉄 '０００２ｇ

[0095] 脱イオン水０００ミリリットル

[0096] ②培地２：グルコース無機塩寒天培地

[0097] グノレコース３０．０ｇ

[0098] 硝酸アンモニウム３．０ｇ

[0099] 燐酸---力リゥム１．０ｇ

[0100] 硫酸マグネシゥム０．５ｇ

[0101] 塩化力リウム０．２５ｇ

[0102] 硫酸第---鉄０．００２ｇ

[0103] 脱イオン水１０００ミリリットル

[0104] 寒天２５．０ｇ３．胞子液の調製

[0105] Ｍｕｃｏｒ属糸状菌をＰＤＡ (ポテトデキストロース寒天培地) プレ---ト上で胞子形成 (２８ ℃で---週間培養) させたものに対し、 滅菌水１０ミリリットルを用いて胞子/菌糸懸濁液を調製した。

[0106] 次いで、 これを滅菌脱脂綿で濾過した後、 遠心分離により胞子を沈殿させ、 上記培地１ (寒天抜き、 グルコース無機塩培地) ２０ミリリットルに 再懸濁することによって胞子液を調製した。

[0107] ４．試験片の設置及び植菌

[0108] 上記培地２ (グルコース無機塩寒天培地) 上に、 実施例１～３及び比較 例１～３で使用した消臭 ·抗菌性粉末製剤の５ｇ Zリットル水溶液 (０．５質量^{0} /$_{0}$) １ミリリットルをプレ---ト上に塗布した。

[0109] これに上記胞子液を試験片上で微細水滴が形成される程度に噴霧するこ とにより植菌し、 プレ---トをパラフィンフィルムでシ---ルした後、 恒温恒 室槽で培養した。 噴霧は噴霧器と 2連球を用いて行い、 培養条件は、 培養 温度３０ ℃、 湿度９５％、 培養期間は 4週間とした。

[0110] ５．糸状菌生育観察:

[0111] 植菌してから 2週間後に、 試験プレ---ト表面上の菌糸の生育度合いを巨 視観察した。

[0112] ６．結果の判定:

[0113] 以下に示す判定基準により、 菌糸の生育状況の経時変化を判定した。

[0114] [生育判定基準]

[0115] 一 :肉眼において、 菌糸 (カビ) の生育が認められない

[0116] +Z--- :菌糸の生育が僅かに認められる

[0117] ＋ :菌糸の生育が認められる

[0118] 菌糸の顕著な生育及び胞子形成が認められる 表１

[0119]

[0120] ND :検知せず。 実施例１１

[0121] 平均粒径１０μｍの酸化カルシウム水和物粉末 (消臭 ·抗菌粉末製剤) をアクリルエマルジヨン (ガンツ化成 (株) 製、 クロスレン) に配合し、 繊維処理剤を作製した。 消臭 ·抗菌粉末製剤の配合量は、 繊維処理剤の乾 燥質量基準で５０質量％とした。 ポリエステル系基布に、 ロールコータ ---にて上記繊維処理剤を積層し、１００ ℃にて３０分間乾燥させて、 繊維処理剤が乾燥質量で２４ g/m^2塗布された消臭 ·抗菌性付与ポリ エステル基布を製造した。 この基布から５ｃｍＸ５ｃｍのサンプルを切り出し、 実施例１と同様にホルムアルデヒド濃度を測定したところ、 ０．５質量ｐｐｍ未満であった。

[0122] 実施例１２平均粒径５μmの酸化カルシウム水和物粉末 (消臭 ·抗菌粉末製剤) をアクリルエマルジヨン (ガンツ化成 (株) 製、 クロスレン) に配合し、 繊 維処理剤を作製した。 消臭 ·抗菌粉末製剤の配合量は、 繊維処理剤の乾燥 質量基準で 50質量。/。とした。 ポリエステル系基布に、 ロールコーター にて上記繊維処理剤を積層し、１００ ℃にて３０分間乾燥させて、 繊 維処理剤が乾燥質量で２６ g /m^2塗布された消臭 ·抗菌性付与ポリエ ステル基布を製造した。 この基布から５ｃｍＸ５ｃｍのサンプルを切り 出し、 実施例１と同様にホルムアルデヒド濃度を測定したところ、 0. 5質量ｐｐｍ未満であった。

[0123] 実施例１３

[0124] 平均粒径１０の酸化カルシウム水和物粉末 (消臭 ·抗菌粉末製剤) をアクリルエマルジヨン (ガンツ化成 (株) 製、 クロスレン) に配合し、 繊維処理剤を作製した。 消臭 ·抗菌粉末製剤の配合量は、 繊維処理剤の乾 燥質量基準で 50質量%とした。 ポリエステル系基布に、 ロールコータ ーにて上記繊維処理剤を積層し、１００°

Cにて３０分間乾燥させて、繊維処理剤が乾燥質量で５ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍＸ５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、０.５質量ｐｐｍ未満であった。

[0125] 実施例 14

[0126] 平均粒径１０ｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジヨン（ガンツ化成（株）製、クロスレン）に配合し、酸化カルシウム水和物粉末濃度が２質量%の分散液を作製した。この分散液を、３０ｃｍＸ３０ｃｍＸ１２ｍｍの合板上にスプレーを用いて塗布し、酸化カルシウム水和物粉末が２ｇＺ９００ｃｍ^{2}で塗布された、ホルムアルデヒド発生低減合板を製造した。この合板のホルムアルデヒド放散量をＪＩＳＡ１９０１に規定される方法で測定したところ、０.２ｍｇ／リットルであった。一方、未処理の同一材質合板のホルムアルデヒド放散量は０.５ｍｇＺリットルであった。

[0127] 比較例 6

[0128] 水９０質量%、平均粒径８μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）１０質量%の割合で混合した分散液を、湿式ビーズミル（アシザワファインテック（株）製、ＬＭＺ-２）にて湿式粉砕し、平均粒径０.６μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を得た。この消臭・抗菌粉末製剤をアクリルエマルジヨン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量%とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で２７ｇＺｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布は、消臭・抗菌粉末製剤が凝集し、実使用に耐え得るものではなかった。

[0129] 比較例 7

[0130] 平均粒径８μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を分級して得られた平均粒径４５μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）を、アクリルエマルジヨン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量%とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層しようとしたところ、繊維処理剤が固液分離し、積層することができなかった。

[0131] 比較例８平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジヨン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量%とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で０.５ｇＺｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍＸ５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定したところ、２質量ｐｐｍであった。

[0132] 比較例９

[0133] 平均粒径１０μｍの酸化カルシウム水和物粉末（消臭・抗菌粉末製剤）をアクリルエマルジヨン（ガンツ化成（株）製、クロスレン）に配合し、繊維処理剤を作製した。消臭・抗菌粉末製剤の配合量は、繊維処理剤の乾燥質量基準で５０質量%とした。ポリエステル系基布に、ロールコーターにて上記繊維処理剤を積層し、１００℃にて３０分間乾燥させて、繊維処理剤が乾燥質量で５５ｇ／ｍ^{2}塗布された消臭・抗菌性付与ポリエステル基布を製造した。この基布から５ｃｍＸ５ｃｍのサンプルを切り出し、実施例１と同様にホルムアルデヒド濃度を測定した。この濃度は０.５質量ｐｐｍ未満であったが、得られた消臭・抗菌性付与ポリエステル基布の風合いを確認したところ、非常に硬く、実使用に耐え得るものではなかった。産業上の利用可能性

[0134] 本発明の消臭・抗菌性粉末は、各種臭気、各種細菌に対して、高性能の消臭、抗菌効果を有し、広範囲の分野で使用可能なものである。

Google Patents

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A solid concentrate concentrate composition comprising: (a) from 5% to 25%by weight of zinc pyrithione (ZPT), (b) from 8% to 85% by weight of at least one surfactant, preferably, wherein the solid concentrate composition has a penetration hardness of between 20N and 50N according to the test method as disclosed herein.

Cla Search

A61K8/58 Cosmetics or similar toilet preparations characterised by the composition containing organic compounds containing atoms other than carbon, hydrogen, halogen, oxygen, nitrogen, sulfur or phosphorus

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Other languages: FrenchInventor: Charlie Reyes Salvador, Chunpeng Jiang, Zhe LIU, Gbenga Segun SHOWOLE, Yanan Sun, Anurag MAKHANDI, Raul Songco NICDAO, Brian Joseph Lemberg, Edward Dewey Smith, III, Juan Wang, Enjun CHENG, Casey Patrick Kelly, Jason Edward COOK, Patrick Christopher STRENGER, Xiujun Xu, Kai Shiqing Wei

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Description

SOLID CONCENTRATE COMPOSITIONS CONTAINING ZINC PYRITHIONE

FIELD OF THE INVENTION

The present invention relates to solid concentrate compositions comprising zinc pyrithione (ZPT).

BACKGROUND OF THE INVENTION

Pyrithione (also known as 1-Hydroxy-2-pyridinethione, 2-pyridinethiol-1-oxide, 2- mercaptopyridine-N-oxide, pyridine-2-thione-N-oxide, pyridinethione-N-oxide, 2-pyridinethione, pyridinethione, or simply "PT") has been noted for its bactericidal and fungicidal activities. Pyrithione is a bidentate ligand that forms stable complexes with most transitional metals. Metallization of pyrithione often results in highly augmented biocidial activities. Metal salts of pyrithione, such as for example, sodium pyrithione, magnesium pyrithione, barium pyrithione, bismuth pyrithione, strontium pyrithione, copper pyrithione, zinc pyrithione, cadmium pyrithione, and zirconium pyrithione, are widely used as fungicides and bactericides in a broad spectrum of commercial products, such as metalworking fluids, lubricants, paints, cosmetics and toiletries.

Zinc pyrithione (or "ZPT") is especially useful as a broad-spectrum anti-microbial agent and preservative. It is active against both gram-positive and gram-negative bacteria, as well as fungi and yeasts. Therefore, ZPT has been used in various personal care compositions, such as for example, anti-dandruff shampoos, hair conditioners, leave-on tonics, and anti-microbial foot powders. Bar soap is a popular product form for cleansing. A bar soap comprising ZPT is particularly desirable for its broad-spectrum anti-microbial efficacy.

ZPT is a crystalline solid that can generally be mixed into a soap composition from either a powder form, or from a liquid dispersion. Powder form can be difficult to handle because they can generate dust during dosing and mixing. As a result, powder form may require complex and expensive dust handling equipment. Although liquid dispersions are generally easier to handle, they can have their own complexities for shipping, storing and processing. For example, liquid slurries must be regularly stirred during processing to avoid settling, and accurately dosed in every manufacturing line. Additionally, liquid slurries are usually maintained in a system requiring extensive cleaning, sanitizing and waste-water recovery, and may even require a separate preservative just for the liquid slurry. Therefore, a need exist for a convenient means to dose ZPT into a soap composition that can overcome these difficulties. SUMMARY OF THE INVENTION

The present invention is directed to a solid concentrate composition comprising: (a) from 5% to 25% by weight of a zinc pyrithione (ZPT), and (b) from 8% to 85% by weight of at least one surfactant. In an embodiment, the solid concentrate composition has a penetration hardness of between 20 N and 50 N. These and other aspects of the present invention will become more apparent upon reading the following drawings and detailed description of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS FIG. 1 is a bar graph showing the percentage loss of ZPT in seven (7) ZPT-containing concentrate compositions of the present

invention.

FIGs. 2A-2C depict the cylinder, sleeve and weight used to form the cake form from the concentrate compositions for the penetration hardness test.

FIG. 2D shows the Force Gauge Machine (Model Z-44; IMADA Inc., IL, USA) used for the penetration hardness test.

FIGs. 3A-D are photos of solid concentrate compositions of varying penetration hardness.

DETAILED DESCRIPTION OF THE INVENTION

Except as otherwise noted, the articles "a", "an", and "the" mean "one or more."

The term "comprising" means that other steps and other ingredients which do not affect the end result can be added, and this term encompasses the terms "consisting of and "consisting essentially of. The compositions and methods/processes of the present invention can comprise, consist of, and consist essentially of the essential elements and limitations of the invention described herein, as well as any of the additional or optional ingredients, components, steps, or limitations described herein. Particularly, the compositions of the present invention contain ZPT, and one or more additional or optional ingredients as described hereinafter.

All percentages, parts and ratios are based upon the total weight of the compositions of the present invention, unless otherwise specified. All such weights as they pertain to listed ingredients are based on the active level and, therefore do not include carriers or by-products that may be included in commercially available materials. The components, including those which may optionally be added, as well as methods for preparation, and methods for use, are described in detail below.

"Solid" as used herein refers to concentrate compositions that are solid (i.e., dry and not flowable like a liquid or semi-liquid) at about 15%> relative humidity, and at about 25° C, unless otherwise specified. The concentrate composition in solid form can be in the form of an agglomerate, granule, flake, extrudate, bar, tablet or any combination thereof. Preferably, the term "solid" refers to materials that have a penetration hardness of 20 N to 50 N as measured by a Force Gauge Machine (Model Z2-44) available from IMADA Inc. (Illinois, USA) according to the test method as described herein.

"Penetration hardness" as used herein is a reflection of how much force (N) is required to break a cake formed from the concentrate composition of the present invention according to the test method as described herein. "Penetration hardness" is also a way to represent the stickiness of the concentrate composition. For example, higher values represent more sticky material, and lower values represent more powdery material.

"Bar soaps" as used herein refers to solid or semi-solid articles for washing, bathing, and cleaning that contain either soap surfactants, synthetic surfactants, or mixtures thereof (i.e., semisynthetics) as described hereinafter. A bar soap as used herein is not limited to a bar shape but can have any regular or irregular shape, including but not limited to: cubic, rectangular, spherical, oval, cylindrical, pyramidal and the like. The bar soaps of the present invention are preferably, but not necessarily, characterized by a volume ranging from 1 cm^3 to 1,000 cm^3, more preferably from 10 cm^3 to 500 cm^3, and most preferably from 50 cm^3 to 200 cm^3, and a weight ranging from 0.5 g to 5 Kg, more preferably from 1 g to 1 Kg, and most preferably from 10 g to 500 g. SOLID CONCENTRATE COMPOSITIONS

The present invention relates to a solid concentrate composition comprising: (a) from 5% to 25% by weight of a zinc pyrithione (ZPT); and (b) from 8% to 85% by weight of at least one surfactant.

The inventors have found a simple and reliable means to dose ZPT into a soap composition by concentrating the ZPT active into a solid concentrate composition having from 5% to 25% by weight of a ZPT. For example, a solid concentrate composition can be prepared at a concentration about 10-fold or even greater, higher than the desired ZPT concentration in the soap composition. Such a ZPT-containing solid concentrate composition can be prepared at one single manufacturing station and cost effectively shipped in its compacted form to other sites for dilution with standard medium known to those skilled in the art to form finished products.

The solid concentrate composition thus prepared is easy to store, transport, and add to any soap manufacturing line using simple equipment that already exists on soap manufacturing lines, without the need for incurring additional complex material handling capital. Because the concentrate composition is a solid with compliant properties, like a soap noodle, it is easy to distribute the concentrate composition throughout the whole soap batch homogeneously during the soap manufacturing process by the conventional soap milling and extruding operations. For example, the solid concentrate composition can be diluted into solid bar soaps. Alternatively, the solid concentrate composition can be dissolved into a liquid tank to form liquid soap compositions.

According to one embodiment, the concentrate composition further comprises (c) from 1.4% to 15% by weight of a chelant, wherein the chelant has a log KZ „L of greater than 2, wherein the log K-znL is the log of a conditional stability constant of the chelant with Zn calculated at pH 7, 25 °C, 0.1 M NaCl. Preferably, the chelant has a

Conditional Stability Constant for zinc higher than 3, and more preferably higher than 6. It has been found that in order to maintain a desired level of the affinity for the chelant, the pH of the concentrate composition further comprises a sufficient quantity of a buffer system comprising an organic acid and/or a salt thereof to provide the concentrate composition with a pH from about 4 to about 11 at 25 °C. The conditional stability constant is a parameter commonly used in the art to practically assess the stability of metal-chelant complex at a given pH. A detailed discussion on conditional stability constant can be found for example in "Dow Chelating Agents" published by Dow Chemical Company Limited, and U.S. Publication US2013/0174863 (P&G), incorporated herein by reference. The calculated stability constants for a range of chelants with Zn are given below in Table 1 :

Table 1

	Calculated Conditional Stability Constants for Chelants with Zn at specified pH (log Conditional Stability Constant*)	
Chelants	Zn (pH 7)	Zn (pH 10)
HPNO	5.1	5.1
HEDP	7.3	10.1
EDTA	14.0	16.4

*Ionic strength of 0.1.

In another embodiment, the concentrate composition further comprises (c) from 1.4% to 15% be weight of a chelant, wherein the chelant is a metal phosphonate complex comprising one or more phosphonate chelants co-ordinately bonded to one or more metal ions. In yet another embodiment, the concentrate composition further comprises (c) from 1.4% to 15% be weight of a chelant, wherein the chelant is a metal-pyridine oxide complex that is co-ordinately bonded to a metal ion. Alternatively, the concentrate composition of the present invention further comprises (c) from 1.5% to 5%, or 3% to 10%, or 10% to 15%, or 9% to 12% by weight of a chelant, wherein the chelant is a metal-pyridine oxide complex or a metal phosphonate complex that is co-ordinately bonded to one or more metal ions.

The metal in the metal-phosphonate complex or metal pyridine oxide complex is selected from the group consisting of iron, copper and zinc, preferably zinc. However, it will be understood by one skilled in the art that other metals can be selected according to the Irving Williams Series. Without wishing to be bound by theory, according to Irving Williams Series, a more stable complex can be formed between phosphonate or pyridine oxide and metal ions having smaller ionic radius. For example, Fe^{3+} has a radius of 0.64 A, which is smaller than Cu^{2+} which has a radius of 0.73 A, and which is in turn smaller than that of Zn^{2+} 0.74A. Thus, this might help to explain why there can be pyrithione discoloration in ZPT-containing bar soaps in the presence of other transition metal sources (e.g., copper and iron).

The present invention also relates to low moisture solid concentrate compositions due to the introduction of water by ZPT. Therefore, more ZPT in the concentrate composition raises the water content of the concentrate composition so that it can become sticky and challenging to process. Preferably, by limiting the water level to, e.g., 0%> to 10%>, alternatively 3% to 10%>, alternatively 0% to 5%, low moisture noodles would allow for higher ZPT concentration. The additional benefit of having low water level means that less or no preservative is required to be added to the concentrate composition.

One important advantage of the concentrate composition is that it eliminates the need for special equipment to manufacture the concentrate at every production site. This can be important in light of cost considerations. However, as noted above, by introducing higher levels of ZPT there is the potential stickiness issue delivered by higher concentrations of ZPT and associated water. The inventors have surprisingly discovered that a solid concentrate composition comprising: (a) from 5% to 25% by weight of a ZPT; and (b) from 8% to 85% by weight of at least one surfactant, wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as described herein, avoids, or at least mitigates, aid to reduce and/or eliminate the stickiness problem. ZINC PYRITHIONE (ZPT)

Preferably, but not necessarily, ZPT is present in the concentrate composition as a spherical or platelet form, while the ZPT particles have an average size of up to about 20 microns, more preferably up to about 10 microns, even more preferably up to about 5 microns, and most preferably up to about 2.5 microns to about 0.3 microns, 0.2 microns or 0.1 microns, as determined according to light scattering based particle size measurements as known to those skilled in the art using equipment, such as, Horiba LA910 particle size apparatus or its equivalent. Alternatively, ZPT is present in a particulate form that is non-platelet and non- spherical, having a configuration selected from the group consisting of rods, needles, cylinders, cones, ellipsoids, prisms, parallelepipeds, pyramids, tetrahedrons, hexahedrons, octahedrons, dodecahedrons,

icosahedrons, and combinations thereof, as described by U.S. Patent 6,242,007.

ZPT may be incorporated into the concentrate composition of the present invention in the form of a powder or slurry. For example, ZPT can be added to the concentrate composition as a powder, then only one dust handling apparati is needed for one manufacturing line. Thereafter, the manufacturing process would not require any other manufacturing line to require the special dust handling machinery and thereby minimizing capital expenditures. In a preferred embodiment, the ZPT included in the solid concentrate composition is a dry powder ZPT in platelet particle form ("platelet ZPT"). The platelet ZPT can have a median particle diameter of, for example, from about 0.05 to about 10 microns, alternatively from about 0.1 to about 8 microns, and alternatively from about 0.2 to about 5 microns, and alternatively about 3 microns. The platelet ZPT can also have a thickness of, for example, from about 0.1 to about 15 microns, alternatively from about 0.5 to about 1 micron, alternatively from about 0.6 to about 0.8 microns, and alternatively from about 0.6 to about 0.7 microns, as described in U.S. Patent Publication 2012/0219610. The platelet ZPT thickness can be measured microscopically using an electron scanning microscope as known to those skilled in the art, or alternatively measured from microphotographs .

ZPT as used in the present invention may be made by reacting 1-hydroxy-2- pyridinethione (i.e., pyrithione acid) or a soluble salt thereof with a zinc salt (e.g., $ZnSO_4$) to form a ZPT precipitate, as illustrated by the disclosures of U.S. Patent 2,809,971 , or processed into platelet ZPT using, for example, sonic energy as illustrated by U.S. Patent 6,682,724, or by any other methods currently known in the art.

Solid concentrate compositions of the present invention can be used in order to deliver a less expensive product. The ZPT level in the concentrate composition of the present invention is from 5% to 25% by weight of the ZPT. In an embodiment, the level is from 6% to 8% by weight of the ZPT. Higher levels of ZPT in the concentrate composition is beneficial because less of the concentrate composition is required thus permitting for a less expensive product. Without wishing to be bound by theory, higher ZPT activity in the ZPT raw material used in the concentrate composition would be advantageous because lower amounts of the ZPT would be required thereby introducing less water, so that higher ZPT level can be present in the concentrate composition.

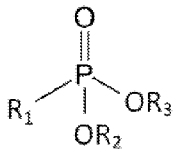
In an embodiment, the solid concentrate composition can be used to form bar soaps. Preferably, but not necessarily, the bar soaps of the present invention contain ZPT in the amount ranging from about 0.01% to about 10% by total weight of such compositions. More preferably, such bar soaps contain from about 0.1% to about 5.0% ZPT by total weight.

METAL-PHOSPHONATE COMPLEX

In an embodiment, the solid concentrate compositions of the present invention may comprise a metal-phosphonate complex, which comprises one or more phosphonate chelants that are co-ordinately bonded to one or more metal ions. The metal is selected from the group consisting of iron, copper and zinc. However, it will be understood by one skilled in the art that other metals can be selected according to the Irving Wiliams Series, which refers to the relative stability of complexes formed by a metal ion.

In a preferred embodiment, the metal is zinc. Such zinc-phosphonate complex has a surprising and unexpected effect on stabilizing the ZPT against potential transchelation by iron chelating iron that may be present and improving the discoloration resistance of the ZPT- containing concentrate compositions and/or bar soaps made from such concentrate compositions, which is demonstrated by a significant increase in its resistance to laboratory-induced discoloration in comparison with control samples containing ZPT only or with uncomplexed phosphonate chelant.

In a particularly preferred embodiment of the present invention, the phosphonate chelant comprises one or more functional groups of the formula (I):

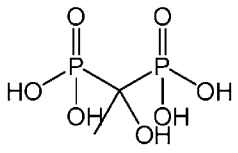


(I),

wherein Ri is a linear, branched or cyclic, saturated or unsaturated, substituted or unsubstituted C_{1}-C_{20} hydrocarbon group, and wherein R_2 and R_3 are independently selected from the group consisting of hydrogen and Ri. Preferably, both R_2 and R_3 are hydrogen. Exemplary phosphonate chelants that are suitable for practice of the present invention include, but are not limited to. 2-aminoethyl phosphoric acid (AEP), N-phosphonomethyl aminodiacetic acid (PMIDA), 1-hydroxyethane-1,1-diphosphonic acid (HEDP), amino tris(methylene phosphonic acid) (ATMP), ethylenediamine tetra(methylene phosphenic acid) (EDTMP), diethylenetriamine penta(methylene phosphonic

acid) (DTPMP), phytic acid, and nitriliotrimethylene phosphonic acid (NTP).

A representative species of phosphonate chelant that is particularly useful for the practice of the present invention is HEDP, which has the chemical structure of formula (II):



(II).

As a chelant, HEDP is capable of forming coordination complexes with transition metal ions in solution. Specifically, one or more HEDP can be bonded to one or more zinc ions to form a Zn-HEDP complex, which is a particularly preferred Zn-phosphonate compound for the present invention. It is important to note that zinc ions may be able to form various complexes with HEDP, with one or more HEDP attached to one or more zinc ions. In solution, zinc ions and HEDP may undergo speciation to form a mixture of different complex species, and the relative concentration of such complex species can vary depending on the chemical environment they are in, such as pH and the presence of other metal ions or chelant species. For ease of reference, all such complex species are herein referred to as the "Zn-HEDP complex," regardless of the actual number of HEDP or zinc ions included, and they are all included within the scope of the present invention.

The amount of zinc-phosphonate complex present in the concentrate compositions of the present invention may range from about 1% to about 15% by total weight of such concentrate compositions. More preferably, such concentrate compositions contains from about 2% to about 10% zinc-phosphonate complex, still more preferably from about 4% to about 8% or from about 4.5% to about 7% by total weight.

The zinc-phosphonate complex as used in the present invention can be pre-formed by reacting the phosphonate chelant with zinc oxide or a soluble zinc salt, such as $ZnSO_4$, $ZnCl_2$, or a mixture thereof. The reactant solution can then be added into the concentrate compositions.

Alternatively, the zinc-phosphonate complex can be formed in situ by directly adding the precursors, i.e., the phosphonate chelant and zinc oxide or the soluble zinc salt, into the concentrate compositions, which will directly complex with each other in the concentrate compositions. The phosphonate compound and zinc oxide or zinc salt can be added either in dry power form or pre-dissolved/dispersed in a solution.

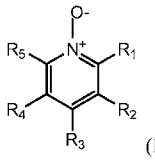
In another embodiment, the phosphonate chelant comprises a zinc-phosphonate complex and wherein the molar ratio of zinc to the phosphonate chelant ranges from 2: 1 and 4: 1. In yet another embodiment, the molar ratio of ZPT to zinc-phosphonate complex in the concentrate compositions of the present invention is preferably ranging from 1.2: 1 to 3: 1, respectively, more preferably from 1.5 to 2: 1.

METAL-PYRIDINE OXIDE COMPLEX

In another embodiment, the concentrate compositions of the present invention may comprise a metal-pyridine oxide complex, which comprises a pyridine oxide compound that is co-ordinately bound to a metal ion. In an embodiment, the metal is selected from the group consisting of iron, copper and zinc. However, it will be understood by one skilled in the art that other metals can be selected according to the Irving Wiliams Series, which refers to the relative stability of complexes formed by a metal ion.

In a preferred embodiment, the metal is zinc. Such zinc-pyridine oxide complex has a surprising and unexpected effect on the discoloration resistance of the ZPT-containing bar soap compositions, which is demonstrated by a significant increase in its resistance to laboratory- induced discoloration in comparison with control samples containing ZPT only. According to this embodiment, the zinc-pyridine oxide complex acts in synergy with ZPT to improve the antimicrobial effect of the bar soaps made from the concentrate compositions, especially against gram-positive bacteria. It may further provide an extended shelf life for such concentrate compositions and/or bar soaps made from such concentrate compositions.

In a particularly preferred embodiment of the present invention, the pyridine oxide compound has the chemical structure of formula (III):

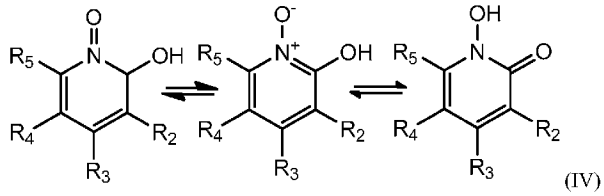


(III)

wherein R 1s R2, R3, R4, and R 5 are each independently selected from the group consisting of H, OH, a halogen (such as F, CI, Br, and I), NO, N0 2, and a

C1-C12

organic group that is linear or branched, saturated or unsaturated, substituted or unsubstituted. More preferably, Ri or R 5 is OH, and R 2, R3, and R4 is each independently selected from the group consisting of H, OH, and a Ci-C 8 alkyl, alkylene, alkyne, or aryl group. It is to be understood that various potential and actual resonate structures of the pyridine oxides may exist (i.e., the bond between the N and O atoms and/or the bond between the neighboring C atom and -OH group may resonate between a single bond and a double bond), for example, according to the chemical structures of formula (IV):

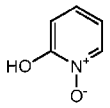


(IV)

It is intended that all of the reasonable resonate structures are meant to be represented by the formula (IV) hereinabove and are thereby included within the scope of the present invention.

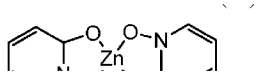
Useful pyridine oxide compounds that can be employed in the practice of the present invention include 2-hydroxypyridine-N-oxide ("HPNO"), N-hydroxy-6-octyloxy-2(1H)-pyridone, ciclopirox olamine, piroctone olamine, and derivatives thereof.

A representative species of pyridine oxide compounds that is particularly useful for the practice of the present invention is 2-hydroxypyridine-N-oxide ("HPNO"), which has the chemical structure of formula (V):



(V)

As a bidentate chelant, HPNO is capable of forming co-ordination complexes with transition metal ions in solution. Specifically, two HNPO can be bound to one zinc ion to form a Zn-HNPO complex with the chemical structure of formula (VI):



Zn-HPNO is a particularly preferred Zn-pyridine oxide compound for the present invention. It is important to note that zinc ions can form various complexes with HPNO, with one, two, three, or even four HPNO attached to one zinc ion, although only the complex with two HPNO attached to one zinc ion as shown by formula (VI) has a neutral charge. In solution, zinc ions and HPNO may undergo speciation to form a mixture of different complex species, and the relative concentration of such complex species can

vary depending on the chemical environment they are in, such as pH and the presence of other metal ions or chelant species. For ease of reference, all such complex species are herein referred to as the "Zn-HPNO complex," regardless of the actual number of HPNO included, and they are all included within the scope of the present invention.

Various derivatives or salts of HPNO with similar chemical structure can also form similar complexes with Zn ions and are therefore also useful for the practice of the present invention. Exemplary HPNO derivatives or salts include, but are not limited to: 6-hydroxy-3- pyridine sulfonic acid, 1 -oxide (CAS 191672-18-1); 2-hydroxy-4-pyridine carboxylic acid, 1- oxide (CAS 13602-64-7); 5-ethoxy-2-pyridinol, 2-acetate, 1-oxide (CAS 51984-49-7); 1-(3- hydroxy-2-oxido-4-isoquinolinyl)-ethanone (CAS 65417-65-4); 6-hydroxy-3 -pyridine carboxylic acid, 1-oxide (CAS 90037-89-1); 2-methoxy-4-quinolinecarbonitrile, 1-oxide (CAS 379722-76- 6); 2-pyridine carboxylic acid, 6-hydroxy-, 1-oxide (CAS 1094194-45-2); 3-pyridine carboxylic acid, 2-hydroxy-, 1-oxide (CAS 408538-43-2); 2-pyridinol, 3-nitro-, 1-oxide (CAS 282102-08-3); 3-pyridine propanenitrile, 2-hydroxy-, 1-oxide (193605-60-6); 3-pyridine ethanol, 2-hydroxy-, 3- acetate, 1-oxide (CAS 193605-56-0); 2-pyridinol, 4-bromo-, 1-oxide (CAS 170875-41-9); 2- pyridinol, 4,6-dibromo-, 2-acetate, 1-oxide (CAS 170875-40-8); 2-pyridinol, 4,6-dibromo, 1- oxide (CAS 170875-38-4); 2-pyridinol, 4-(2-aminoethyl)-, 1-oxide (CAS 154403-93-7); 2-pyridinol, 5-(2-aminoethyl)-, 1-oxide (CAS 154403-92-6); 3-pyridine propanoic acid, a-amino-6- hydroxy-, 1-oxide (CAS 134419-61-7); 2-pyridinol, 3,5-dimethyl, 1-oxide (CAS 102074-62-4); 2-pyridinol, 3-methyl-, 1-oxide (CAS 99969-07-0); 2-pyridinol, 3,5-dinitro, 1-oxide (CAS 98136-47-1); 2-pyridinol, 3,5-dibromo-, 1-oxide (CAS 98136-29-9); 2-pyridinol, 4-methyl-6-(2- methylpropyl)-, 1-oxide (CAS 91408-77-4); 2-pyridinol, 3-bromo-4,6-dimethyl-, 1-oxide (CAS 91408-76-3); 2-pyridinol, 4,5,6-trimethyl-, 1-oxide (CAS 91408-75-2); 2-pyridinol, 6-heptyl-4- methyl-, 1-oxide (CAS 91408-73-0); 2-pyridinol, 6-(cyclohexylmethyl)-4-methyl-, 1-oxide (CAS 91408-72-9); 2-pyridinol, 6-bromo-, 1-oxide (CAS 89284-00-4); 2-pyridinol, 5-bromo-, 1-oxide (CAS 89283-99-8); 2-pyridinol, 3,5-dichloro-4,6-difluoro-, 1-oxide (CAS 33693-37-7); 2- pyridinol, 3,4,5,6-tetrachloro-, 1-oxide (CAS 32835-63-5); 2-pyridinol, 6-methyl-, 1-oxide (CAS 14420-62-3); 2-pyridinol, 5-nitro-, 1-oxide (CAS 14396-03-3); 2-pyridinol, 4-methyl-5-nitro-, 1-oxide (CAS 13602-77-2); 2-pyridinol, 4-chloro-5-nitro-, 1-oxide (CAS 13602-73-8); 2-pyridinol, 4-chloro-, 1-oxide (CAS 13602-65-8); 2-pyridinol, 4-nitro-, 1-oxide (CAS 13602-63-6); and 2- pyridinol, 4-methyl-, 1-oxide (CAS 1952-64-3), and mixtures thereof. These compounds are commercially available from, for example, Sigma-Aldrich (St. Louis, MO) and/or Aces Pharma (Branford, CT).

The amount of zinc-pyridine oxide complex present in the concentrate compositions of the present invention may range from about 0.01% to about 15% by total weight of such compositions. More preferably, such concentrate compositions contains from about 0.05% to about 10% zinc-pyridine oxide complex, still more preferably from about 0.1% to about 7% or from about 0.5% to about 5%, alternatively from 1% to 3%, alternatively combinations thereof, by total weight.

The zinc-pyridine oxide complex as used in the present invention is present in the concentrate compositions as particles, which can be pre-formed by reacting the pyridine oxide compound with a soluble zinc salt, such as $ZnSO_4$, $ZnCl_2$, or a mixture thereof, thereby forming an insoluble precipitate. The term "soluble" as used herein refers to a solubility of at least 0.01 gram per liter in an aqueous solution at 25 °C. The precipitate is then processed into dry powders or used to form a colloidal or slurry composition containing particulates dispersed in a solution, which can be subsequently added into the concentrate compositions.

Alternatively, the particles of zinc-pyridine oxide complex can be formed in situ by directly adding the precursors, i.e., the pyridine oxide compound and the soluble zinc salt, into the concentrate compositions, which will complex with each other in the concentrate compositions to form particles. The pyridine oxide compound and zinc salt can be added either in dry power form or pre-dissolved in a solution.

The particles of zinc-pyridine oxide complex are characterized by an average particle size ranging from about 0.05 micron to about 5,000 microns, preferably from about 0.1 micron to about 2,000 microns, more preferably from about 0.2 micron to about 1,000 microns, and most preferably from about 1 micron to about 600 microns.

The particle size of the zinc-pyridine oxide complex can be readily controlled by modulating the homogenization rate when mixing the soluble zinc salt and the pyridine oxide compound, i.e., the faster the homogenization, the slower the particle growth rate, and consequently the smaller the particles. The particles can further be processed by milling or grinding to achieve a more uniform particle size distribution.

In another embodiment, wherein the chelant is a zinc-pyridine oxide complex and wherein the molar ratio of zinc to the zinc-pyridine oxide complex ranges from 5: 1 to 1 :5. In yet another embodiment, the molar ratio of ZPT to Zn-pyridine oxide complex in the concentrate compositions of the present invention is preferably ranging from about 5: 1 to about 1 : 10, more preferably from about 4: 1 to about 1 :6, still more preferably from about 3: 1 to about 1 :2, and most preferably about 2: 1 to about 1 :1.

PH AND PH ADJUSTING AGENTS

Stability of concentrate compositions is important, and can be impacted by pH. The concentrate compositions are preferably characterized by a pH value ranging from 4 to 11 when dispersed in a 1 wt% aqueous solution. More preferably, the concentrate compositions have a pH range of 4 to 9 or 9 to 11, even more preferably from 9.9 to 10.7, yet even more preferably from 10.1 to 10.6, and most preferably from 10.1 to 10.4.

In an embodiment, the solid concentrate composition of the present has a pH value ranging from 9 to 11 when dispersed in a 1 wt% aqueous solution. Of this embodiment, the composition comprises a C6-C22 fatty acid, C8-C22 fatty alcohol, or mixtures thereof. In another embodiment, the solid concentrate composition of the present has a pH value ranging from 4 to 9 when dispersed in a 1 wt% aqueous solution. Of this embodiment, the composition comprises a Cg-Cis fatty acid, C8-C20 fatty alcohol or mixtures thereof.

This pH range is particularly beneficial for maintaining the dissolution equilibrium of ZPT and the Zn-phosphonate complex or Zn-pyridine oxide complex in the concentrate compositions, and can thereby extend or maximize the shelf life of the concentrate composition. The pH of the concentrate compositions of the present invention can be readily adjusted or modulated by various mechanisms. For example, the pH modulation can be achieved by adjusting the amounts of raw materials used for soap-making, i.e., fats, oils, and base materials such as sodium or potassium hydroxide, so as to reach a final concentrate composition with the desired pH value. For another example, the pH modulation can be achieved using a pH buffering agent, such as potassium carbonate or zinc carbonate. Further, the pH modulation can also be achieved through employment of an acidic pH adjusting agent.

In a preferred, but not necessary, embodiment of the present invention, the pH modulation is achieved by using an acid. Not all acids are suitable for practice of the present invention, and it has been observed that certain acids will aggravate the ZPT discoloration, while other acids help to reduce or alleviate it.

Particularly, it has been discovered that acids having an acid dissociation constant (pKa) of no more than 10 measured at a temperature of 25 °C and an ferric ion-complex stability constant (logKf) of no more than 8 measured at a temperature of 25 °C and an ion strength of 0.1M are particularly effective in reducing or alleviating the ZPT discoloration problem. The term "ferric ion-complex stability constant" as used herein refers to the stability constant of a complex formed between the acid of interest and ferric ions. Preferably, the acids are characterized by a pKa of no more than 8 and a logKf of no more than 6 measured under the same conditions as described hereinabove. More preferably, the acids are characterized by a pKa of no more than 6 and a logKf of no more than 4, as measured under the same conditions as described hereinabove.

Most preferably, acids used for practice of the present invention are selected from the group consisting of sulfuric acid, nitric acid, phosphoric acid, lactic acid, formic acid, acrylic acid, pyruvic acid, malonic acid, glyceric acid, glycine, L-alanine, β-alanine, methylglycine, maleic acid, dihydroxytartaric acid, creatinine, asparagine, N-glycylglycine, butanoic acid, betaine, valine, N-propylglycine, 5-aminopentanoic acid, trimethylacetic acid, pentanoic acid, benzoic acid, C6-C22 fatty acids, and combinations thereof. Fatty acids are particularly preferred acidic pH adjusting agents for the practice of the present invention.

Any fatty acids with total carbon numbers ranging from C_6 to C_{22} can be used for the practice of the present invention. Exemplary fatty acids include, but are not limited to: caproic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, myristoleic acid, palmitoleic acid, sapienic acid, oleic acid, elaidic acid, vaccenic acid, linoleic acid, linoelaidic acid, a-linolenic acid, arachidonic acid, eicosapentaenoic acid, erucic acid, docosahexaenoic acid, and the like. Particularly useful fatty acids for the practice of the present invention are saturated or unsaturated fatty acids with total carbon numbers ranging from C_{12} to C_{22}, such as, for example, lauric acid, myristic acid, palmitic acid, stearic acid, palmitoleic acid, oleic acid, and behenic acid.

In contrast, certain acids, such as hydrochloric acid, citric acid, aspartic acid, picolinic acid, 4-pyridinecarboxylic acid, 3-pyridinecarboxylic acid, tartaric acid, oxalic acid and glutamic acid, have been found to further aggravate the ZPT discoloration problem. It is therefore desirable, although not necessary, to formulate the concentrate compositions of the present invention with as little of these types of acids as possible. Preferably, the concentrate compositions of the present invention are substantially free of hydrochloric acid, citric acid, aspartic acid, picolinic acid, 4-pyridinecarboxylic acid, 3-pyridinecarboxylic acid, tartaric acid, oxalic acid, glutamic acid, or any combination thereof.

REDUCING AGENTS The concentrate compositions of the present invention may optionally comprise one or more reducing agents, which are preferably, but not necessarily, selected from sterically hindered phenols. Such reducing agents can further improve the discoloration resistance of the concentrate compositions, and/or soap bars formed therefrom, as well as extending the shelf life thereof.

Sterically hindered phenolic reducing agents suitable for the use of the present invention are characterized by a molecular weight above 500 Da. Preferred examples include 2,4- dimethyl-6-octyl-phenol; 2,6-di-t-butyl-4-methyl phenol (i.e., butylated hydroxy toluene); 2,6-di- t-butyl-4-ethyl phenol; 2,6-di-t-butyl-4-n-butyl phenol; 2,2'-methylenebis(4-methyl-6-t-butyl phenol); 2,2'-methylenebis(4-ethyl-6-t-butyl phenol); 2,4-dimethyl-6-t-butyl phenol; 4- hydroxymethyl-2, 6-di-t-butyl phenol; n-octadecyl-beta(3, 5-di-t-butyl-4- hydroxyphenyl)propionate; 2,6-dioctadecyl-4-methyl phenol; 2,4,6-trimethyl phenol; 2,4,6- triisopropyl phenol; 2,4,6-tri-t-butyl phenol; 2-t-butyl-4,6-dimethyl phenol; 2,6-methyl-4- didodecyl phenol; tris(3,5-di-t-butyl-4-hydroxy isocyanurate, and tris(2-methyl-4-hydroxy-5-t- buty lpheny l)butane .

®

More preferred are pentaerythrityl tetra-di-t-butyl hydroxyhydrocmnamate (Tinoguard TT, BASF); octadecyl-3,5-di-t-butyl-4-hydroxy-hydrocinnamate (NAUGARD 76, Uniroyal Chemical; IRGANOX 1076, Ciba-Geigy); tetrakis+methylene(3,5-di-t-butyl-4-hydroxy- hydrocinnamate)} methane (NAUGARD 10, Uniroyal Chemical; IRGANOX 1010, Ciba-Geigy); 2,2'-oxamido bis+ethyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)} propionate (NAUGARD XL-1, Uniroyal Chemical); 1,2-bis(3,5-di-t-butyl-4-hydroxyhydrocinnamoyl)hydrazine (IRGANOX MD 1024,Ciba-Geigy); 1,3,5-tris(3,5-di-t-butyl-4-hydroxybenzyl)-s-triazine-2,4,6

(IH,3H,5H)trione (IRGANOX 3114,Ciba-Geigy); 1,3,5-tris(4-t-butyl-3-hydroxy-2,6- dimethylbenzyl)-s-triazine-2,4,6-(IH,3H,5H)trione (CYANOX 1790, American Cyanamid Co.); 1 ,3,5-trimethyl-2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)benzene (ETHANOX 330, Ethyl Corp.); 3,5-di-t-butyl-4-hydroxyhydrocinnamic acid triester with 1,3,5-tris(2-hydroxyethyl)-5-triazine- 2,4,6(1 H,3H,5H)-trione, and bis(3,3-bis(4-hydroxy-3-t-butylphenyl)butanoic acid)glycolester.

Most preferred reducing agents for the practice of the present invention are pentaerythrityl tetra-di-t-butyl hydroxyhydrocmnamate, which is commercially available under the trade name of Tinogard ® TT from BASF (Monheim, Germany).

The amount of reducing agent present in the concentrate compositions of the present invention may range from about 0.001% to about 5% by total weight of such concentrate compositions. More preferably, such concentrate compositions contains from about 0.01% to about 1% of the reducing agent, and most preferably from about 0.02% to about 0.5%>, by total weight of such concentrate compositions.

SOAP SURFACTANTS

The concentrate compositions of the present invention will typically comprise a soap surfactant, or in short "soap", in an amount ranging from about 8%, 10%, 15% or 20% to about 85%o, 80%), 75%o, or 70%>. The term "soap" is used herein in its popular sense, i.e., the alkali metal or alkanol ammonium salts of alkane- or alkene monocarboxylic acids. Sodium, magnesium, potassium, calcium, mono-, di- and tri-ethanol ammonium cations, or combinations thereof are suitable for purposes of the present invention. In general, sodium soaps are used in the concentrate compositions of this invention, but from about 1% to about 25% of the soap may be ammonium, potassium, magnesium, calcium or a mixture of these soaps. The soaps useful herein are the well known alkali metal salts of alkanoic or alkenoic acids having about 12 to 22 carbon atoms, preferably about 12 to about 18 carbon atoms. They may also be described as alkali metal carboxylates of alkyl or alkene hydrocarbons having about 12 to about 22 carbon atoms.

It can be preferred to use soaps having the fatty acid distribution of tallow and vegetable oil (i.e., "fatty acid soaps"). More preferably, the vegetable oil is selected from the group consisting of peanut oil, grapeseed oil, corn oil, olive oil, palm oil, coconut oil, palm kernel oil, palm oil stearine, and hydrogenated rice bran oil, or mixtures thereof, since these are among the more readily available fats. Especially preferred are palm oil stearine, palm kernel oil, and/or coconut oil. The proportion of fatty acids having at least 12 carbon atoms in coconut oil soap is about 85%o. This proportion will be greater when mixtures of coconut oil and fats such as tallow, palm oil, or non-tropical nut oils or fats are used, wherein the principal chain lengths are C_{16} and higher. A preferred soap is sodium soap having a mixture of about 50%> tallow, 30%> palm oil stearine, and 20% palm kernel oil or coconut oil.

Soaps may be made by the classic kettle boiling process or modern continuous soap manufacturing processes wherein natural fats and oils such as tallow or coconut oil or their equivalents are saponified with an alkali metal hydroxide using procedures well known to those skilled in the art. Alternatively, the soaps may be made by neutralizing fatty acids, such as lauric (C_{12}), myristic (C14), palmitic (C_{16}), or stearic (C_{18}) acids with an alkali metal hydroxide or carbonate. SYNTHETIC SURFACTANTS

Synthetic surfactants can be utilized in the present concentrate compositions, either in combination with or in place of the soap surfactants described hereinabove, to further improve the lathering properties of the bar soap during use. When a majority of the surfactants in the concentrate compositions of the present invention are synthetic surfactants rather than soap surfactants, the pH value of the concentrate compositions can be readily broaden to the relatively lower pH range of 7 to 9. In certain embodiments, the pH value of such concentrate compositions may approach the neutral pH range of 6 to 8, which is particularly beneficial because the resulting bar soaps manufactured from the concentrate compositions are more gentle and less irritating to the skin.

The synthetic surfactants useful in this invention include anionic, amphoteric, nonionic, zwitterionic, and cationic surfactants. Synthetic surfactants are typically incorporated in the present concentrate compositions at a level of from about 0.1% to about 90%, preferably from about 0.5%) to about 85%, and more preferably from about 0.75%> to about 50%>, by weight of the concentrate composition.

Examples of anionic surfactants include but are not limited to alkyl sulfates, anionic acyl sarcosinates, methyl acyl taurates, N-acyl glutamates, acyl isethionates, alkyl ether sulfates, alkyl sulfosuccinates, alkyl phosphate esters, ethoxylated alkyl phosphate esters, trideceth sulfates, protein condensates, mixtures of ethoxylated alkyl sulfates and the like. Alkyl chains for these surfactants are C8-C22, preferably Cio-Cig and, more preferably, C_{12}-C_{14} alkyls.

Zwitterionic surfactants can be exemplified by those which can be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds, in which the aliphatic radicals can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and one contains an anionic water- solubilizing group, for example, carboxy, sulfonate, sulfate, phosphate, or phosphonate. Examples include: 4- [N,N-di(2-hydroxyethyl)-N-octadecylammonio] -butane- 1-carboxylate; 5- [S-3-hydroxypropyl-S-hexadecylsulfonio]-3 hydroxypentane-1 -sulfate; 3-[P,P-P-diethyl-P 3,6,9 trioxatetradecyl-phosphonio]-2-hydroxypropane-1 -phosphate; 3-[N,N-dipropyl-N-3-dodecoxy-2- hydroxypropylammonio] -propane- 1 -phosphonate;3 -(N,N-di-methyl-N-hexadecylammonio)propane- 1 -sulfonate; 3 -(N,N-dimethyl-N-hexadecylammonio)-2- hydroxypropane- 1 -sulfonate; 4-(N,N-di(2-hydroxyethyl)-N-(2 hydroxydodecyl)ammonio]- butane- 1-carboxylate; 3-[S-ethyl-S-(3-dodecoxy-2-hydroxypropyl)sulfonio]-propane-1- phosphate, 3-(P,P-dimethyl-P-dodecylphosphonio)-propane-1-phosphonate; and 5-[N,N-di(3- hydroxypropyl)-N-hexadecylammonio]-2-hydroxy-pentane-1 -sulfate.

Examples of amphoteric surfactants which can be used in the concentrate compositions of the present invention are those which can be broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water solubilizing group, e.g., carboxy, sulfonate, sulfate, phosphate, or phosphonate. Examples of compounds falling within this definition are sodium 3- dodecylaminopropionate, sodium 3-dodecylaminopropane sulfonate; N-alkyltaurines, such as the one prepared by reacting dodecylamine with sodium isethionate according to the teaching of U.S. Pat. 2,658,072; N-higher alkyl aspartic acids, such as those produced according to the teaching of U.S. Pat. 2,438,091 ; and the products sold under the trade name "Miranol" and described in U.S. Pat. 2,528,378. Other amphoterics such as betaines are also useful in the present composition. Examples of betaines useful herein include the high alkyl betaines such as coco dimethyl carboxymethyl betaine, lauryl dimethyl carboxy-methyl betaine, lauryl dimethyl alpha- carboxyethyl betaine, cetyl dimethyl carboxymethyl betaine, lauryl bis-(2-hydroxyethyl)carboxy methyl betaine, stearyl bis-(2-hydroxypropyl)carboxymethyl betaine, oleyl dimethyl gamma- carboxypropyl betaine, lauryl bis-(2-hydro-xypropyl)alpha-carboxyet- hyl betaine, etc. The sulfobetaines may be represented by coco dimethyl sulfopropyl betaine, stearyl dimethyl sulfopropyl betaine, amido betaines, amidosulfobetaines, and the like.

Examples of suitable cationic surfactants include stearyldimenthylbenzyl ammonium chloride; dodecyltrimethylammonium chloride; nonylbenzylethyldimethyl ammonium nitrate; tetradecylpyridinium bromide; laurylpyridinium chloride; cetylpyridinium chloride; laurylpyridinium chloride, laurylisoquinolium bromide; ditallow(Hydrogenated)dimethyl ammonium chloride; dilauryldimethyl ammonium chloride; and stearalkonium chloride; and other cationic surfactants known in the art.

Nonionic surfactants useful in this invention can be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydeophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature.

A preferred synthetic surfactant for use in the present compositions is sodium laureth-3 sulfate. Sodium laureth sulfate tends to provide excellent lathering properties, especially when combined with sodium tripolyphosphate as the inorganic salt in the concentrate compositions. In an embodiment, the solid concentrate compositions of the present invention comprises surfactant selected from the group consisting of primary alcohol sulfates, alpha olefin sulfonates, acyl isethionates and mixtures thereof.

OTHER INGREDIENTS

During the soap making process, the concentrate compositions can be mixed with additional ingredients such as, for example, inorganic salts (especially inorganic zinc salts, such as zinc carbonate, zinc sulfate, zinc nitrate, zinc fluoride, zinc chloride, zinc borate, and the like as well as zinc oxide). A particularly preferred inorganic salt is zinc carbonate. In a particularly preferred embodiment of the present invention, the concentrate compositions can be used to form bar soaps containing zinc carbonate at an amount ranging from about 0.01% to about 5%, more preferably from about 0.1% to about 3%, and most preferably from about 1% to about 2% by total weight of the composition. Zinc carbonate provided at such an amount is particularly effective in reducing or removing malodor.

The concentrate compositions of the present invention may further be mixed with one or more optional ingredients selected from the group consisting of: structurants, such as raw starch, pregelatinzed starch, carboxymethyl cellulose, polyacrylate polymer, Carbopol, carregeenan, xanthan gum, polyethylene glycol, polyethylene oxide, and the like; free fatty acids, such as those derived from tallow, coconut, palm and palm kernel; humectants; cationic polymers, such as cationic polysaccharides, cationic polyalkylene imines, cationic hydroxyethyl cellulose, and the like; brighteners; fillers, such as silica, talc, and the like; perfumes; sequestering agents; coloring agents, opacifiers and pearlizers, such as titanium dioxide.

All of these are useful in enhancing the appearance, smell or other cosmetic/sensory properties of the products made from the concentrate compositions. For example, with bar soaps, the appearance can be transparent, translucent, or opaque, and the color thereof can be white, off- white, cream, yellow, pink, red, green, purple, blue and black. In one embodiment, the bar soaps is opaque with a white or off-white color.

PREPARATION METHODS

Concentrate compositions of the present invention can be made via a number of different processes. In an aspect, the present invention is directed to a method of manufacturing a solid concentrate composition, comprising the steps of: (a) forming a mixture that comprises from 5% to 25% by weight of a zinc pyrithione (ZPT), and from 8% to 85% by weight of at least one surfactant by total weight of the mixture; and (b) shaping the mixture to form a solid concentrate composition. In an embodiment of this method, wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as disclosed herein.

Preferably, the concentrate compositions of the present invention can be made by the following process. First, all the ingredients, such as the surfactant, ZPT (either as solid powder material or an aqueous dispersion), and fillers, are added into an amalgamator and blended for approx. 5 to 10 mins to form coated pellets. Then the coated pellets from the amalgamator are refined by high shearing force, either in the refining plodder or in the roll mill, to achive good homogeneity. The resultant mixture from then is extruded in a plodder and shaped into pellets by the pore plate at the plodder outlet.

Bar soaps made from the concentrate composition, as described above, can be made via a number of different processes. In another aspect, the present invention is directed to a method of forming a bar soap, comprising the steps of: (a) forming a mixture by diluting a solid concentrate composition that comprises from 5% to 25% by weight of a zinc pyrithione (ZPT), and from 8% to 85% by weight of at least one surfactant by total weight of the mixture; and (b) shaping the mixture to form a solid concentrate composition. In an embodiment of this method, wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as disclosed herein.

Preferably, the present compositions are made via a process that involves milling, resulting in milled bar soap compositions. A typical milling process of manufacturing a bar soap composition includes: *(a)* a step in which the soap is made through either a continuous process (ConSap or continuous saponification process) or a batch-making process (i.e. neutralization process for hydrolysis fatty acid noodle or kettle process), (b) a vacuum drying step in which the soap is made into soap noodles, (c) an amalgamating step in which the soap noodles are combined with other ingredients of the bar soap composition, (d) a milling step in which a relatively homogeneous mixture is obtained, (e) a plodding step in which the soap mixture is extruded as soap logs and then cut into soap plugs, and (f) a stamping step in which the soap plugs are stamped to yield the finished bar soap composition. The present bar soap can be made using any of the above mentioned manufacturing processes, and the ZPT, the metal-phosphonate complex (or the precursors for in situ forming such complex), and pH adjusting agent, and the reducing agent can be added during the mixing steps of preparing the bar soaps.

Other product forms of the present invention, such as body washes, shower gels, liquid hand soaps, shampoos, facial cleansers, and the like, can be readily formed starting from the concentrate compositions using the conventional mixing or homogenization process known to those skilled in the art.

TEST METHODS ZPT STABILITY

As mentioned hereinabove, zinc pyrithione (ZPT) may undergo transformation upon exposure to oxidizing species, thereby losing its anti-microbial effect over time in environments susceptible to oxidation. Such vulnerability of ZPT to environmental assaults is well known in the art, and various solutions have been proposed to stabilize ZPT with limited success.

It is a surprising and unexpected discovery of the present invention that the above- described metal-phosphonate complex and/or metal-pyridine complex are effective in stabilizing ZPT in concentrate compositions (and bar soaps formed therefrom) to reduce ZPT loss even in harsh chemical environments.

The chemical stability of ZPT is evaluated by an aging test described as follows, so as to determine the percentage loss of ZPT after such aging test. First, a bar soap or concentrate composition containing ZPT is obtained, preferably immediately after it is manufactured. The starting content of ZPT in such bar soap/ concentrate composition (in percentage) is measured by method described hereinafter using a portion of the bar soap/ concentrate composition, or a companion bar/ composition made from the same batch of soap noodle. The bar soap/ concentrate composition is weighed (+/-0.01 g), and its starting weight is recorded. Second, the bar soap/ concentrate composition is subjected to an aging process, during which the bar soap/ concentrate composition is placed inside a sealed water impermeable bag, which is preferably made of polyethylene (PE). The bag containing the bar soap is then left either at room temperature (i.e., about 25 °C), or in a convection oven at an elevated temperature (e.g., 40 °C), for an extended period (e.g., 10 days, 12 days, 14 days, or up to 36 months in certain cases). After the aging, if placed in a convection oven at the elevated temperature, the bar soap/ concentrate composition is taken out of the convection oven and allowed to return to room temperature (i.e., 25 °C). The bar soap/ concentrate composition is weighed again, and its final weight is recorded. The final content of zinc pyrithione in the bar soap/ concentrate composition (in percentage) is measured by the same method as described hereinafter.

Chemical stability of the ZPT is calculated by the following equation to obtain the percentage loss of ZPT:

Final Weighty-Final ZPT Content (%)

% Loss of ZPT] X 100%,

Starting Weighty-Starting ZPT Content (%) The content of ZPT in bar soap/ concentrate compositions is measured herein by an iodine-based titration method, which is described in greater detail in the following sections. The mercapto group in ZPT can be titrated by iodine, which oxidizes it to the disulfide-2,2' dithiobispyridine-l-oxide. If ZPT has already been oxidized or undergone transformation otherwise so that it no longer possesses the mercapto group, it will not be detectible by the iodine-based titration method described hereinafter.

First, a standardized 0.04N iodine solution is prepared. Specifically, anhydrous sodium thiosulphate (with a minimum purity of 99%) is oven-dried for 2 hours at 105 °C and then stored in a dessicator. 0.05 grams (+/-0.0001 g) of the anhydrous sodium thiosulfate is weighed and placed into the 100ml polypropylene beaker of an autotitrator, and 50 ml of deionized water is added to form a standard solution. The autotitrator used herein is preferably a Mettler DL25 or Mettler DM140-SC titrator with platinum ring electrode, which is commercially available from Mettler Toledo International, Inc. (Switzerland), or an equivalent thereof. The autitrator is set up to titrate the standard sodium thiosulfate solution with the iodine solution that is being standardized. Bubbles are eliminated from the burette of the autotitrator, and titration is commenced. Such procedure is repeated twice more, and the results are averaged to obtain a standardized 0.04N iodine solution. The % relative standard deviation (RSD) should be less than 1% of the average.

Next, standardized 0.01 N and 0.006 N iodine solutions are prepared. Specifically, standardized 0.01 N iodine solution is prepared using 0.1 Og (+/-0.0001 g) sodium thiosulphate dissolved in 100 mL deionized water, using 10.0 mL pipetted into the 100 mL autotitrator breaker with 50 mL additional deionized water followed by the titration procedure. Standardized 0.006 N iodine solution is prepared using 3.0 mL of a 0.01 M sodium thiosulphate solution and 40 mL of a solvent (containing 13% v/v hydrochloric acid in 6%> v/v butanol), followed by addition of 40 mL of 1 : 1 hexane/isopropanol. The autotitration procedure is subsequently carried out. The iodine solutions are standardized daily.

The bar soap/ concentrate composition whose ZPT content is to be measured is then shredded using a grater and stirred to form a homogenous mixture. 4.00 grams of the shredded soap is weighed and put into a clean, dry beaker of an autotitrator. 75 mL of hot 6% v/v butanol (which was heated in a boiling-water bath) and 5 mL of concentrated HCl (provided at room temperature) are then added into the beaker. The mixture is agitated vigorously so as to fully dissolve all soluble components. The beaker is subsequently placed in the autotitrator, and bubbles are completely eliminated from the burette. The titration is then initiated and analyzed while the mixture is still warm. The mixture is vigorously agitated during the titration procedure. For compositions with less than 0.2% of ZPT by weight, titration is carried out using the 0.006 N iodine solution. For compositions with higher ZPT concentrations, the initial starting sample weight can be reduced. Titration can be done either manually or by using autotitration procedure by those with skill in the art.

The ZPT content in the bar soap/ concentrate composition is calculated as follows:

V olume o f Io dine S olution (ml)xN X15.88%

ZPT Content (%) = ,

Sample Weight (g)

wherein N is the normality of the standardized iodine solution, and wherein 15.88% is a constant that is derived from:

r n on / Molecular Weight of ZPT X 100% 317.6 X 100% lb.oo% = = ,

Number of Pyrithwne per Molecule X 1000 ml/Liter 2 X 1000 ml/Liter

The above-described procedure is repeated three times for each bar soap composition whose ZPT content is to be measured, and the results are averaged to obtain a final ZPT content in percentage (%) for the specific bar soap/ concentrate composition. All chemical reagents employed hereinabove are high-purity reagents obtained from VWR Scientific (Batavia, Illinois, USA) or other scientific chemical suppliers.

WATER ACTIVITY

Water Activity ("Aw") is a measurement of the energy status of the water in a system. It indicates how tightly water is bound, structurally or chemically, within a composition. Water activity ("Aw") is defined as the ratio of the water vapor pressure over a sample (P) to that over pure water (P o):

$$A_W = \frac{P}{P_0}$$

Water activity of a concentrate composition or a bar soap made from the concentrate composition can be measured electronically using a water activity meter with a sealed chamber and an electrical or optical measurement of the headspace. The meter is calibrated against a series of saturated salt solutions. A bar soap/ concentrate composition to be measured is placed in the chamber held at ambient temperature which is then allowed to equilibrate with the headspace in the chamber. At equilibrium, the relative humidity of the air in the chamber is the same as the water activity of the composition.

For purposes of the present invention, the water activity (Aw) of a bar soap composition can be measured using a Hygrolab 3 Water Activity Meter available from Rotronic, Inc. (Buntington, NY, USA). The following procedure is employed to determine the water activity (Aw) of a bar soap composition:

1. Check the chamber of the meter to make sure it is clean and dry before the test;

2. Cut a bar soap/ concentrate composition into pieces of about 0.2-0.4 cm thick with a stainless steel knife;

3. Put the soap pieces into a clean, dry plastic sample container with a depth of 1/2";

4. Press the soap pieces with a gloved finger lightly to make sure that the bottom of the container is covered by the soap pieces;

5. Put the sample container back into the chamber of the meter and cover it with the chamber top, which contains the electronic headspace measurement apparatus;

6. Wait for the headspace to reach equilibrium (approximately 1-2 hours); and

7. Record the temperature and the Aw value.

Preferably, but not necessarily, the bar soap/ concentrate compositions of the present invention are characterized by a water activity of less than 0.9, more preferably between about 0.4 and 0.9, still more preferably between 0.5 and 0.9, and most preferably between 0.6 and 0.9. The bar soap can be manufactured with a water activity of about 0.85, and during distribution, such bar soap can dehydrate to obtain a lower water activity of between 0.5 and 0.8, or between 0.55 and 0.75, or between 0.6 and 0.75. PENETRATION HARDNESS

The penetration hardness test provides a method to assess the stickiness of the concentrate composition by measuring the force (N) required to break a cake formed by compressing the concentrate particles. The stickier the concentrate particles, the greater the force (N) is required to break the cake. The force (N) required to break the cake is measured at 25 °C, 15% relative humidity, using a Force Gauge Machine (Model Z2-44) as depicted in FIG. 2D, and available from IMADA Inc. (Illinois, USA). The following procedure is employed to determine the penetration hardness of the concentrate compositions:

1. Prepare the solid concentrate composition containing ZPT of the present invention according to the method as disclosed herein above. This concentrate composition can be any solid forms such as, for example, powders, granules, pellets, noodles, chunks, bars and the like.

2. Prepare the cake form by compressing the solid concentrate particles prepared in step 1 in a sleeve-confined space, with reference to FIGs. 2A-2C, according to the following steps: (i) Place a solid perspex cylinder with polished surface with a diameter of 6.35 cm, length of 15.90 cm, and having a hole with a diameter of 0.65 cm through the center of the cylinder and positioned about 9.20 cm from the bottom of the cylinder, as shown in FIG. 2A, under a force gauge machine. Place a perspex sleeve with polished inner surface with the inner diameter of 6.40 cm, length of 15.90 cm, along the outside of the cylinder. Ensure that the cylinder and sleeve are clean and the sleeve moves freely along the cylinder.

(ii) Place a pin (i.e., locking pin) into a hole in the cylinder and rest the sleeve thereon to form a confined space on the top of the cylinder having a volume of 291.2 cm^3.

(iii) Grind the concentrate composition into small concentrate particles with particle size smaller than 1 cm.

(iv) Fill the confined space on the top of the cylinder with the concentrate particles as shown in FIG. 2A. With a straightedge, level the concentrate particles with the top of the sleeve.

(v) Place a lid on top of the sleeve, and then carefully place a 5 Kg weight on top of the lid.

(vi) Remove the locking pin as shown in FIG. 2B.

(vii) After 5 mins, remove the 5 Kg weight and slide the sleeve down, as shown in FIG. 2C, to form the concentrate cake.

3. Measure the force (N) required for a probe to break the concentrate cake using the Force Gauge Machine, with reference to FIG. 2D, and record the peak force (N) value.

(i) Set the force gauge to 0 N and turn on the machine. Switch on the motor to direct the gauge downward to the center of the lid.

(ii) Record the peak force (N) required to break the cake, which is equal to the cake strength or the concentrate composition stickiness.

Note. The Force Gauge Machine and the method as described hereinabove are calibrated according to known standards to those skilled in the art and that are commercially available.

EXAMPLES

Example 1 - Comparative ZPT Stability Test of Concentrate Compositions

Seven different concentrate compositions (Examples A-G) are prepared containing soap noodles with varying levels of ZPT, uncomplexed HEDP, and talc as listed in Table 2 below. Table 2

I .v A I .v B I .v C I .v I) I .v I . I .v I I A . (!

Dry Soap Noodle* 35.62 19.17 40.87 60.10 20.38 48.66 37.34

ZPT (48% active) 15.15 22.26 18.10 15.47 18.40 12.05 16.72

HEDP-4Na 5.19 7.63 6.20 5.30 6.31 2.41 3.34

ZnS04-7H20** 4.04 5.94 4.83 4.13 4.91 1.88 2.60

Talc 40.00 45.00 30.00 15.00 50.00 35.00 40.00

Total 100.00 100.00 100.00 100.00 100.00 100.00 100.00 pH (1% solution) 10.09 9.83 10.14 10.20 9.86 10.25 10.21

*The soap noodle contained the ingredients in Table 3.

Analytical grade available from Tianjin Jiaxin Chemicals Glass Instrument Trading Co., Ltd.

Table 3

I ii Liivd ien i s W I ' r

Sodium palmate (from palm oil and palm oil 49.683

sterine)

Sodium tallowate (from tallow) 16.027

Sodium palm kernelate (from palm kernel oil) 14.424

Unsaponifiable matter 0.540

Citric acid (anhydrous) 0.100

Sodium citrate 0.152

Pentasodium pentetate 0.050

Tetrasodium etidronate 0.050

Sodium chloride (low sodium) 0.553

Glycerine 3.471

Coconut acid 0.950

DI Water Q.S. The initial ZPT contents of Examples A-G are measured according to the ZPT stability test procedures described hereinabove. The samples are then subjected to environment stresses in an incubator at 40 °C with 60% relative humidity (RH) for 90 days, after which the final ZPT contents were re-measured and used to calculate the ZPT loss (w/w%>). The results are summarized in Table 4.

Table 4

Results	Ex. A	Ex. B	Ex. C	Ex. D	Ex. E	Ex. F	Ex. G
Initial ZPT Content (w/w %)	7.272	10.684	8.686	7.426	8.834	5.785	8.026
Final ZPT Content (w/w %)	7.266	10.611	8.681	7.394	8.917	5.768	7.877
ZPT Loss (w/w%)	0.006	0.073	0.005	0.032	(0.083)	0.017	0.149

The ZPT loss (w/w%) from the above Examples are plotted in Fig. 1, which demonstrates that there is no significant ZPT loss for concentrate composition at varying ZPT levels. Example 2 - Comparative ZPT Stability Test of Bar Soaps

Two different bar soaps Examples H and I are prepared from the concentrate composition and other listed ingredients in Table 5.

Table 5.

Raw Materials	Amount (w/w %)	
	Example H	Example I
Dry Soap Noodle*	75.97	75.67
ZPT concentrate (10%)	2.40	2.40
Starch	20.00	20.00
Perfume	1.10	1.40
Tinogard TT**	0.03	0.03
Dye	0.01	0.01
DI water	Q.S.	Q.S.
pH (1% solution)	10.26	10.19

Raw Materials	Amount (w/w %)	
	Example H	Example I
Dry Soap Noodle*	75.97	75.67
ZPT concentrate (10%)	2.40	2.40
Starch	20.00	20.00
Perfume	1.10	1.40
Tinogard TT**	0.03	0.03
Dye	0.01	0.01
DI water	Q.S.	Q.S.
pH (1% solution)	10.26	10.19

*The soap noodle contained the ingredients in Table 3.

im, Germany).

The initial ZPT contents of Examples H and I are measured according to the ZPT stability test procedures described hereinabove. The samples are then subjected to environment stresses in an incubator at 40 °C with 60% relative humidity (RH) for 90 days, after which the final ZPT contents were re-measured and used to calculate the ZPT loss (w/w%). The results are summarized in Table 6 and demonstrate no significant ZPT loss for bar soaps made from concentrate compositions.

Table 6.

Results	Example H	Example I
Initial ZPT Content (w/w %)	0.240	0.240
Final ZPT Content (w/w %)	0.224	0.221
ZPT Loss (w/w%)	0.016	0.019

Example 3 - Penetration Hardness Test of the Concentrate Compositions

Seven different concentrate compositions (Examples J-P) are prepared containing soap noodles with varying levels of ZPT and talc as listed in Table 7 below. The moisture was measured by a Karl Fischer Auto Titrator with the following procedures: 1. Cut sample into small pieces and then weigh out 0.200-0.300 g samples with weight paper.

2. Zero the balance.

3. Add the weighted samples to the titrator vessel careful to avoid spillage. Re-seal the vessel and return the weight paper onto the balance.

4. Record the consumed weight (expressed as the difference on the balance).

5. Enter the consumed weight into the Auto Titrator.

6. Press "ok" to confirm the sample weight. Titration will be performed and data will be printed automatically once the experiment completes.

Table 7

Raw Materials	Amount (w/w %)						
	Ex. J	Ex. K	Ex. L	Ex. M	Ex. N	Ex. O	Ex. P
Dry Soap Noodle*	4.00	8.60	39.60	59.60	77.10	70.40	69.60
ZPT (48% active)	8.00	10.40	10.40	10.40	10.40	10.40	10.40
Talc	88.00	81.00	50.00	30.00	10.00	10.00	5.00
DI Water	0.00	0.00	0.00	0.00	2.5	9.00	15.00
Moisture	3.38%	5.10%	8.54%	10.33%	14.26%	19.60%	27.03%

The force (N) required to break the cakes (Examples J-P) are measured according to the Penetration Hardness test procedures described hereinabove. The results are summarized below in Table 8.

Table 8

Results	Ex. J	Ex. K	Ex. L	Ex. M	Ex. N	Ex. O	Ex. P
Penetration Force (N)	16.83	20.63	30.80	26.78	25.83	42.16	89.83

See Fig. 3A-D, wherein in Fig. 3A depicts concentrate composition with a penetration hardness of < 20 N, Fig. 3B depicts concentrate composition with a penetration hardness of ~ 20 N, Fig. 3C depicts concentrate composition with a penetration hardness of between 20 N to 50 N, and Fig. 3D depicts concentrate composition with a penetration hardness of > 50 N. Therefore, it is preferred that the concentrate compositions of the present invention are characterized by a penetration hardness of 20 N to 50 N, to provide for compositions with the right degree of stickiness.

All ratios are weight ratios unless specifically stated otherwise. All temperatures are in

Celsius degrees (°C), unless specifically stated otherwise. All dimensions and values disclosed herein (e.g., quantities, percentages, portions, and proportions) are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension or value is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40mm" is intended to mean "about 40 mm."

Every document cited herein, including any cross referenced or related patent or application, is hereby incorporated herein by reference in its entirety unless expressly excluded or otherwise limited. The citation of any document is not an admission that it is prior art with respect to any invention disclosed or claimed herein or that it alone, or in any combination with any other reference or references, teaches, suggests or discloses any such invention. Further, to the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

Claims

Hide Dependent

CLAIMS What is claimed is:

1. A solid concentrate composition comprising:

(a) from 5% to 25% by weight of a zinc pyrithione (ZPT); and

(b) from 8% to 85% by weight of at least one surfactant.

2. The solid concentrate composition according to claim 1 , wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as disclosed herein.

3. The solid concentrate composition according to claim 1 , comprising from 6% to 8% by weight of the ZPT.

4. The solid concentrate composition according to claim 1 , further comprising (c) from 1.4% to 15% by weight of a chelant, wherein the chelant has a log K $_{ZnL}$ of greater than 2, wherein the log K-znL is the log of a conditional stability constant of the chelant with Zn calculated at pH 7, 25 °C, 0.1 M NaCl.

5. The solid concentrate composition according to claim 1 , further comprising (c) from 1.4% to 15% by weight of a chelant and wherein the chelant is a metal phosphonate complex comprising one or more phosphonate chelants co-ordinately bonded to one or more metal ions.

6. The solid concentrate composition according to claim 1 , further comprising (c) from 1.4% to 15% by weight of a chelant and wherein the chelant is a metal-pyridine oxide complex that is co-ordinately bonded to a metal ion.

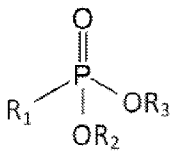
7. The solid concentrate composition according to claim 5 or 6, wherein the metal is selected from the group consisting of iron, copper, and zinc.

8. The solid concentrate composition according to claim 7, wherein the metal is zinc.

9. The solid concentrate composition according to claim 8, wherein the phosphonate chelant comprises a zinc-phosphonate complex and wherein the molar ratio of zinc to the phosphonate chelant ranges from 2: 1 and 4: 1.

10. The solid concentrate composition according to claim 8, wherein the chelant is a zinc- pyridine oxide complex and wherein the molar ratio of zinc to the zinc-pyridine oxide complex ranges from 5: 1 to 1 :5.

11. The solid concentrate composition according to claim 5, wherein the one or more phosphonate chelants each comprises one or more functional groups of the formula (I):



(I)

wherein Ri is a linear, branched or cyclic, saturated or unsaturated, substituted or unsubstituted C $_{1}$-C$_{20}$ hydrocarbon group, and wherein R $_{2}$ and R3 are independently selected from the group consisting of hydrogen and Ri.

12. The solid concentrate composition according to claim 11 , wherein the one or more phosphonate chelants are selected from the group consisting of 2-aminoethyl phosphoric acid (AEP), N-phosphonomethyl aminodiacetic acid (PMIDA), 1 -hydroxy ethane- 1,1- diphosphonic acid (HEDP), amino tris(methylene phosphonic acid) (ATMP), ethylenediamine tetra(methylene phosphonic acid) (EDTMP), diethylenetriamine penta(methylene phosphonic acid) (DTPMP), phytic acid, nitrilotrimethylene phosphonic acid (NTP), and combinations thereof.

13. The solid concentrate composition according to claim 12, wherein the one or more phosphonate chelants comprise HEDP.

14. The solid concentrate composition according to claim 1, having a pH value ranging from 9 to 11 when dispersed in a 1 wt% aqueous solution.

15. The solid concentrate composition of claim 14, wherein the composition comprises a C6-C $_{22}$ fatty acid, C8-C $_{22}$ fatty alcohol, or mixtures thereof.

16. The solid concentrate composition according to claim 1, having a pH value ranging from 4 to

9 when dispersed in a 1 wt% aqueous solution.

17. The concentrate composition according to claim 16, wherein the composition further comprises a C_{8}-C_{18} fatty acid, C_{8}-C_{20} fatty alcohol or mixtures thereof.

18. The solid concentrate composition according to claim 1, wherein the surfactant is selected from the group consisting of primary alcohol sulfates, alpha olefin sulfonates, acyl isethionates and mixtures thereof.

19. The solid concentrate composition of claim 1, wherein the ZPT has a particle size of less than

10 microns.

20. A method for manufacturing a solid concentrate composition, comprising the steps of:

(a) forming a mixture that comprises from 5% to 25% by weight of a zinc pyrithione (ZPT), and from 8% to 85% by weight of at least one surfactant by total weight of the mixture; and

(b) shaping the mixture to form a solid concentrate composition.

21. The method according to claim 20, wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as disclosed herein.

22. A method of forming a bar soap, comprising the steps of:

(a) forming a mixture by diluting a solid concentrate composition that comprises from 5% to 25% by weight of a zinc pyrithione (ZPT) and from 8% to 85% by weight of at least one surfactant by total weight of the mixture; and

(b) shaping the mixture to form a bar soap.

23. The method according to claim 22, wherein the solid concentrate composition has a penetration hardness of between 20 N and 50 N according to the test method as disclosed herein.

Patent Citations (5)

Publication number	Priority date	Publication date	Assignee	Title
WO2006058708A1 *	2004-11-22	2006-06-06	Unilever Plc	Improved detergent composition
CN101589786A *	2006-12-15	2009-11-25	宝洁公司	Composition comprising pyrithione or a polyvalent metal salt of a pyrithione and furametpyr
US20130045260A1 *	2011-08-15	2013-02-21	Edward Dewey Smith, III	Personal Care Compositions Having Dried Zinc Pyrithione-Polymer Aggregates
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WO2013166718A1 *	2012-05-11	2013-11-14	The Procter & Gamble Company	Personal cleansing compositions comprising zinc pyrithione
Family To Family Citations				

* Cited by examiner, † Cited by third party

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CA2098429A1	1994-10-01	Preserved wet wipes
EP0858321B1	2004-01-14	Personal cleansing composition
CZ286401B6	2000-04-12	Use of inorganic redox-active substances
US4379753A	1983-04-12	Hair care compositions
US4089945A	1978-05-16	Antidandruff shampoos containing metallic cation complex to reduce in-use sulfide odor
CN101827575B	2014-03-05	Stable liquid cleansing compositions containing fatty acyl isethionate surfactant
EP0094386B1	1984-09-12	Zinc-pyridinethione salts, a process for making thereof and hair care compositions containing them
CA2360983A1	2000-09-08	Chemical method for making pyrithione particle dispersions
CN101170908	2011-09-14	Structured surfactant system
US3753930A	1973-08-21	Phenylbismuth bis(2-pyridinethiol 1-oxide)
JPH10504832A	1998-05-06	Surfactant composition
US6805874B1	2004-10-19	Method and skin cleansing compositions for dermatological basic treatment

EP0903136A1	2002-06-12	Liquid compositions comprising hydrophobically modified polyalkylene glycols as mildness actives

Priority And Related Applications

Priority Applications (4)

Application	Priority date	Filing date	Title
PCT/CN2013/072648	2013-03-14	2013-03-14	Bar soap compositions containing zinc pyrithione and a zinc-pyridine oxide complex
CNPCT/CN2013/072648		2013-03-14	
PCT/CN2013/074366	2013-04-18	2013-04-18	Personal care compositions containing zinc pyrithione and zinc-phosphonate complex
CNPCT/CN2013/074366		2013-04-18	

Applications Claiming Priority (7)

Application	Filing date	Title
CA2904420A	2014-03-13	Solid concentrate compositions containing zinc pyrithione
CN201480008186.0A	2014-03-13	Solid concentrate compositions containing zinc pyrithione
BR112015023254A	2014-03-13	Solid concentrated compositions containing zinc pyrithione
EP14763357.2A	2014-03-13	Solid concentrate compositions containing zinc pyrithione
US14/653,469	2014-03-13	Solid concentrate compositions containing zinc pyrithione
MX2015012191A	2014-03-13	Solid concentrate compositions containing zinc pyrithione.
PH12015501873A	2015-08-25	Solid concentrate compositions containing zinc pyrithione

Legal Events

Date	Code	Title	Description
2014-11-05	121	Ep: the epo has been informed by wipo that ep was designated in this application	**Ref document number**: 14763357 **Country of ref document**: EP **Kind code of ref document**: A1
2015-08-13	REEP		**Ref document number**: 2014763357

			Country of ref document: EP
2015-08-13	WWE	Wipo information: entry into national phase	**Ref document number**: 2014763357 **Country of ref document**: EP
2015-09-08	ENP	Entry into the national phase in:	**Ref document number**: 2904420 **Country of ref document**: CA
2015-09-10	WWE	Wipo information: entry into national phase	**Ref document number**: MX/A/2015/012191 **Country of ref document**: MX
2015-09-14	NENP	Non-entry into the national phase in:	**Ref country code**: DE
2015-09-14	WWE	Wipo information: entry into national phase	**Ref document number**: 14853489 **Country of ref document**: US
2015-12-08	REG	Reference to national code	**Ref country code**: BR **Ref legal event code**: B01A **Ref document number**: 112015023254 **Country of ref document**: BR
2017-07-18	ENP	Entry into the national phase in:	**Ref document number**: 112015023254 **Country of ref document**: BR **Kind code of ref document**: A2 **Effective date**: 20150914

Data provided by IFI CLAIMS Patent Services

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PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To:
Krebs, Jay A
THE PROCTER & GAMBLE COMPANY
Global IP Services
One Procter & Gamble Plaza, C9
Cincinnati OH 45202
ETATS-UNIS D'AMERIQUE

Sun only

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT AND THE WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44.1)

Applicant's or agent's file reference 14861M-GL	Date of mailing *(day/month/year)* 25 March 2019 (25-03-2019) **FOR FURTHER ACTION** See paragraphs 1 and 4 below
International application No. PCT/US2018/039972	International filing date *(day/month/year)* 28 June 2018 (28-06-2018)
Applicant THE PROCTER & GAMBLE COMPANY	

1. [X] The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.

   Filing of amendments and statement under Article 19:
   The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

   When? The time limit for filing such amendments is normally two months from the date of transmittal of the International Search Report.

   How? Directly to the International Bureau of WIPO, 34 chemin des Colombettes 1211 Geneva 20, Switzerland, Fascimile No.: (41-22) 338.82.70

   For more detailed instructions, see *PCT Applicant's Guide*, International Phase, paragraphs 9.004 - 9.011.

2. [ ] The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.

3. [ ] **With regard to any protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

   [ ] the protest together with the decision thereon has been transmitted to the International Bureau together with any applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

   [ ] no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Reminders**

   The applicant may **submit comments on an informal basis on the written opinion of the International Searching Authority** to the International Bureau. These comments will be made available to the public after international publication. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established.

   Shortly after the expiration of **18 months from the priority date, the international application will be published** by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau before the completion of the technical preparations for international publication (Rules 90*bis*.1 and 90*bis*.3).

   Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase **until 30 months** from the priority date (in some Offices even later); otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for **entry into the national phase** before those designated Offices. In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19 months. For details about the applicable time limits, Office by Office, see www.wipo.int/pct/en/texts/time_limits.html and the *PCT Applicant's Guide*, National Chapters.

   Within **22 months from the priority date, the applicant may request that a supplementary international search be carried out** by a different International Searching Authority that offers this service (Rule 45*bis*.1). The procedure for requesting supplementary international search is described in the *PCT Applicant's Guide*, International Phase, paragraphs 8.006-8.032.

Name and mailing address of the International Searching Authority European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040 Fax: (+31-70) 340-3016	Authorized officer CHRISTENSEN, Jette Tel: +49 (0)89 2399-8052

Form PCT/ISA/220 (July 2017)

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	FOR FURTHER ACTION	see Form PCT/ISA/220 as well as, where applicable, item 5 below.
14861M-GL		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/US2018/039972	28 June 2018 (28-06-2018)	30 June 2017 (30-06-2017)

Applicant

THE PROCTER & GAMBLE COMPANY

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 8 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

a. With regard to the **language**, the international search was carried out on the basis of:

[X] the international application in the language in which it was filed

[ ] a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))

b. [ ] This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6*bis*(a)).

c. [ ] With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. [ ] **Certain claims were found unsearchable** (See Box No. II)

3. [X] **Unity of invention is lacking** (see Box No III)

4. With regard to the **title**,

[X] the text is approved as submitted by the applicant

[ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

[X] the text is approved as submitted by the applicant

[ ] the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6. With regard to the **drawings**,

a. the figure of the **drawings** to be published with the abstract is Figure No. 1

[X] as suggested by the applicant

[ ] as selected by this Authority, because the applicant failed to suggest a figure

[ ] as selected by this Authority, because this figure better characterizes the invention

b. [ ] none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2018/039972

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☒ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2018/039972

A. CLASSIFICATION OF SUBJECT MATTER
INV. A61K8/44 A61K8/55 A61K8/42 A61K8/49 A61K8/41
A61K8/34 A61K8/19 A61K8/02 A61Q15/00 A61K8/33
A61K8/368 A61K8/67 A61K8/81 A61K8/92 A61K8/96

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)
A61K A61Q

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 906 454 A (MELANSON JR JOHN D [US] ET AL) 6 March 1990 (1990-03-06)	1,5,7,9
Y	claims 1,2,6-8	8,13-15
A	examples 1-6	2-4,6, 10-12

X	WO 2017/106279 A1 (PROCTER & GAMBLE [US]) 22 June 2017 (2017-06-22) examples compositions U and V; table 4C	1,5,9

X	US 2016/326091 A1 (RUDOLPH THOMAS [DE] ET AL) 10 November 2016 (2016-11-10) Variant d; example F example *	1,5,9

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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* 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
11 March 2019	25/03/2019

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Grenouillat, N

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Form PCT/ISA/210 (second sheet) (April 2005)

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2017/172873 A1 (BANOWSKI BERNHARD [DE] ET AL) 22 June 2017 (2017-06-22)	1,5,8,9
Y	claim 1 paragraphs [0129], [0132], [0081],	2-4,6,7, 10-14
A	[0137]	15
Y	JP 2008 110999 A (MANDOM CORP) 15 May 2008 (2008-05-15) abstract paragraphs [0017], [0026], [0027], [0029], [0060]	1-15
Y	US 2014/154189 A1 (POLSON GEORGE [US] ET AL) 5 June 2014 (2014-06-05)	1-14
A	claims 1-20 paragraphs [0015], [0033], [0034], [0007], [0013], [0032] examples 1,2	15
Y	US 2001/046479 A1 (LANDA ANDREW SJAAK [GB] ET AL) 29 November 2001 (2001-11-29)	1-14
A	claims 1,14 paragraphs [0055], [0091] tables 1B, 2	15
Y	US 6 624 126 B1 (KASUGA KENNICHI [JP] ET AL) 23 September 2003 (2003-09-23)	1-6,8-14
A	claims 1,3,5 column 8, lines 56-62 column 4, lines 36-42 column 5, lines 23-24 column 3, lines 39-41,61-63	7,15
Y	US 2013/045910 A1 (MIRACLE GREGORY SCOT [US] ET AL) 21 February 2013 (2013-02-21)	1-6,8-14
A	example * tables 1,2 paragraphs [0110] - [0114]	7,15
Y	US 2015/196477 A1 (STARK CYNTHIA MARIE [US] ET AL) 16 July 2015 (2015-07-16)	15
A	claims 1,8,11-13,15,18 paragraphs [0002], [0028], [0039], [0040]	1-14
Y	US 2013/045907 A1 (LANZALACO ANTHONY CHARLES [US] ET AL) 21 February 2013 (2013-02-21)	15
A	claims 1,3-5,7,8,16,17 paragraphs [0067], [0065], [0083]	1-14

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INTERNATIONAL SEARCH REPORT

International application No
PCT/US2018/039972

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2004/089092 A1 (IDEMITSU TECHNOFINE CO LTD [JP]; AMANO KAZUTOSHI [JP]; TAGUCHI TOSHIHA) 21 October 2004 (2004-10-21)	15
A	claim 1 examples 3,9,12 comparative example 1	1-14

Form PCT/ISA/210 (continuation of second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2018/039972

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		BR 9000873 A	13-02-1991
		CA 2008115 A1	23-08-1990
		CN 1046095 A	17-10-1990
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		JP H03197418 A	28-08-1991
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		PH 26758 A	28-09-1992
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P&G Ex. 2001
Page 268

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2018/039972

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		ES 2283418 T3	01-11-2007
		HU 0204294 A2	28-04-2003
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FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-9

a deodorant stick comprising a substituted or unsubstituted 2-pyridinol-N-oxide material and at least one material as defined in claim 1

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2. claims: 10-14

a deodorant stick comprising a substituted or unsubstituted 2-pyridinol-N-oxide material and a chelant, wherein the deodorant stick is substantially anhydrous

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3. claim: 15

a deodorant stick comprising an antimicrobial powder having an average particle size from 1 to 5 micron, wherein the antimicrobial powder is substantially free of aluminium

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Information on Search Strategy - Pilot phase (see OJ 2015, A86)
The type of information contained in this sheet may change during the pilot for improving the usefulness of this new service.

Application Number
PCT/US2018/039972

TITLE: DEODORANT AND ANTIPERSPIRANT COMPOSITIONS

APPLICANT: THE PROCTER & GAMBLE COMPANY

IPC CLASSIFICATION: A61K8/44, A61K8/55, A61K8/42, A61K8/49, A61K8/41, A61K8/34, A61K8/19, A61K8/02, A61Q15/00, A61K8/33, A61K8/368, A61K8/67, A61K8/81, A61K8/92, A61K8/96

EXAMINER: Grenouillat, N

CONSULTED DATABASES: CAPLUS, EPODOC, GNPD, REG

CLASSIFICATION SYMBOLS DEFINING EXTENT OF THE SEARCH:

IPC:

CPC: A61K8/44, A61K8/55, A61K8/42, A61K8/4926, A61K8/41, A61K8/347, A61K8/19, A61K8/0229, A61K2800/412, A61K2800/31, A61K2800/30, A61Q15/00

FI/F-TERMS:

KEYWORDS OR OTHER ELEMENTS FEATURING THE INVENTION:

EPO FORM P04A42

Electronic Patent Application Fee Transmittal

Application Number:	16716542
Filing Date:	17-Dec-2019
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Filer:	Kathleen Yates Carter/Kelley Werdman
Attorney Docket Number:	15149C

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Miscellaneous:				
SUBMISSION- INFORMATION DISCLOSURE STMT	1806	1	240	240
		Total in USD ($)		**240**

Electronic Acknowledgement Receipt

EFS ID:	40283143
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Customer Number:	27752
Filer:	Kathleen Yates Carter/Kelley Werdman
Filer Authorized By:	Kathleen Yates Carter
Attorney Docket Number:	15149C
Receipt Date:	14-AUG-2020
Filing Date:	17-DEC-2019
Time Stamp:	06:08:34
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$240
RAM confirmation Number	E20208D708573616
Deposit Account	162480
Authorized User	Kelley Werdman

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.16 (National application filing, search, and examination fees)

37 CFR 1.17 (Patent application and reexamination processing fees)

37 CFR 1.19 (Document supply fees)

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Transmittal Letter	15149C_SIDS_Letter.pdf	16365 ad14e3a7b21c4a6f220c02be3f4eb7ed603f00e9	no	2
Warnings:					
Information:					
2	Information Disclosure Statement (IDS) Form (SB08)	15149C_SIDS.pdf	96477 d4de341f23d610402a66e36c10a9a3500e44e921	no	6
Warnings:					
Information:					
This is not an USPTO supplied IDS fillable form					
3	Foreign Reference	BE825146.pdf	4468064 5c50010ef7ef6d6c7ddf7a02ad145254524aad6c	no	33
Warnings:					
Information:					
4	Foreign Reference	GB1347950.pdf	677286 2f81fcddd17da6ed92396ee59d48ed2f914e3699	no	10
Warnings:					
Information:					
5	Foreign Reference	GB2048229.pdf	740052 8db3c970f467996cbd8cc4a2a3644c167f10d3be	no	7
Warnings:					
Information:					
6	Foreign Reference	GB2144992.pdf	431423 4ec4270ddd7d946ca919c98f73d09c3aaf4d65fe	no	10
Warnings:					

Information:					
7	Foreign Reference	JP2008110999.pdf	1233266 52dc1371ccf3b18e40608875b66ed9aad5732e15	no	23
Warnings:					
Information:					
8	Foreign Reference	WO2004089092.pdf	3283870 5010b5ff1e8e1caa7e2735924f956a68d987b74b	no	45
Warnings:					
Information:					
9	Foreign Reference	WO2014139449.pdf	1313176 819680b24385250905c412f176ce488f00e84a5e	no	23
Warnings:					
Information:					
10	Non Patent Literature	Kontoghiorghes_2_Hydroxypyridine_N.pdf	419550 085f7940a39434641b12122034ad2d30510152b9	no	6
Warnings:					
Information:					
11	Non Patent Literature	14861M_ISR.pdf	1805108 06fc3667d5e817e936b4309aba7ec38aa27d3801	no	10
Warnings:					
Information:					
12	Fee Worksheet (SB06)	fee-info.pdf	30400 f5d8e8dde8920159762688b86d7b89890095277f	no	2
Warnings:					
Information:					
			Total Files Size (in bytes):	14515037	

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.	:	16/716,542
Inventor(s)	:	David Arthur STURGIS, et al.
Filed	:	December 17, 2019
Art Unit	:	1615
Examiner	:	Melissa S Mercier
Docket No.	:	15149C
Confirmation No.	:	3081
Customer No.	:	27752
Title	:	DEODORANT COMPOSITIONS

INFORMATION DISCLOSURE STATEMENT

Pursuant to 37 C.F.R. §§ 1.56, 1.97 and 1.98, it is respectfully requested that the document(s) cited in this submission be carefully considered by the Examiner and made of record in this case. As provided in §1.97(g), no representation is made or intended that a thorough art search was made. As provided in 37 C.F.R. §1.97(h), this Information Disclosure Statement does not constitute an admission of any kind, and specifically is not an admission that the document(s) cited in this submission is (are), or is (are) considered to be, material to the patentability of the above-identified patent application, as defined in 37 C.F.R. §1.56(b).

This Information Disclosure Statement is being submitted under 37 C.F.R. §1.97(c). I hereby elect to pay the fee set forth in 37 C.F.R. §1.17(p). Authorization is hereby given to charge the fee set forth in 37 C.F.R. §1.17(p) to Deposit Account Number 16-2480 in the name of The Procter & Gamble Company.

Additional Items to be Noted by the Examiner

In accordance with 37 C.F.R. §1.98(a)(2), foreign patent documents and/or non-patent literatures are provided.

The International Search Report from the PCT/EPO is listed herein and a copy is provided.

Respectfully submitted,

THE PROCTER & GAMBLE COMPANY

/Kathleen Y. Carter/

Kathleen Y. Carter
Registration No. 60,583
(513) 983-2114

Date: August 13, 2020
Customer No. 27752

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	16716542
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	1615
Examiner Name	Melissa S. Mercier
Attorney Docket Number	--15149C-US

U.S.PATENTS

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1					

If you wish to add additional U.S. Patent citation information please click the Add button.

U.S.PATENT APPLICATION PUBLICATIONS

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20190000734	A1	2019-01-03	Sturgis, David Arthur	

If you wish to add additional U.S. Published Application citation information please click the Add button.

FOREIGN PATENT DOCUMENTS

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							☐

If you wish to add additional Foreign Patent Document citation information please click the Add button

NON-PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	16716542
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	1615
	Examiner Name	Melissa S. Mercier
	Attorney Docket Number	--15149C-US

	1		☐

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

Examiner Signature		Date Considered	

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.	:	16/716,542
Inventor(s)	:	David Arthur STURGIS, et al.
Filed	:	December 17, 2019
Art Unit	:	1615
Examiner	:	Melissa S Mercier
Docket No.	:	15149C
Confirmation No.	:	3081
Customer No.	:	27752
Title	:	DEODORANT COMPOSITIONS

INFORMATION DISCLOSURE STATEMENT

Pursuant to 37 C.F.R. §§ 1.56, 1.97 and 1.98, it is respectfully requested that the document(s) cited in this submission be carefully considered by the Examiner and made of record in this case. As provided in §1.97(g), no representation is made or intended that a thorough art search was made. As provided in 37 C.F.R. §1.97(h), this Information Disclosure Statement does not constitute an admission of any kind, and specifically is not an admission that the document(s) cited in this submission is (are), or is (are) considered to be, material to the patentability of the above-identified patent application, as defined in 37 C.F.R. §1.56(b).

This Information Disclosure Statement is being submitted under 37 C.F.R. §1.97(c). I hereby elect to pay the fee set forth in 37 C.F.R. §1.17(p). Authorization is hereby given to charge the fee set forth in 37 C.F.R. §1.17(p) to Deposit Account Number 16-2480 in the name of The Procter & Gamble Company.

Respectfully submitted,

THE PROCTER & GAMBLE COMPANY

/Kathleen Y. Carter/
Signature

Kathleen Y. Carter
Registration No. 60,583
(513) 983-2114

Date: October 22, 2020
Customer No. 27752

Electronic Patent Application Fee Transmittal

Application Number:	16716542
Filing Date:	17-Dec-2019
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Filer:	David Michael Weirich/Deepti Rastogi
Attorney Docket Number:	15149C

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Miscellaneous:				
SUBMISSION- INFORMATION DISCLOSURE STMT	1806	1	260	260
		Total in USD ($)		**260**

Electronic Acknowledgement Receipt

EFS ID:	40916693
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Customer Number:	27752
Filer:	David Michael Weirich/Deepti Rastogi
Filer Authorized By:	David Michael Weirich
Attorney Docket Number:	15149C
Receipt Date:	22-OCT-2020
Filing Date:	17-DEC-2019
Time Stamp:	13:20:49
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$260
RAM confirmation Number	E20200LD22005148
Deposit Account	
Authorized User	

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Information Disclosure Statement (IDS) Form (SB08)	15149C_IDSListofReferences_10_22_2020.pdf	65653 e92b5de5a525ec5f8724558f300477bda47f6c0e	no	3
Warnings:					
Information:					
This is not an USPTO supplied IDS fillable form					
2	Transmittal Letter	15149C_IDSLetter_10_22_2020.pdf	74699 5f84aaffcc676b3726cf654e12ed4f09debede23	no	1
Warnings:					
Information:					
3	Fee Worksheet (SB06)	fee-info.pdf	30408 fd86f84a59f02cfda3cbcb9a67aeda54b9347f1f	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	170760		

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.	:	16/716,542
Inventor(s)	:	David Arthur STURGIS, et al.
Filed	:	December 17, 2019
Art Unit	:	1615
Examiner	:	Melissa S Mercier
Docket No.	:	15149C
Confirmation No.	:	3081
Customer No.	:	27752
Title	:	DEODORANT COMPOSITIONS

RESPONSE TO FIRST OFFICE ACTION

INTRODUCTORY REMARKS

The Director is hereby authorized to charge all required fees (including any extension of time fees) and to credit any overpayments in this and any concurrent or future correspondence to Deposit Account No. 16-2480.

In response to the Office Action dated August 4, 2020, please amend the above-identified application as follows, consider the following remarks and reconsider the application.

Claims begin on page 2 of this paper.

Remarks begin on page 4 of this paper.

P&G Ex. 2001
Page 288

Electronic Acknowledgement Receipt

EFS ID:	41006730
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Customer Number:	27752
Filer:	Kathleen Yates Carter/Kelley Werdman
Filer Authorized By:	Kathleen Yates Carter
Attorney Docket Number:	15149C
Receipt Date:	02-NOV-2020
Filing Date:	17-DEC-2019
Time Stamp:	11:02:17
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		15149C_Response_to_OA_dtd_8-4-20.pdf	109027 c5617d5bb8ba87455daf02c10d0e71d61502e7ae	yes	5

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Applicant Arguments/Remarks Made in an Amendment	4	5
	Claims	2	3
	Amendment/Req. Reconsideration-After Non-Final Reject	1	1
Warnings:			
Information:			
	Total Files Size (in bytes):	109027	

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

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New International Application Filed with the USPTO as a Receiving Office

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REMARKS

Claim Status

Claims 1-15 are pending in the present application. No additional claims fee is believed to be due. Claim 2 is amended to correct the spelling of salicylic.

Applicants submit that these changes do not involve any introduction of new matter. Consequently, entry of these changes is believed to be in order and is respectfully requested.

OBJECTION

Claim 2 is objected to for the misspelling of the word salicylic. Claim 2 has been amended with the correct spelling, and Applicants respectfully request that the objection be withdrawn.

DOUBLE PATENTING

Claims 1-15 are rejected on the ground of nonstatutory double patenting over US 10,543,164. Per telephone communication with the Examiner, Applicants understand that Claims 1-15 are not rejected for nonstatutory double patenting over U.S. Application 16/715,542 as stated in the Office Action, but instead are rejected over U.S. Application 16/021,931.

U.S. 10,543,164 and 16/021,931 are commonly owned with the instant application. Without acquiescing to the properness of these rejections, Applicants are submitting a terminal disclaimer to facilitate allowance of the present application. Accordingly, Applicants respectfully request that the rejection be withdrawn.

Conclusion

This response represents an earnest effort to place the present application in proper form. In view of the foregoing, the Applicant respectfully requests the Examiner to reconsider this application, and allow the pending claims.

Respectfully submitted,

THE PROCTER & GAMBLE COMPANY

By /Kathleen Y. Carter/
Kathleen Y. Carter
Registration No. 60,583
(513) 983-2114

Date: November 2, 2020
Customer No. 27752

AMENDMENTS TO THE CLAIMS

The following listing of claims replaces all prior versions and listings of the claims in the application.

1. (Original) A deodorant stick comprising:
   a. at least about 25% of a liquid triglyceride;
   b. at least one antimicrobial; and
   c. a primary structurant with a melting point of at least about 50°Celsius; and

   wherein said stick is free of an aluminum salt.

2. (Currently Amended) The deodorant stick of claim 1, wherein the antimicrobial is selected from the group consisting of baking soda, magnesium hydroxide, hexamidine, magnesium carbonate, zinc carbonate, thymol, magnesium hydroxide & magnesium carbonate hydroxide, sodium carbonate, magnesium carbonate hydroxide, partially carbonated magnesium hydroxide, calcium carbonate, polyvinyl formate, salicylic ~~salycilic~~ acid, niacinamide, cinnamon essential oil, cinnamon bark essential oil, cinnamic aldehyde and combinations thereof.

3. (Original) The deodorant stick of claim 1, wherein the antimicrobial is a powder with a water solubility of at most about 90 g/L at 25°C.

4. (Original) The deodorant stick of claim 1, wherein the deodorant stick is substantially free of baking soda.

5. (Original) The deodorant stick of claim 1, wherein the deodorant stick is substantially free of a synthetic fragrance.

6. (Original) The deodorant stick of any claim 1, wherein the deodorant stick is substantially free of silicones.

7. (Original) The deodorant stick of claim 1, wherein the deodorant stick comprises from 0% to 5% silicones.

8. (Original) The deodorant stick of claim 1, comprising at least about 35% of a liquid triglyceride.

9. (Original) The deodorant stick of claim 1, wherein the antimicrobial comprises baking soda.

10. (Original) The deodorant stick of claim 1, wherein the antimicrobial comprises magnesium hydroxide.

11. (Original) The deodorant stick of claim 1, wherein the stick comprises from about 8% to about 20% of the primary structurant.
12. (Original) The deodorant stick of claim 1, wherein the primary structurant has a melting point from about 50°C to 75°C.

13. (Original) The deodorant stick of claim 1, comprising less than 5% of secondary structurants having a melting point above 60°C.

14. (Original) The deodorant stick of claim 1, wherein said stick comprises less than 8% of secondary structurants having a melting point of at least about 60°Celsius.

15. (Original) The deodorant stick of claim 1, wherein said stick has a hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM-D 1321 needle.

Doc Code: DIST.E.FILE **Document Description: Electronic Terminal Disclaimer - Filed**	PTO/SB/25 PTO/SB/26 U.S. Patent and Trademark Office Department of Commerce

Electronic Petition Request	**TERMINAL DISCLAIMER TO OBVIATE A PROVISIONAL DOUBLE PATENTING REJECTION OVER A PENDING "REFERENCE" APPLICATION AND TERMINAL DISCLAIMER TO OBVIATE A DOUBLE PATENTING REJECTION OVER A "PRIOR" PATENT**
Application Number	16716542
Filing Date	17-Dec-2019
First Named Inventor	David Sturgis
Attorney Docket Number	15149C
Title of Invention	DEODORANT COMPOSITIONS

☒ Filing of terminal disclaimer does not obviate requirement for response under 37 CFR 1.111 to outstanding Office Action

☒ This electronic Terminal Disclaimer is not being used for a Joint Research Agreement.

Owner	Percent Interest
The Procter & Gamble Company	100 %

The owner(s) of percent interest listed above in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of any patent granted on pending reference Application Number(s)

16021931 filed on 06/28/2018

as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed prior to the grant of any patent on the pending reference application. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and any patent granted on the reference application are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.

In making the above disclaimer, the owner does not disclaim the terminal part of any patent granted on the instant application that would extend to the expiration date of the full statutory term of any patent granted on said reference application, "as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed prior to the grant of any patent on the pending reference application," in the event that any such patent granted on the pending reference application: expires for failure to pay a maintenance fee, is held unenforceable, is found invalid by a court of competent jurisdiction, is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321, has all claims canceled by a reexamination certificate, is reissued, or is in any manner terminated prior to the expiration of its full statutory term as shortened by any terminal disclaimer filed prior to its grant.

The owner(s) with percent interest listed above in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of prior patent number(s)

as the term of said prior patent is presently shortened by any terminal disclaimer. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the prior patent are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.

In making the above disclaimer, the owner does not disclaim the terminal part of the term of any patent granted on the instant application that would extend to the expiration date of the full statutory term of the prior patent, "as the term of said prior patent is presently shortened by any terminal disclaimer," in the event that said prior patent later:
- expires for failure to pay a maintenance fee;
- is held unenforceable;
- is found invalid by a court of competent jurisdiction;
- is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321;
- has all claims canceled by a reexamination certificate;
- is reissued; or
- is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer.

(●) Terminal disclaimer fee under 37 CFR 1.20(d) is included with Electronic Terminal Disclaimer request.

( ) I certify, in accordance with 37 CFR 1.4(d)(4), that the terminal disclaimer fee under 37 CFR 1.20(d) required for this terminal disclaimer has already been paid in the above-identified application.

Applicants claims the following fee status:

( ) Small Entity

( ) Micro Entity

(●) Regular Undiscounted

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

THIS PORTION MUST BE COMPLETED BY THE SIGNATORY OR SIGNATORIES

I certify, in accordance with 37 CFR 1.4(d)(4) that I am:

(●) An attorney or agent registered to practice before the Patent and Trademark Office who is of record in this application

Registration Number 60583

( ) A sole inventor

( ) A joint inventor; I certify that I am authorized to sign this submission on behalf of all of the inventors as evidenced by the power of attorney in the application

( ) A joint inventor; all of whom are signing this request

Signature	/Kathleen Y. Carter/
Name	Kathleen Y. Carter

*Statement under 37 CFR 3.73(b) is required if terminal disclaimer is signed by the assignee (owner).
Form PTO/SB/96 may be used for making this certification. See MPEP § 324.

Electronic Patent Application Fee Transmittal

Application Number:	16716542
Filing Date:	17-Dec-2019
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Filer:	Kathleen Yates Carter/Kelley Werdman
Attorney Docket Number:	15149C

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
STATUTORY OR TERMINAL DISCLAIMER	1814	1	170	170
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension-of-Time:				
Miscellaneous:				
			Total in USD ($)	**170**

Doc Code: DISQ.E.FILE
Document Description: Electronic Terminal Disclaimer – Approved

Application No.: 16716542

Filing Date: 17-Dec-2019

Applicant/Patent under Reexamination: Sturgis

Electronic Terminal Disclaimer filed on November 2, 2020

☒ APPROVED

This patent is subject to a terminal disclaimer

☐ DISAPPROVED

Approved/Disapproved by: Electronic Terminal Disclaimer automatically approved by EFS-Web

U.S. Patent and Trademark Office

Electronic Acknowledgement Receipt

EFS ID:	41006905
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Customer Number:	27752
Filer:	Kathleen Yates Carter/Kelley Werdman
Filer Authorized By:	Kathleen Yates Carter
Attorney Docket Number:	15149C
Receipt Date:	02-NOV-2020
Filing Date:	17-DEC-2019
Time Stamp:	11:16:15
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$170
RAM confirmation Number	E2020A2B16120073
Deposit Account	162480
Authorized User	Kelley Werdman

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.16 (National application filing, search, and examination fees)

37 CFR 1.17 (Patent application and reexamination processing fees)

37 CFR 1.19 (Document supply fees)

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Terminal Disclaimer-Filed (Electronic)	eTerminal-Disclaimer.pdf	36087 cb35251b0bec0baf58017b32f9513350a391603a	no	3
Warnings:					
Information:					
2	Fee Worksheet (SB06)	fee-info.pdf	30181 b5e5c69bf36c4980598adf50bd8139ba797b3607	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	66268		

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PTO/SB/06 (09-11)
Approved for use through 1/31/2014. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 16/716,542	Filing Date 12/17/2019	☐ To be Mailed

ENTITY: ☑ LARGE ☐ SMALL ☐ MICRO

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA		RATE ($)	FEE ($)
☐ BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A		N/A	
☐ SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A		N/A	
☐ EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A		N/A	
TOTAL CLAIMS (37 CFR 1.16(i))	minus 20 =	*		x $100 =	
INDEPENDENT CLAIMS (37 CFR 1.16(h))	minus 3 =	*		x $460 =	
☐ APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).				
☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))					
* If the difference in column 1 is less than zero, enter "0" in column 2.				TOTAL	

APPLICATION AS AMENDED - PART II

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT	11/02/2020	CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	* 15	Minus	** 20	= 0		x $100 =	0
	Independent (37 CFR 1.16(h))	* 1	Minus	*** 3	= 0		x $480 =	0
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	0

		(Column 1)		(Column 2)	(Column 3)			
AMENDMENT		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA		RATE ($)	ADDITIONAL FEE ($)
	Total (37 CFR 1.16(i))	*	Minus	**	=		x $0 =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=		x $0 =	
	☐ Application Size Fee (37 CFR 1.16(s))							
	☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							
							TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.	SLIE
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".	/ADRIENE D. SELLMAN/
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".	
The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.	

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

NOTICE OF ALLOWANCE AND FEE(S) DUE

27752 7590 12/09/2020

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

EXAMINER
MERCIER, MELISSA S

ART UNIT	PAPER NUMBER
1615	

DATE MAILED: 12/09/2020

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	12/17/2019	David Arthur Sturgis	15149C	3081

TITLE OF INVENTION: DEODORANT COMPOSITIONS

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	03/09/2021

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

27752 7590 12/09/2020

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

(Typed or printed name)
(Signature)
(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	12/17/2019	David Arthur Sturgis	15149C	3081

TITLE OF INVENTION: DEODORANT COMPOSITIONS

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	03/09/2021

EXAMINER	ART UNIT	CLASS-SUBCLASS
MERCIER, MELISSA S	1615	424-065000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

❑ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

❑ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 _____
2 _____
3 _____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE (B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent) : ❑ Individual ❑ Corporation or other private group entity ❑ Government

4a. Fees submitted: ❑ Issue Fee ❑ Publication Fee (if required) ❑ Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

❑ Electronic Payment via EFS-Web ❑ Enclosed check ❑ Non-electronic payment by credit card (Attach form PTO-2038)

❑ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

❑ Applicant certifying micro entity status. See 37 CFR 1.29

❑ Applicant asserting small entity status. See 37 CFR 1.27

❑ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____ Date _____

Typed or printed name _____ Registration No. _____

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	12/17/2019	David Arthur Sturgis	15149C	3081

27752 7590 12/09/2020

THE PROCTER & GAMBLE COMPANY
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ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

EXAMINER	
MERCIER, MELISSA S	
ART UNIT	PAPER NUMBER
1615	

DATE MAILED: 12/09/2020

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Notice of Allowability	Application No. 16/716,542	Applicant(s) Sturgis et al.	
	Examiner MELISSA S MERCIER	Art Unit 1615	AIA (FITF) Status Yes

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to Applicants remarks and amended claims filed on November 2, 2020.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 1-15 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   Certified copies:

   a) ☐ All b) ☐ Some *c) ☐ None of the:

   1. ☐ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

   Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

1. ☐ Notice of References Cited (PTO-892)
2. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.
5. ☐ Examiner's Amendment/Comment
6. ☑ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/MELISSA S MERCIER/
Primary Examiner, Art Unit 1615

DETAILED ACTION

Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

Summary

Receipt of Applicant's remarks and amended claims filed on November 2, 2020 is acknowledged. Claims 1-15 are pending in this application. Claim 2 has been amended. All claims are under examination in this application.

Information Disclosure Statement

Receipt of the Information Disclosure Statement filed on August 14, 2020 and October 22, 202 is acknowledged. Signed copies are attached to this office action.

Withdrawn Objections/Rejections

Claim Objections

The objection of claim 2 because salicylic is spelled incorrectly as "salycilic" has been withdrawn in view of Applicants amendment to correct the spelling.

Double Patenting

The rejection of claims 1-15 on the ground of nonstatutory double patenting as being unpatentable over claims 1-20 of U.S. Patent No. 10,543,164 has been withdrawn in view of Applicant's filing of a terminal disclaimer.

The rejection of claims 1-15 on the ground of nonstatutory double patenting as being unpatentable over claims 1-19 of U.S. Patent No. Allowed Application 16/021931

(not yet assigned) has been withdrawn in view of Applicant's filing of a terminal disclaimer.

Reasons for Allowance

The following is an examiner's statement of reasons for allowance: The instant claims are free of the prior art.

The closest prior art is that of Lee et al. (US 2002/0086039). Lee discloses cosmetic compositions, for example antiperspirant and deodorant products that can be in the form of a stick. Lee additionally discloses components such as PPG-14 butyl ether, baking soda, dimethicone, isopropyl myristate, mineral oil, octyldodecanol and stearyl alcohol can be included in said sticks. Lee, however, discloses active ingredients such as aluminum zirconium tetrachlorohydrex gly or aluminum chlorohyrate are included.

Additional prior art includes Oblong et al. (US 2007/0203240). Oblong discloses personal care compositions comprising niacinamide, stearyl alcohol, dimethicone, and petrolatum. However, the composition is a moisturizing skin cream/lotion. Examples of solid antiperspirants comprise aluminum zirconium tetrachlorohydrex glycinate.

Scavone (US 6,485,717) and (US 7,033,579) discloses anhydrous antiperspirant and deodorant composition comprising stearyl alcohol, niacinamide, and dimethicone, however, Scavone also requires the use of aluminum zirconium tetrachlorohydrex glycinate.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably

accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to MELISSA S MERCIER whose telephone number is (571)272-9039. The examiner can normally be reached on M-F 5:30 am to 4 pm EST.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Robert Wax can be reached on 571-272-0623. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see https://ppair-my.uspto.gov/pair/PrivatePair. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/MELISSA S MERCIER/
Primary Examiner, Art Unit 1615

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	16/716,542	Sturgis et al.
	Examiner	**Art Unit**
	MELISSA S MERCIER	1615

CPC						
Symbol					**Type**	**Version**
A61K	/	8	/	92	F	2013-01-01
A61Q	/	15	/	00	I	2013-01-01
A61K	/	8	/	19	I	2013-01-01
A61K	/	8	/	732	I	2013-01-01
A61K	/	8	/	375	I	2013-01-01
A61K	/	8	/	0229	I	2013-01-01
A61K	/	8	/	0241	I	2013-01-01
A61K	/	8	/	37	I	2013-01-01
A61K	/	8	/	4926	I	2013-01-01
A61K	/	8	/	922	I	2013-01-01
A61K	/	2800	/	34	A	2013-01-01
A61Q	/	17	/	005	A	2013-01-01
A61K	/	2800	/	30	A	2013-01-01
A61K	/	2800	/	74	A	2013-01-01

CPC Combination Sets							
Symbol					**Type**	**Set**	**Ranking**	**Version**
	/		/					

NONE		Total Claims Allowed:	
(Assistant Examiner)	(Date)	15	
/MELISSA S MERCIER/ Primary Examiner, Art Unit 1615	24 November 2020	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	none

U.S. Patent and Trademark Office

Part of Paper No.: 20201123

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	16/716,542	Sturgis et al.
	Examiner	**Art Unit**
	MELISSA S MERCIER	1615

INTERNATIONAL CLASSIFICATION				
CLAIMED				
A61K8/92	/	8	/	92
A61Q15/00	/	15	/	00
A61K8/19	/	8	/	19
A61K8/02	/	8	/	02
A61Q17/00	/	17	/	00

NON-CLAIMED				
A61K8/73	/	8	/	73
A61K8/37	/	8	/	37
A61K8/49	/	8	/	49

US ORIGINAL CLASSIFICATION	
CLASS	**SUBCLASS**

CROSS REFERENCES(S)						
CLASS	**SUBCLASS (ONE SUBCLASS PER BLOCK)**					

NONE (Assistant Examiner) (Date)	**Total Claims Allowed:** 15	
/MELISSA S MERCIER/ Primary Examiner, Art Unit 1615 (Primary Examiner) 24 November 2020 (Date)	O.G. Print Claim(s) 1	O.G. Print Figure none

U.S. Patent and Trademark Office

Part of Paper No.: 20201123

Issue Classification	Application/Control No.	Applicant(s)/Patent Under Reexamination
	16/716,542	Sturgis et al.
	Examiner	**Art Unit**
	MELISSA S MERCIER	1615

☑ Claims renumbered in the same order as presented by applicant ☐ CPA ☐ T.D. ☐ R.1.47

CLAIMS

Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original	Final	Original
1	1	10	10												
2	2	11	11												
3	3	12	12												
4	4	13	13												
5	5	14	14												
6	6	15	15												
7	7														
8	8														
9	9														

NONE		**Total Claims Allowed:**	
(Assistant Examiner)	(Date)	15	
/MELISSA S MERCIER/ Primary Examiner, Art Unit 1615	24 November 2020	O.G. Print Claim(s)	O.G. Print Figure
(Primary Examiner)	(Date)	1	none

U.S. Patent and Trademark Office

Part of Paper No.: 20201123

Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	16/716,542	Sturgis et al.
	Examiner	Art Unit
	MELISSA S MERCIER	1615

CPC - Searched*		
Symbol	Date	Examiner
no classified search performed	07/31/2020	MMercier

CPC Combination Sets - Searched*		
Symbol	Date	Examiner

US Classification - Searched*			
Class	Subclass	Date	Examiner

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

Search Notes		
Search Notes	Date	Examiner
east- see attached	11/24/2020	MMercier
google-natural deodorant	07/31/2020	MMercier
google-deodorant SAME triglyceride SAME antimicrobial	07/31/2020	MMercier
palm-inventor search	07/31/2020	MMercier

Interference Search			
US Class/CPC Symbol	US Subclass/CPC Group	Date	Examiner
A61K	deodorant AND liquid triglyceride AND antimicrobial	11/24/2020	MMercier

/MELISSA S MERCIER/ Examiner, Art Unit 1615	

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (**Not for submission under 37 CFR 1.99)**		
	Application Number	16716542
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	1615
	Examiner Name	Melissa S. Mercier
	Attorney Docket Number	--15149C-US

U.S.PATENTS						
Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1					
If you wish to add additional U.S. Patent citation information please click the Add button.						

U.S.PATENT APPLICATION PUBLICATIONS						
Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20190000734	A1	2019-01-03	Sturgis, David Arthur	
If you wish to add additional U.S. Published Application citation information please click the Add button.						

FOREIGN PATENT DOCUMENTS								
Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2]	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1							☐
If you wish to add additional Foreign Patent Document citation information please click the Add button								

NON-PATENT LITERATURE DOCUMENTS			
Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T[5]

/MELISSA S MERCIER/ 11/24/2020

EFS Web 2.1.17

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	16716542
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	1615
	Examiner Name	Melissa S. Mercier
	Attorney Docket Number	--15149C-US

	1		☐

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

Examiner Signature	/MELISSA S MERCIER/	Date Considered	11/24/2020

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

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The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

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6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

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9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

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Page 319

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Bibliographic Data

Application No: 16/716,542

Foreign Priority claimed: ○ Yes ◉ No

35 USC 119 (a-d) conditions met: ☐ Yes ☑ No ☐ Met After Allowance

Verified and Acknowledged: /MELISSA S MERCIER/ Examiner's Signature | Initials

Title: DEODORANT COMPOSITIONS

FILING or 371(c) DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.
12/17/2019	424	1615	15149C
RULE			

APPLICANTS

The Procter & Gamble Company, Cincinnati, OH,

INVENTORS

David Arthur Sturgis, Montgomery, OH, UNITED STATES

Lindsey Michelle Britt, West Chester, OH, UNITED STATES

CONTINUING DATA

This application is a CON of 16021965 06/28/2018 PAT 10543164

16021965 has PRO of 62647104 03/23/2018

16021965 has PRO of 62527180 06/30/2017

FOREIGN APPLICATIONS

IF REQUIRED, FOREIGN LICENSE GRANTED**

01/21/2020

STATE OR COUNTRY

UNITED STATES

ADDRESS

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202
UNITED STATES

FILING FEE RECEIVED

$1,720

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
S1	194	"4049792"	USPAT	OR	OFF	2019/05/30 06:43
S2	117	"ASTM D-1321"	USPAT	OR	OFF	2019/05/30 06:47
S3	117	"ASTM D-1321"	USPAT	OR	ON	2019/05/30 06:47
S4	12	"ASTM D-1321" AND antimicrobial	USPAT	OR	ON	2019/05/30 06:50
S5	33	"ASTM D-1321" AND antimicrobial	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 06:50
S6	10	"ASTM D-1321" AND deodorant	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:23
S7	122	"ASTM D-1321" AND hardness	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:25
S8	82	"ASTM D-1321" SAME hardness	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:26
S9	467	"3792068"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 08:26
S10	174	"3792068"	USPAT	OR	ON	2019/05/30 08:27
S11	162	"3887692"	USPAT	OR	ON	2019/05/30 08:27
S12	167	"3904741"	USPAT	OR	ON	2019/05/30 08:28
S13	187	"5019375"	USPAT	OR	ON	2019/05/30 08:29
S14	17	"529806"	USPAT	OR	ON	2019/05/30 08:30
S15	144	"5429816"	USPAT	OR	ON	2019/05/30 08:30

EAST Search History (Prior Art)

S16	54	"5891424"	USPAT	OR	ON	2019/05/30 08:32
S17	65	"5976514"	USPAT	OR	ON	2019/05/30 08:33
S18	18	"4906454"	USPAT	OR	ON	2019/05/30 08:34
S19	41	"5972319"	USPAT	OR	ON	2019/05/30 08:35
S20	20	"6177066"	USPAT	OR	ON	2019/05/30 08:35
S21	1	"8460720"	USPAT	OR	ON	2019/05/30 08:42
S22	1	"8574559"	USPAT	OR	ON	2019/05/30 08:44
S23	6	"8672067"	USPAT	OR	ON	2019/05/30 08:45
S24	1	"9468596"	USPAT	OR	ON	2019/05/30 08:50
S25	3	"9949920"	USPAT	OR	ON	2019/05/30 09:04
S26	0	"20030235546"	USPAT	OR	ON	2019/05/30 09:04
S27	1	"20030235546"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:05
S28	1	"20050281767"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:05
S29	1	"20110076309"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:06
S30	1	"20110196786"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:06
S31	1	"20160235661"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:11
S32	1	"20190000730"	US-PGPUB; USPAT	OR	ON	2019/05/30 09:12
S33	978,061	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone)	US-PGPUB; USPAT	OR	ON	2019/05/30 09:18
S34	220,470	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:06

EAST Search History (Prior Art)

S35	224,991	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss)	US-PGPUB; USPAT	OR	ON	2019/05/30 12:10
S36	509,700	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:15

EAST Search History (Prior Art)

S37	215,206	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") AND ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	US-PGPUB; USPAT	OR	ON	2019/05/30 12:55

EAST Search History (Prior Art)

S38	47,436	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) AND ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") AND ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND (stick hardness)	US-PGPUB; USPAT	OR	ON	2019/05/30 13:18

EAST Search History (Prior Art)

S39	38,478	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	US-PGPUB; USPAT	OR	ON	2019/05/30 13:19

EAST Search History (Prior Art)

S40	15,506	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde")	USPAT	OR	ON	2019/05/30 13:20

EAST Search History (Prior Art)

S41	1,327	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND stick	USPAT	OR	ON	2019/05/30 13:29

EAST Search History (Prior Art)

S42	198	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide") AND stick	USPAT	OR	ON	2019/05/30 13:31

EAST Search History (Prior Art)

S43	438	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant	USPAT	OR	ON	2019/05/30 13:32

EAST Search History (Prior Art)

S44	127	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	USPAT	OR	ON	2019/05/30 13:33

EAST Search History (Prior Art)

S45	399	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol" orokerite candelilla carnauba espartograss "cork wax" guaruma "rice oil wax" "sugar cane wax" ouricury "montan ester wax" "sunflower wax" shellac "microcrystalline wax" "sasol wax" polyethylene polymethylene "ethylene glycol dipalmitate" "behenyl behenate" "glyceryl tribehenate" "hydrogenated castor oil" "castor wax" "behenyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/05/30 14:20
S46	218	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/13 15:50

EAST Search History (Prior Art)

S47	218	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/14 07:07
S48	5	"4005186"	US-PGPUB; USPAT	OR	ON	2019/06/17 14:23
S49	5	"6485717"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2019/06/17 19:39
S50	17	"5744146"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:47
S51	1	"9517193"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:55
S52	1	"20170252288"	US-PGPUB; USPAT	OR	ON	2019/10/07 06:57
S53	6	"6231842"	US-PGPUB; USPAT	OR	ON	2020/07/28 13:38
S54	3	"10543164"	US-PGPUB; USPAT	OR	ON	2020/07/28 13:42

EAST Search History (Prior Art)

S55	373	(olive coconut sunflower jojoba avocado canola corn mineral shea "PPG-14 butyl ether" "isopropyl myristate" petrolatum "butyl stearate" "cetyl octanoate" "butyl myristate" "myristyl myristate" finsolv octyldodecanol "isostearyl isostearate" "octododecyl benzoate" "isostearyl lactate" "isostearyl palmitate" "isobutyl stearate" dimethicone) SAME ("japan wax" "lemon wax" "grapefruit wax" "beeswax" "ceresine" "paraffin" "hydrogenated jojoba" "ethylene glycol distearate" "stearyl stearate" "palmityl stearate" "stearyl behenate" "cetearyl behenate" "hydrogenated high erucic acid rapeseed oil" "stearyl alcohol") SAME ("baking soda" "magnesium hydroxide" "hexamidine" "magnesium carbonate" "zinc carbonate" "thymol" "sodium carbonate" "magnesium carbonate hydroxide" "partially carbonated magnesium hydroxide" "calcium carbonate" "polyvinyl formate" "salycilic acid" "niacinamide" "cinnamon essential oil" "cinnamon bark essential oil" "cinnamic aldehyde") AND deodorant SAME (stick solid)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/07/30 16:49
S56	0	"2016048425"	US-PGPUB; USPAT	OR	ON	2020/08/19 16:09
S57	10	"2016048425"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/08/19 16:09
S58	8	"5298640"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:23
S59	18	"5744146"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:23
S60	12	"5744146"	USPAT	OR	ON	2020/10/02 15:23
S61	18	"5744146"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:42
S62	17	"6624126"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:44
S63	2	"9517193"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:45
S64	3	"10555884"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:56
S65	1	"20010046479"	US-PGPUB; USPAT	OR	ON	2020/10/02 15:58
S66	1	"20130045907"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:01

EAST Search History (Prior Art)

S67	2	"20130045910"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:03
S68	2	"20140154189"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:04
S69	1	"20150196477"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:04
S70	1	"20160074300"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:05
S71	2	"20160326091"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:07
S72	1	"20170172873"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:07
S73	2	"20170252288"	US-PGPUB; USPAT	OR	ON	2020/10/02 16:11
S74	3	"10470999"	US-PGPUB; USPAT	OR	ON	2020/10/05 07:52
S75	10	"2016048425"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 07:55
S76	3	"20170252288"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 09:55
S77	17	"10555884"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 11:42
S78	244,356	triglyceride	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:20
S79	39,037	triglyceride AND antimicrobial	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:20

EAST Search History (Prior Art)

S80	4,026	triglyceride AND antimicrobial AND deodorant	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:20
S81	657	triglyceride AND antimicrobial SAME (beryllium magnesium calcium piroctone) AND deodorant	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:21
S82	418	triglyceride AND antimicrobial SAME (beryllium magnesium calcium piroctone) AND deodorant AND stick	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:29
S83	292	triglyceride AND antimicrobial SAME (beryllium magnesium calcium piroctone) AND deodorant AND stick AND fragrance SAME oil	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2020/10/05 13:36
S84	14,203	((("THE") near3 ("PROCTER") near3 ("&") near3 ("GAMBLE") near3 ("COMPANY"))). AS,AANM.	USPAT	OR	OFF	2020/10/29 14:33
S85	75	((("STURGIS") near3 ("David")) OR (("BRITT") near3 ("Lindsey"))).INV.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	OFF	2020/10/29 14:33

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT
(Not for submission under 37 CFR 1.99)

Application Number	16716542
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	1615
Examiner Name	Melissa S Mercier
Attorney Docket Number	--15149C-US

U.S.PATENTS

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	3792068	A	1974-02-12	Luedders, W	
	2	3887692	A	1975-06-03	Gilman, William S	
	3	3904741	A	1975-09-09	Jones, John L	
	4	4120948	A	1978-10-17	Shelton, David Lee	
	5	4359456	A	1982-11-16	Gosling, Keith	
	6	5298640	A	1994-03-29	Callaghan, David T	
	7	5675013	A	1997-10-07	Hani, Rahim	
	8	6624126	B1	2003-09-23	Kasuga et al.	

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ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

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	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	9	7033576	B2	2006-04-25	Chevallier, Yvonick	
	10	10555884	B2	2020-02-11	Sturgis, David Arthur	

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U.S.PATENT APPLICATION PUBLICATIONS

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20010046479	A1	2001-11-29	Landa, Andrew Sjaak	
	2	20070003499	A1	2007-01-04	Shen, Yanfei	
	3	20130045907	A1	2013-02-21	Lanzalaco, Anthony Charles	
	4	20130045910	A1	2013-02-21	Miracle, Gregory Scot	
	5	20140154189	A1	2014-06-05	Polson et al.	
	6	20150196477	A1	2015-07-16	Stark, Cynthia Marie	
	7	20160074300	A1	2016-03-17	Salvador, Charlie Reyes	

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	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	8	20160326091	A1	2016-11-10	Rudolph, Thomas	
	9	20170172873	A1	2017-06-22	Banowski, Bernhard	
	10	20190000736	A1	2019-01-03	Sturgis, David Arthur	
	11	20190276389	A1	2019-09-12	Wos, John August	Equiv. for WO 2017/106279 A1
	12	20200000694	A9	2020-01-02	Sturgis, David Arthur	
	13	20200214957	A1	2020-07-09	Sturgis, David Arthur	

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FOREIGN PATENT DOCUMENTS

Examiner Initial*	Cite No	Foreign Document Number[3]	Country Code[2] i	Kind Code[4]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear	T[5]
	1	825146	BE	A1	1975-08-04	Procter & Gamble		☒
	2	1347950	GB	A	1985-03-20	Bristol-Myers		☐
	3	2048229	GB	A	1980-12-10	Gillette Co		☐

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	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	4	2144992	GB	A	1985-03-20	Gillette Co		☐
	5	2008110999	JP	A	2008-05-15	Mandom Corp		☒
	6	2004089092	WO	A1	2004-10-21	Idemitsu Technofine Co Ltd		☒
	7	2014139449	WO	A1	2014-09-18	Procter & Gamble		☐

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NON-PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published.	T5
	1	~~All Office Actions, U.S. Patent Appl. No. 16/021,877 (14861M), SEE PAIR~~	☐
	2	~~All Office Actions, U.S. Patent Appl. No. 16/021,931 (15150), SEE PAIR~~	☐
	3	All Office Actions, U.S. Patent Appl. No. 16/021,965 (15149), SEE PAIR	☐
	4	All Office Actions, U.S. Patent Appl. No. 16/716,540 (14861MC), SEE PAIR	☐
	5	International Search Report; International Application No. PCT/US2018/039972; date of mailing 25 March 2019; 10 pages.	☐

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	Examiner Name	Melissa S Mercier
	Attorney Docket Number	--15149C-US

	6	Kontoghiorghes, George J., "2-Hydroxypyridine-N -Oxides: Effective New Chelators in Iron Mobilisation", Biochimica et Biophysica Acta, Vol. 924, No. 1, April 16, 1987, pp. 13-18.	☐

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Examiner Signature	11/24/2020	Date Considered	11/24/2020

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

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PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

27752 7590 12/09/2020

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

Linda S. Wilson	(Typed or printed name)
/Linda S Wilson/	(Signature)
March 5, 2021	(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	12/17/2019	David Arthur Sturgis	15149C	3081

TITLE OF INVENTION: DEODORANT COMPOSITIONS

APPLN. TYPE	ENTITY STATUS	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	UNDISCOUNTED	$1200	$0.00	$0.00	$1200	03/09/2021

EXAMINER	ART UNIT	CLASS-SUBCLASS
MERCIER, MELISSA S	1615	424-065000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 Kathleen Y. Carter
2
3

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: The Procter & Gamble Company

(B) RESIDENCE: (CITY and STATE OR COUNTRY): Cincinnati, OH

Please check the appropriate assignee category or categories (will not be printed on the patent) : ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☒ Issue Fee ☒ Publication Fee (if required) ☐ Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

☒ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☒ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. 16-2480

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature /Kathleen Y. Carter/ Date March 2, 2021

Typed or printed name Kathleen Y. Carter Registration No. 60,583

Electronic Patent Application Fee Transmittal

Application Number:	16716542
Filing Date:	17-Dec-2019
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Filer:	Kathleen Yates Carter/Linda Wilson
Attorney Docket Number:	15149C

Filed as Large Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
UTILITY APPL ISSUE FEE	1501	1	1200	1200

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension-of-Time:				
Miscellaneous:				
			Total in USD ($)	**1200**

Electronic Acknowledgement Receipt

EFS ID:	42096468
Application Number:	16716542
International Application Number:	
Confirmation Number:	3081
Title of Invention:	DEODORANT COMPOSITIONS
First Named Inventor/Applicant Name:	David Arthur Sturgis
Customer Number:	27752
Filer:	Kathleen Yates Carter/Linda Wilson
Filer Authorized By:	Kathleen Yates Carter
Attorney Docket Number:	15149C
Receipt Date:	05-MAR-2021
Filing Date:	17-DEC-2019
Time Stamp:	06:57:15
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	$1200
RAM confirmation Number	E202135758441405
Deposit Account	162480
Authorized User	Linda Wilson

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.16 (National application filing, search, and examination fees)

37 CFR 1.17 (Patent application and reexamination processing fees)

37 CFR 1.19 (Document supply fees)

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Issue Fee Payment (PTO-85B)	15149C-IssueFeeTransmittal-AsFiled-3-5-21.pdf	175536 d05bd373c26d7c90326afbb11b2c916381f97352	no	1
Warnings:					
Information:					
2	Fee Worksheet (SB06)	fee-info.pdf	30032 6df76feec8b1c10c9c46f446211b6b851f5eb204	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	205568		

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)		
	Application Number	
	Filing Date	2019-12-17
	First Named Inventor	STURGIS David Arthur (STURG/DA)
	Art Unit	
	Examiner Name	
	Attorney Docket Number	--15149C-US

	9	9468596	B2	2016-10-18	Eizen	
	10	9949920	B2	2018-04-24	Hakim Noha N.	

If you wish to add additional U.S. Patent citation information please click the Add button. Add

U.S.PATENT APPLICATION PUBLICATIONS Remove

Examiner Initial*	Cite No	Publication Number	Kind Code[1]	Publication Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	20030235546	A1	2003-12-25	Mattai, Jairajh ; et al.	
	2	20050281767	A1	2005-12-22	Walling et al.	
	3	20110076309	A1	2011-03-31	MISNER H STEVEN; FAN AIXING; KILPATRICK-LIVERMAN L	
	4	~~20110196786~~	~~A1~~	~~2017-07-13~~	~~Avon Products, Inc.~~	
	5	20160235661	A1	2016-08-18	~~AXIM Biotechnologies, Inc.~~ Changoer et al.	
Change(s) applied to document, /C.G./ 1/19/2021	6	20190000730	A1	2019-01-03	~~DEAD SEA BROMINE COMPANY LTD.~~ Abuelhaiga et al.	

If you wish to add additional U.S. Published Application citation information please click the Add button. Add

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/MELISSA S MERCIER/

07/30/2020

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /M.S.M/

Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (03-15)
Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Not for submission under 37 CFR 1.99)

Application Number	
Filing Date	2019-12-17
First Named Inventor	STURGIS David Arthur (STURG/DA)
Art Unit	
Examiner Name	
Attorney Docket Number	–15149C-US

U.S.PATENTS Remove

Examiner Initial*	Cite No	Patent Number	Kind Code[1]	Issue Date	Name of Patentee or Applicant of cited Document	Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear
	1	4906454		1990-03-06	Melanson, Jr.	
	2	5972319		1999-10-26	Linn , et al.	
	3	6177066	B1	2001-01-23	Pataut	
	4	6503944		2003-01-07	Chanchani	
	5	7425321	B2	2008-09-16	Lemoine	
Change(s) applied to document, /C.G./ 1/19/2021	6	8460720	B2	2013-06-11	~~Tom's of Maine, Inc.~~ Bergeron et al.	
	7	8574559	B2	2013-11-05	Banowski	
	8	8672067	B2	2014-03-18	Toyota Jidosha Kabushiki Kaisha	

/MELISSA S MERCIER/ 07/30/2020

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UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
16/716,542	04/06/2021	10966915	15149C	3081

27752 7590 03/17/2021

THE PROCTER & GAMBLE COMPANY
GLOBAL IP SERVICES
CENTRAL BUILDING, C9
ONE PROCTER AND GAMBLE PLAZA
CINCINNATI, OH 45202

ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(application filed on or after May 29, 2000)

The Patent Term Adjustment is 0 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Data Management (ODM) at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

David Arthur Sturgis, Montgomery, OH;
The Procter & Gamble Company, Cincinnati, OH;
Lindsey Michelle Britt, West Chester, OH;

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The USA offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to encourage and facilitate business investment. To learn more about why the USA is the best country in the world to develop technology, manufacture products, and grow your business, visit SelectUSA.gov.

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ELECTRONIC ACKNOWLEDGEMENT RECEIPT

APPLICATION #	RECEIPT DATE / TIME	ATTORNEY DOCKET #
16/716,542	**09/19/2023 11:16:16 AM ET**	**15149C**

Title of Invention

DEODORANT COMPOSITIONS

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	10966915
CONFIRMATION #	3081	FILED BY	Stephanie Powell
PATENT CENTER #	62820248	FILING DATE	12/17/2019
CUSTOMER #	27752	FIRST NAMED INVENTOR	David Arthur Sturgis
CORRESPONDENCE ADDRESS	-	AUTHORIZED BY	Kathleen Carter

Documents

TOTAL DOCUMENTS: 2

DOCUMENT	PAGES	DESCRIPTION	SIZE (KB)
petition-request.pdf	3	Terminal Disclaimer-Filed (Electronic)	50 KB
grantLetter.pdf	1	Terminal Disclaimer-Electronic-Approved	19 KB

Digest

DOCUMENT	MESSAGE DIGEST(SHA-512)
petition-request.pdf	5A5C470F204FD6DCCF0172E88A7C689555A5E45560BD6665C D8BF4BE5C8DEBBAD5A583F6A8D6821B401748789964F4D92A

BB9DA004BE6DDD47538700CD44D632

grantLetter.pdf 01D8276C40A612C4CE10F0F80410F520076ADA161B8E457573
3C19A8CE4FF1CAB9E207930B942C723D2325AEEDEDA81F2D
DA1D2DF524D079AFA8C2CBD343D603

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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ELECTRONIC PAYMENT RECEIPT

APPLICATION #	RECEIPT DATE / TIME	ATTORNEY DOCKET #
16/716,542	**09/19/2023 11:16:16 AM ET**	**15149C**

Title of Invention

DEODORANT COMPOSITIONS

Application Information

APPLICATION TYPE	Utility - Nonprovisional Application under 35 USC 111(a)	PATENT #	10966915
CONFIRMATION #	3081	FILED BY	Stephanie Powell
PATENT CENTER #	62820248	AUTHORIZED BY	Kathleen Carter
CUSTOMER #	27752	FILING DATE	12/17/2019
CORRESPONDENCE ADDRESS	-	FIRST NAMED INVENTOR	David Arthur Sturgis

Payment Information

PAYMENT METHOD	PAYMENT TRANSACTION ID	PAYMENT AUTHORIZED BY
DA / 162480	E20239IB16305143	Kathleen Carter

FEE CODE	DESCRIPTION	ITEM PRICE($)	QUANTITY	ITEM TOTAL($)
1814	STATUTORY DISCLAIMER, INCLUDING TERMINAL DISCLAIMER	170.00	1	170.00
			TOTAL AMOUNT:	**$170.00**

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application

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If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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APPROVAL LETTER

APPLICATION #	FILING DATE	APPLICANT/PATENT UNDER REEXAMINATION
16/716,542	12/17/2019	David Sturgis

Title of Invention

DEODORANT COMPOSITIONS

Electronic terminal disclaimer filed on 09/19/2023

☑ Approved

This patent is subject to a Terminal Disclaimer

Approved / Disapproved by: Electronic Terminal Disclaimer automatically approved

UNITED STATES
PATENT AND TRADEMARK OFFICE

P.O. Box 1450
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TERMINAL DISCLAIMER TO OBVIATE A PROVISIONAL DOUBLE PATENTING REJECTION OVER A PENDING "REFERENCE" APPLICATION

APPLICATION #	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET #
16716542	12/17/2019	David Sturgis	15149C

Title of Invention

DEODORANT COMPOSITIONS

☑ Filing of terminal disclaimer does not obviate requirement for response under 37 CFR 1.111 to outstanding Office Action

☑ This electronic Terminal Disclaimer is not being used for a Joint Research Agreement.

Owner	Percent interest
The Procter &Gamble Company	100%
Total	100%

The owner(s) of percent interest listed above in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of any patent granted on pending reference Application Number(s)

Application #	Filing Date
17979822	11/03/2022
17961089	10/06/2022

as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed

prior to the grant of any patent on the pending reference application. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and any patent granted on the reference application are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.
In making the above disclaimer, the owner does not disclaim the terminal part of any patent granted on the instant application that would extend to the expiration date of the full statutory term of any patent granted on said reference application, "as the term of any patent granted on said reference application may be shortened by any terminal disclaimer filed prior to the grant of any patent on the pending reference application," in the event that any such patent granted on the pending reference application: expires for failure to pay a maintenance fee, is held unenforceable, is found invalid by a court of competent jurisdiction, is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321, has all claims canceled by a reexamination certificate, is reissued, or is in any manner terminated prior to the expiration of its full statutory term as shortened by any terminal disclaimer filed prior to its grant.

The owner(s) of percent interest listed above in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of prior patent number(s)

Patent #
11540999
11497706

as the term of said prior patent is presently shortened by any terminal disclaimer. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the prior patent are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.
In making the above disclaimer, the owner does not disclaim the terminal part of the term of any patent granted on the instant application that would extend to the expiration date of the full statutory term of the prior patent, "as the term of said prior patent is presently shortened by any terminal disclaimer," in the event that said prior patent later:
- expires for failure to pay a maintenance fee;
- is held unenforceable;
- is found invalid by a court of competent jurisdiction;
- is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321;
- has all claims canceled by a reexamination certificate;
- is reissued; or
- is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer.



Terminal disclaimer fee under 37 CFR 1.20(d) included with Electronic Terminal Disclaimer request.

Applicant claims the following entity status:
Regular Undiscounted

I hereby declare that all statements made herein of my own knowledge are true and that all statemnts made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

I certify, in accordance with 37 CFR 1.4(d)(4) that I am: An attorney or agent registered to practice before the Patent and Trademark Office who is of record in this application

Signature	Name	Registration #
/Kathleen Y. Carter/	Kathleen Carter	60583

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* Statement under 37 CFR 3.73(b) is required if terminal disclaimer is signed by the assignee (owner). Form PTO/SB/96 may be used for making this certification. See MPEP 324.