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(54) **ANTIPERSPIRANT ACTIVE COMPOSITIONS
HAVING SEC CHROMATOGRAM
EXHIBITING HIGH SEC PEAK 4 INTENSITY**

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(57) **ABSTRACT**

An antiperspirant active composition comprising an aluminum salt having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, exhibiting a SEC chromatogram having a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution. The composition can optionally include zirconium. Also, methods of making.

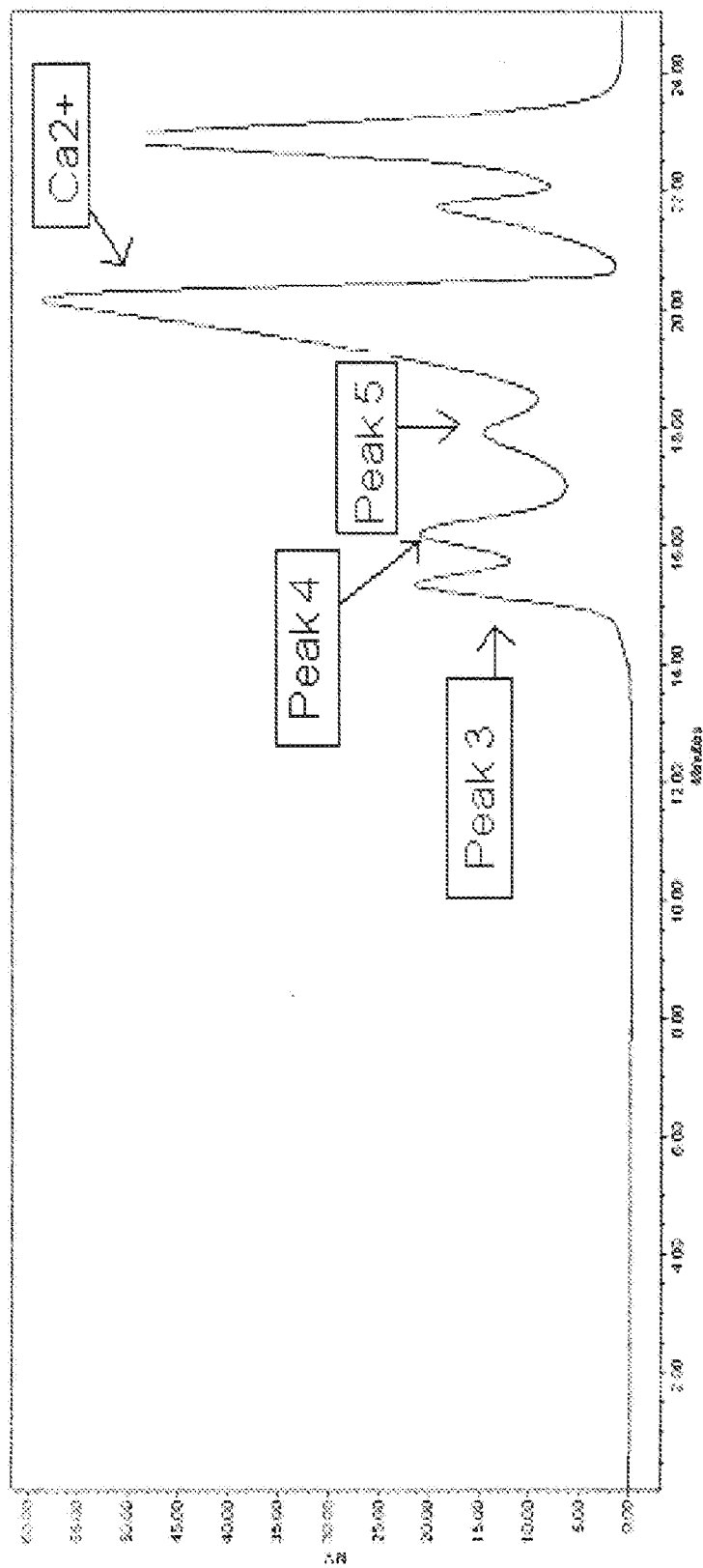


Figure 1

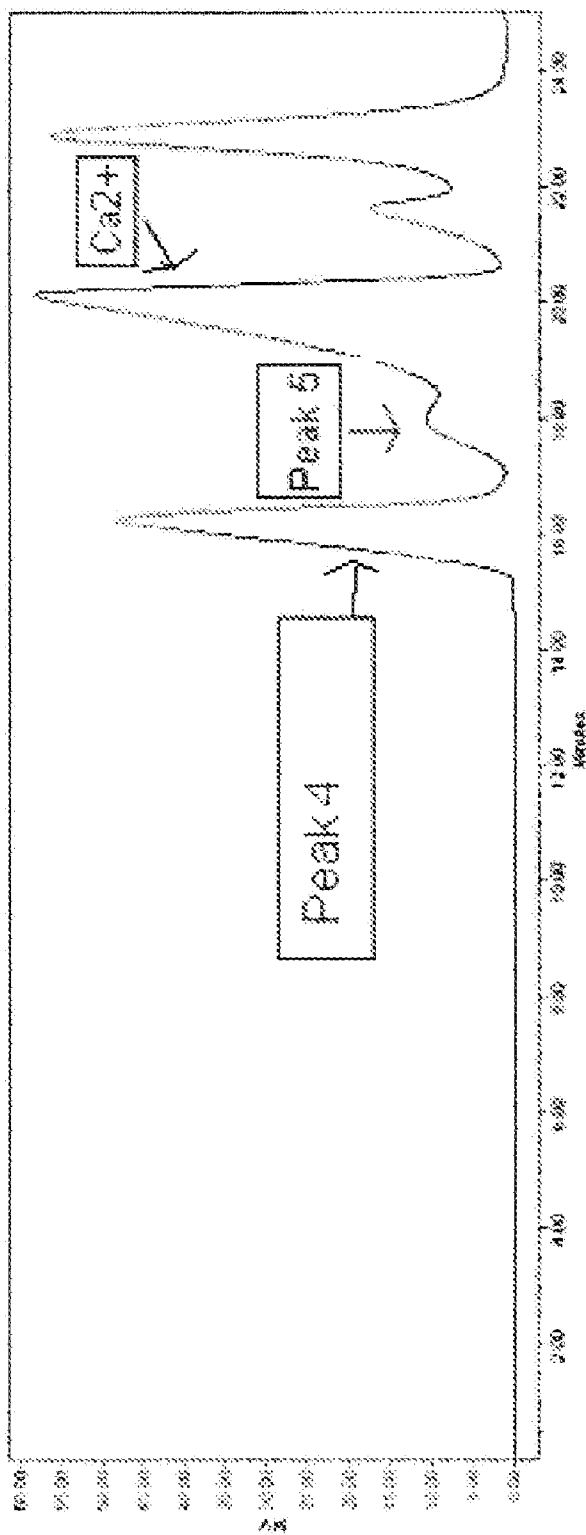


Figure 2

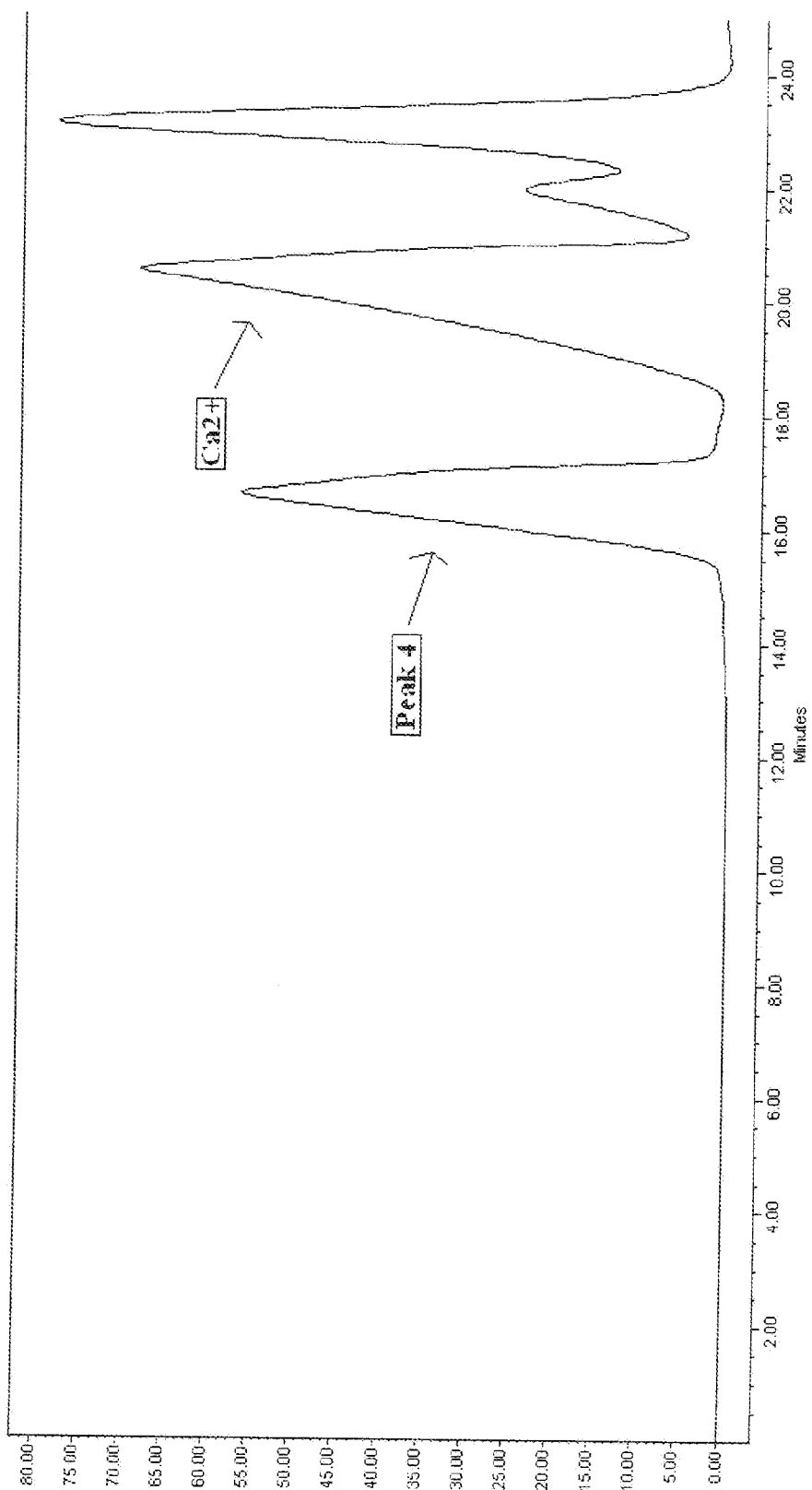


Figure 3

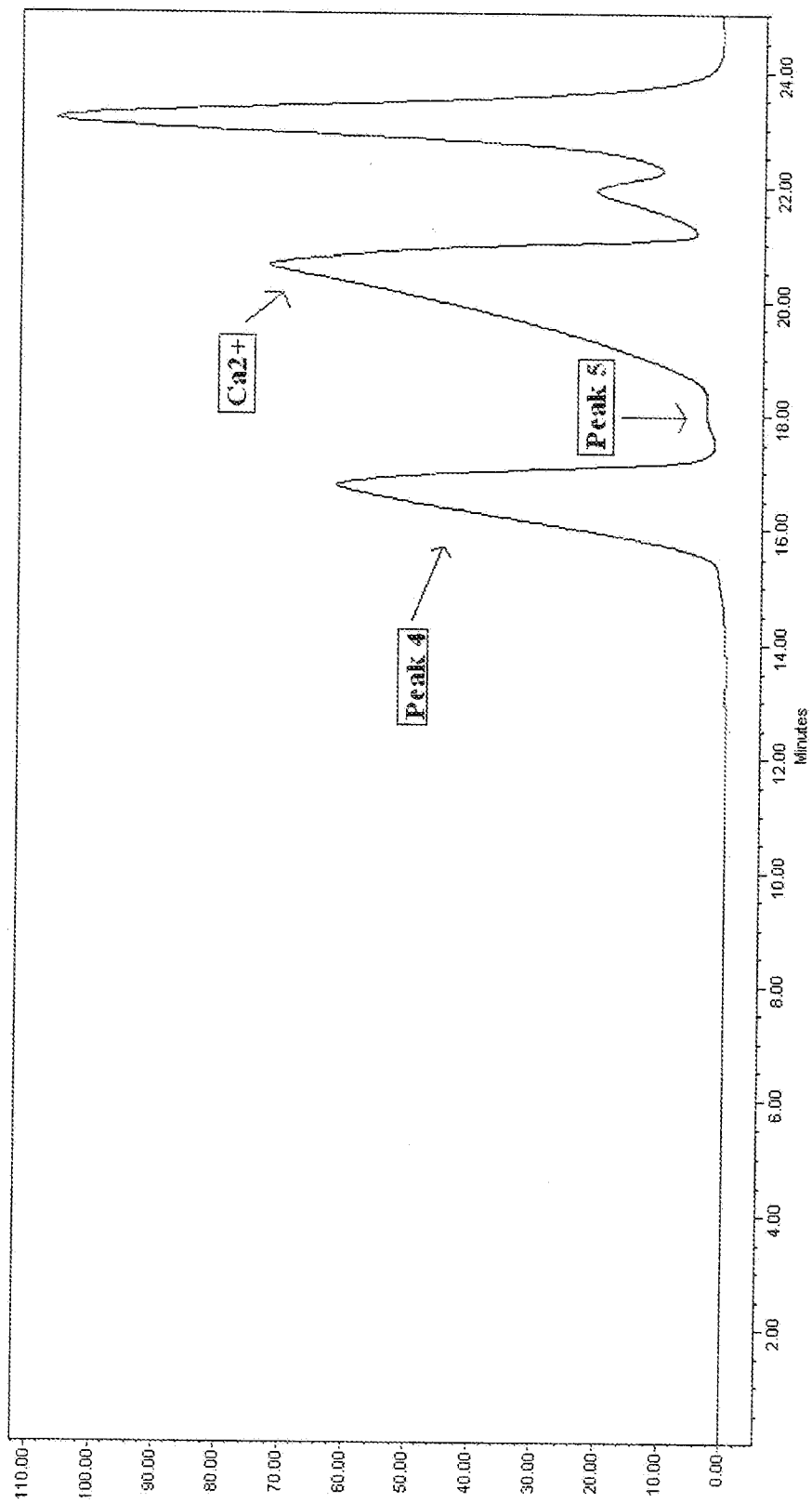


Figure 4

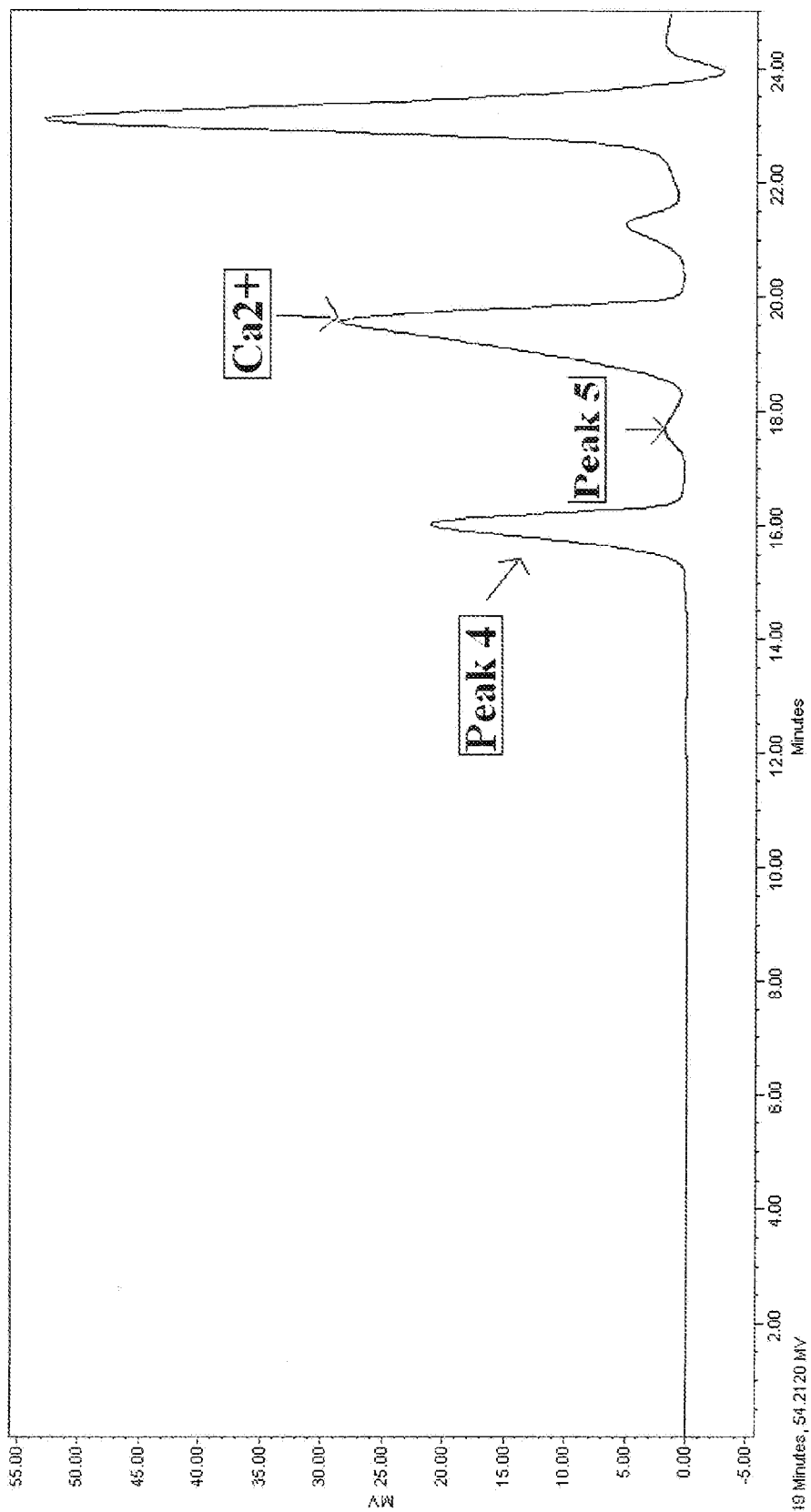


Figure 5

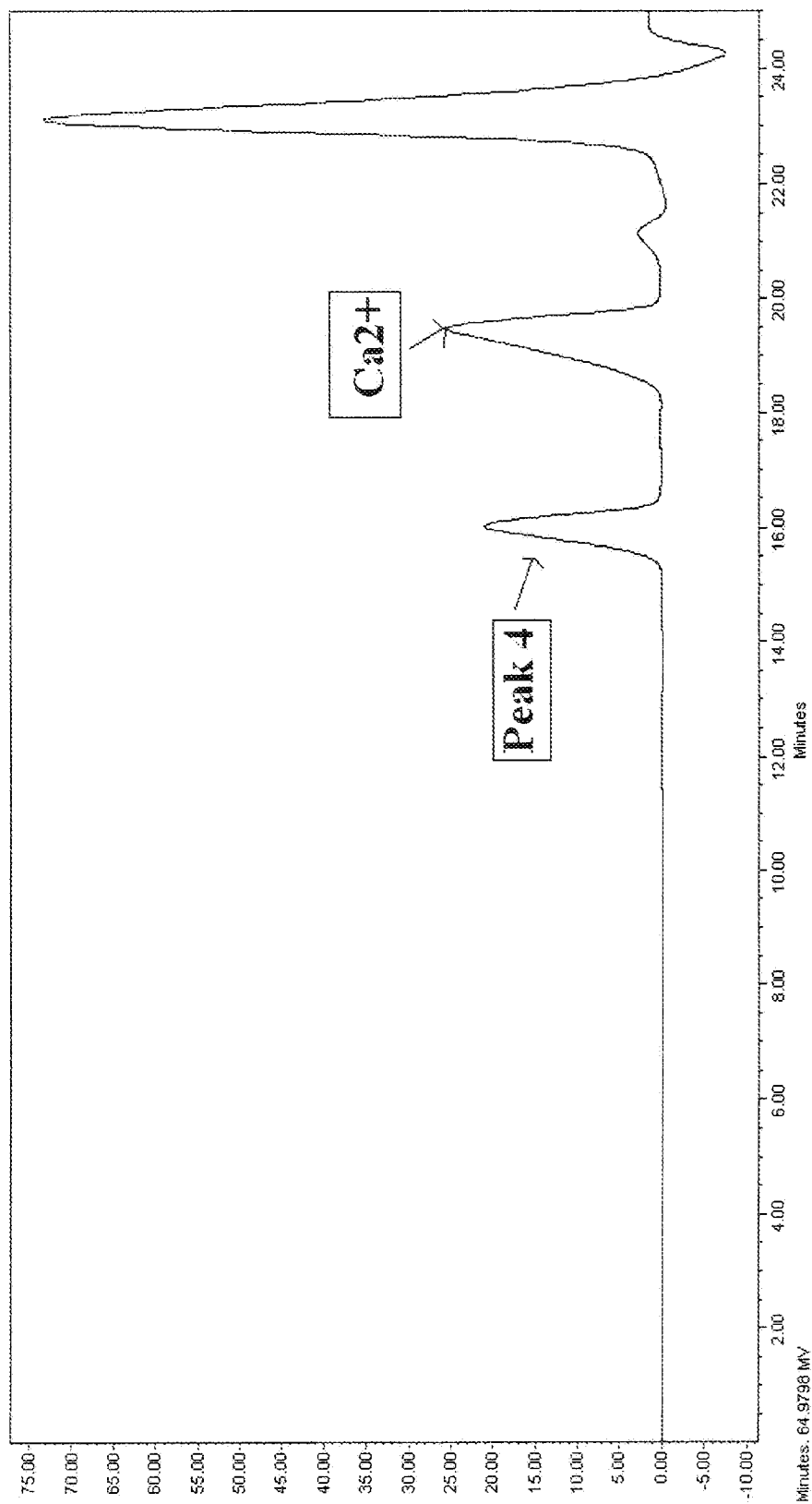


Figure 6

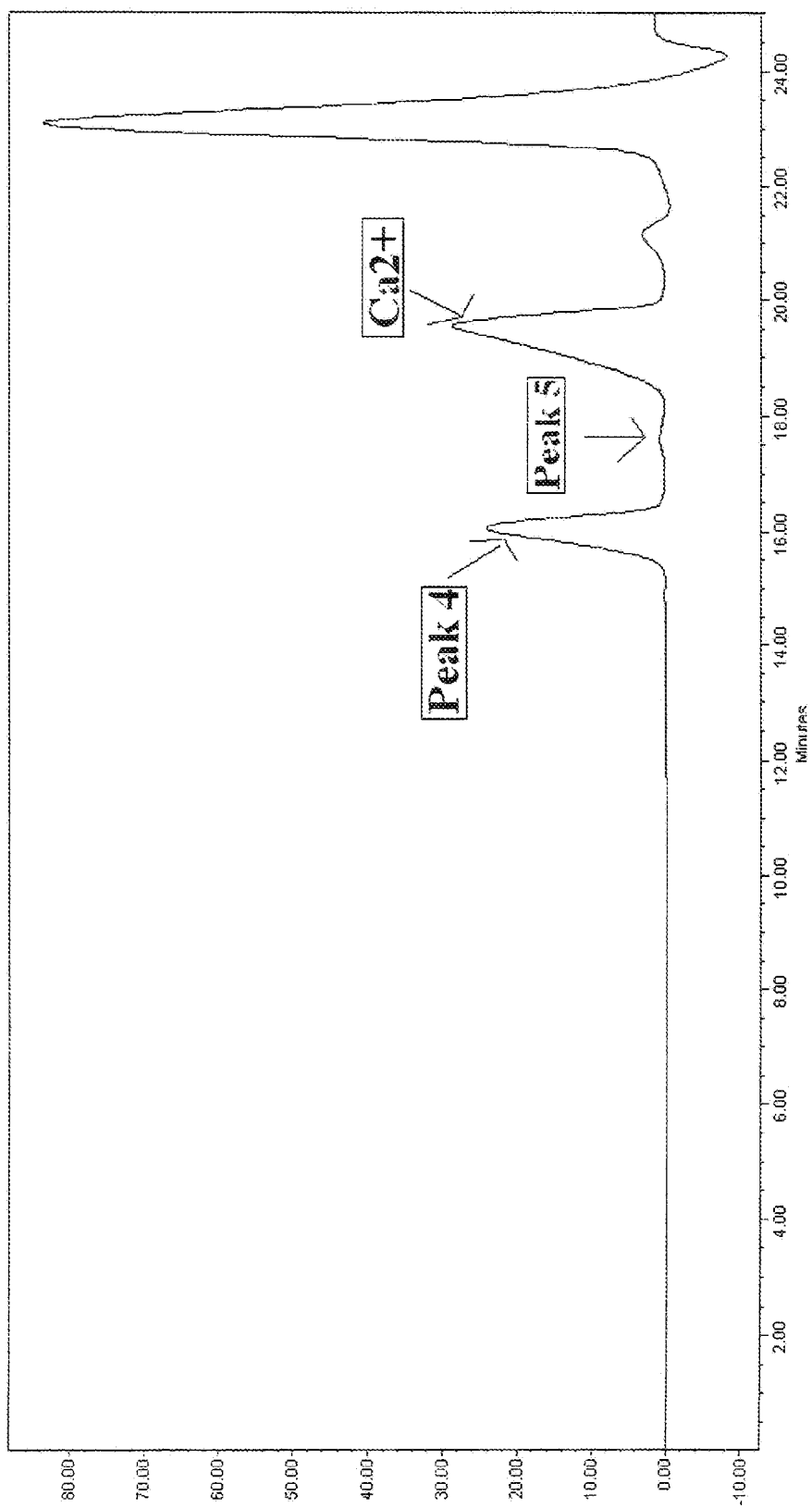


Figure 7

**ANTIPERSPIRANT ACTIVE COMPOSITIONS
HAVING SEC CHROMATOGRAM
EXHIBITING HIGH SEC PEAK 4 INTENSITY**

BACKGROUND

[0001] Antiperspirant salts, such as aluminum chlorohydrate (also called aluminum chlorohydrate polymeric salts and abbreviated here as “ACH”) and aluminum zirconium glycine salts (abbreviated here as “ZAG”, “ZAG complexes” or “AZG”), are known to contain a variety of polymeric and oligomeric species with molecular weights (MW) of 100-500,000. It has been clinically shown that, in general, the smaller the species, the higher the efficacy for reducing sweat.

[0002] In an attempt to increase the quality and quantity of smaller aluminum and/or zirconium species, a number of efforts have focused on (1) how to select the components of ACH and ZAG that affect the performance of these materials as antiperspirants and (2) how to manipulate these components to obtain and/or maintain the presence of smaller types of these components. These attempts have included the development of analytical techniques to identify the components. Size exclusion chromatography (“SEC”) or gel permeation chromatography (“GPC”) are methods frequently used for obtaining information on polymer distribution in antiperspirant salt solutions. With appropriate chromatographic columns, generally five distinctive groups of polymer species can be detected in commercial ACH and ZAG complexes appearing in a chromatogram as peaks 1, 2, 3, 4 and a peak known as “5.6”. Peak 1 is the larger Zr species (greater than 60 Angstroms). Peaks 2 and 3 are larger aluminum species. Peak 4 is smaller aluminum species (aluminum oligomers, or small aluminum cluster) and has been correlated with enhanced efficacy for both Al and Al/Zr salts. Peak 5, 6 is the smallest aluminum species. Various analytical approaches for characterizing the peaks of ACH and various types of ZAG actives are found in “Antiperspirant Actives—Enhanced Efficacy Aluminum-Zirconium-Glycine (AZG) Salts” by Dr. Allan H. Rosenberg (Cosmetics and Toiletries Worldwide, Fondots, D. C. ed., Hartfordshire, UK: Aston Publishing Group, 1993, pages 252, 254-256).

[0003] Attempts to activate antiperspirant salts to produce materials having improved efficacy have included developing processes for obtaining composition having large amounts of Peak 4 species. None of these efforts, however, have resulted in an antiperspirant composition having a composition with little or no Peak 3 and optionally little or no Peak 5.

SUMMARY OF INVENTION

[0004] The present invention provides for an antiperspirant active composition comprising an aluminum salt having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, exhibiting a SEC chromatogram having a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution, and optionally including zirconium.

[0005] The present invention also provides for a method of making an antiperspirant active composition that exhibits a SEC chromatogram having a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution comprising:

[0006] I) heating an aqueous solution containing an aluminum salt having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, optionally with a buffer agent, at

a temperature of about 50° C. to about 95° C. to reflux for a period of time of about 1 hour to about 5 hours to obtain an aluminum salt solution;

[0007] II) adding an aqueous solution of an inorganic base to obtain an aluminum salt solution having an OH:Al molar ratio of about 2:1 to about 2.6:1 to obtain a pH adjusted aluminum salt solution having a pH of about 2 to about 5; and

[0008] III) optionally adding an aqueous solution containing a zirconium compound to the pH adjusted aluminum salt solution to thereby obtain an aluminum-zirconium salt solution having a molar ratio of aluminum to zirconium of about 5:1 to about 10:1.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] The accompanying drawings, which are included to provide further understanding of the disclosure and are incorporated in and constitute a part of this specification, illustrate embodiments of the disclosure and, together with the description, serve to explain the principles of the disclosure.

[0010] FIG. 1 illustrates an SEC chromatogram of a prior art antiperspirant active composition.

[0011] FIG. 2 illustrates an SEC chromatogram having exclusive peaks 4 and 5 for an inventive product, Example 1, of the present invention.

[0012] FIG. 3 illustrates an SEC chromatogram having exclusive peak 4 for an inventive product, Example 2, of the present invention.

[0013] FIG. 4 illustrates an SEC chromatogram having exclusive peaks 4 and 5 for an inventive product, Example 3, of the present invention.

[0014] FIG. 5 illustrates an SEC chromatogram having exclusive peaks 4 and 5 for an inventive product, Example 4, of the present invention.

[0015] FIG. 6 illustrates an SEC chromatogram having exclusive peak 4 for an inventive product, Example 5, of the present invention.

[0016] FIG. 7 illustrates an SEC chromatogram having exclusive peaks 4 and 5 for an inventive product, Example 6, of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0017] As used throughout, ranges are used as a shorthand for describing each and every value that is within the range. Any value within the range can be selected as the terminus of the range.

[0018] The present invention is directed to an antiperspirant active composition having a high SEC peak 4 in aqueous solution. The composition is obtained by a stepwise procedure to neutralize aluminum chloride in a solution (optionally buffered) using inorganic bases. The antiperspirant active compositions obtained by this stepwise procedure include aluminum salts having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, the aluminum salt has a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution. The composition may optionally include zirconium.

[0019] Optionally, a buffer can be included. Buffers that can be used can be chosen from amino acids, glycine, and betaine. The buffer to aluminum molar ratio in certain embodiments can be about 0.1:1 to about 3:1. In another embodiment, the buffer to aluminum molar ratio is about

0.5:1 to about 2:1. In another embodiment, the buffer to aluminum molar ratio is about 1:1 to about 1.5:1.

[0020] The compositions may be made in a variety of ways involving a stepwise procedure to neutralize aluminum chloride in solution (optionally buffered) using inorganic basic salts. The procedure generally includes the step of heating an aqueous solution containing an aluminum chloride compound (optionally with a buffer agent) at a temperature of about 50° C. to about 95° C. to reflux for a period of time of about 1 hour to about 5 hours. The solution has a buffer agent to aluminum molar ratio of about 0.1:1 to about 3:1. To adjust the pH of the aluminum salt solution, an aqueous solution of an inorganic base is added to the heated solution to thereby obtain a pH adjusted aluminum salt solution having a hydroxide to aluminum molar ratio of about 1:1 to about 4:1, and a pH of about 2 to about 5. In one such embodiment, the hydroxide to aluminum molar ratio of about 2:1 to about 3:1. In another such embodiment, the hydroxide to aluminum molar ratio is about 2.1:1 to about 2.6:1. A zirconium salt may also be added to the pH adjusted aluminum salt solution. In one other such embodiment, the molar ratio of Al:Zr is about 5:1 to about 10:1. The antiperspirant active composition has a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution.

[0021] In one embodiment, an aqueous aluminum chloride salt solution is buffered with betaine monohydrate and held at about 50° C. to about 95° C. to reflux for a period time of about 1 to about 6 hours. To the heated solution, an aqueous solution of an inorganic base is added dropwise over a period of time of about 1 to about 3 hours while maintaining the aluminum-betaine solution at about 50° C. to about 95° C. to reflux. In one such embodiment, the solution has a betaine to aluminum molar ratio of about 1.1. In another such embodiment, the solution has a betaine to aluminum molar ratio of about 1.25.

[0022] In another embodiment, a $ZrOCl_2$ solution is added to the pH adjusted aluminum-betaine solution. In one such embodiment, the molar ratio of Al:Zr is about 8. In another such embodiment, the molar ratio of Al:Zr is about 7. In one other such embodiment, the molar ratio of Al:Zr is about 9.

[0023] In another embodiment, an aqueous aluminum chloride solution is buffered with glycine and held at about 50° C. to about 95° C. to reflux for a period time of about 1 to about 6 hours. To the heated solution, an aqueous solution of an inorganic base is added dropwise over a period of time of about 1 to about 3 hours while maintaining the aluminum-glycine solution at about 50° C. to about 95° C. to reflux. In one such embodiment, the solution has an aluminum to glycine molar ratio of about 0.4. In another such embodiment, the solution has an aluminum to glycine molar ratio of about 0.8.

[0024] In another embodiment, a $ZrOCl_2$ solution is added to the pH adjusted aluminum-glycine solution. In one such embodiment, the molar ratio of Al:Zr is about 8. In another such embodiment, the molar ratio of Al:Zr is about 7. In one other such embodiment, the molar ratio of Al:Zr is about 9.

[0025] For the above methods, the aluminum chloride salt and inorganic base may be obtained from a variety of sources. In one embodiment, the aluminum chloride salt includes aluminum trichloride, aluminum chlorohexahydrate and aluminum dichlorohydrate. In one such embodiment, the aluminum chloride salt is aluminum chlorohexahydrate.

[0026] In one embodiment, the inorganic base can be at least one base chosen from metal hydroxides, calcium

hydroxide, strontium hydroxide, barium hydroxide, metal oxides, calcium oxide, strontium oxide, and barium oxide.

[0027] The present invention provides for aluminum antiperspirant active compositions and/or aluminum-zirconium antiperspirant active compositions having high levels of low molecular weight Al and Zr species. As illustrated in FIGS. 2 to 7, the high levels of low molecular weight Al and Zr species is reflected in a SEC trace that has an intense Peak 4, low Peaks 1, 2, 3 and 5. The polymerization of the antiperspirant actives in aqueous solutions and the correspondent gelation process were followed by monitoring the molecular weight profile of the polyoxohalides in time by SEC. The relative retention time ("Kd") for each of these peaks varies depending on the experimental conditions, but the peaks remain relative to each other. Data for Tables in the examples was obtained using an SEC chromatogram using the following parameters: Waters®600 analytical pump and controller, Rheodyne® 7725I injector, Protein-Pak® 125 (Waters) column, Waters 2414 Refractive Index Detector. 5.56 mM nitric acid mobile phase, 0.50 ml/min flow rate, 2.0 microliter injection volume. Data was analyzed using Water® Empower software (Waters Corporation, Milford, Mass.). The concentration of the antiperspirant in solution does not affect the retention time in the machine.

[0028] The design of modern AP salts aims at actives with high levels of low molecular weight Al and Zr species, which is reflected in a SEC trace that has intense Peak 4 and low Peaks 1, 2, and 3. Throughout the present study, the levels of the species corresponding to these peaks are estimated based on the following ratios (or percentages):

$$f_{Pi} = \frac{Pi}{\sum Pj} \quad i = 1, 2, 3, 4, 5; \quad j = 2, 3, 4, 5$$

where f_{Pi} is the fraction of peak i, and Pi or Pj are the intensity of peaks Pi or Pj, respectively. The amount of low molecular weight Al species will be correlated with the fraction, f_{P4} , or percentage, $f_{P4} \times 100$, of SEC-Peak 4. In brief, a preferred antiperspirant salt would have a very low f_{P1} , f_{P2} , f_{P3} , and/or f_{P5} , and a high f_{P4} .

[0029] In certain embodiments, the ratio of Peak 4 to Peak 3 is at least 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 25, 30, 35, 40, 45, 50, 60, 70, 80, 90, 100, or any number up to infinity.

[0030] In one embodiment, an aluminum salt and/or aluminum-zirconium salt, in aqueous solution, exhibit a SEC profile wherein the SEC Peak 4 to Peak 3 intensity ratio is at least 7. In such embodiments, the percentage of SEC Peak 4 of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram is: at least 50%; at least 60%; at least 70%; at least 80%; at least 90%, or 95 to 100%. In another such embodiment, the SEC Peak 4 area is 100%.

[0031] In another embodiment, the aluminum salt and/or the aluminum-zirconium salt, in aqueous solution, exhibits a SEC profile wherein the SEC Peak 4 to Peak 3 intensity ratio is at least 7 and exhibits low percentage of SEC Peak 3. In such embodiments, the composition has the percentage of SEC Peak 3 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram is: less than about 10%; less than about 5%; less than about 2%; less than about 1%; less than about 0.9%; less than about 0.8%; less than about 0.7%; less than about 0.6%; of less than about 0.5%; less than about

0.4%; less than about 0.3%; less than about 0.2%; or less than about 0.1%. In another such embodiment, the composition has no SEC Peak 3 area.

[0032] In another embodiment, the aluminum salt and/or the aluminum-zirconium salt, in aqueous solution, exhibits a SEC profile wherein the SEC Peak 4 to Peak 3 intensity ratio is at least 7 and exhibits low percentages of SEC Peak 5. In such embodiments, the percentage of SEC Peak 5 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram is: less than about 30%; less than about 20%; less than about 10%; less than about 5%; or less than about 1%. In another such embodiment, the composition has no SEC Peak 5 area.

[0033] In another embodiment, the aluminum salt and/or the aluminum-zirconium salt, in aqueous solution, exhibits a SEC profile wherein the SEC Peak 4 to Peak 3 ratio is at least 7, and exhibits a low percentage of SEC Peak 1 and a low percentage of SEC Peak 2. In such embodiment, the percentage of SEC Peak 1 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram is: less than about 10%; a SEC Peak 1 area less than about 5%; less than about 2%; less than about 1%; less than about 0.9%; less than about 0.8%; of less than about 0.7%; less than about 0.6%; less than about 0.5%; less than about 0.4%; less than about 0.3%; less than about 0.2%; or less than about 0.1%. In another embodiment, the complex has no SEC Peak 1 area. In another embodiment, the percentage of SEC Peak 2 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram is: less than about 10%; less than about 5%; less than about 2%; less than about 1%; less than about 0.9%; less than about 0.8%; less than about 0.7%; less than about 0.6%; less than about 0.5%; less than about 0.4%; less than about 0.3%; less than about 0.2%; or less than about 0.1%. In another embodiment, the composition has no SEC Peak 2 area.

[0034] The aluminum antiperspirant active compositions and/or aluminum-zirconium antiperspirant active compositions may be used in a variety of antiperspirant products. If the product is used as a solid powder, the size of the particles of antiperspirant active of the invention can be any desired size, and may include conventional sizes such as in the range of 2 to 100 microns, with selected grades having an average particle size of 30-40 microns; finer sized grades having an average particle size distribution of 2-10 microns with an average size of about 7 microns as made by a suitable dry-grinding method; and micronized grades having an average particle size of less than about or equal to 2 microns, or less than about or equal to 1.5 microns.

[0035] The compositions of this invention may be used to formulate antiperspirants having improved efficacy. Such antiperspirants include solids such as sticks and creams (creams sometimes being included in the term "soft solid"), gels, liquids (such as are suitable for roll-on products), and aerosols. The forms of these products may be suspensions or emulsions. These antiperspirant actives can be used as the antiperspirant active in any antiperspirant composition.

[0036] Examples of suitable formulations include the following:

[0037] Sticks—Stick products may be made with conventional gelling agents such as stearyl alcohol and dibenzylidene sorbitol. A sample formulation is as follows:

40-55% (particularly 45%);
cyclomethicone (especially D5 cyclomethicone);
20-30% (particularly 21%);
stearyl alcohol 7-15% (particularly 10%);

talc 15-22% (particularly 22%); and
antiperspirant active of the invention in particle form; and
1-3% (particularly 2%) fragrance.

[0038] Roll Ons having a sample formulation:
45-65% (particularly 55%) cyclomethicone (especially D5 cyclomethicone);

0.1-10% (particularly 3%) cyclomethicone/dimethicone copolyol (such as Dow Corning 2-5185C) 10-25% (particularly 20%);

antiperspirant active of the invention in solution form (25-45% actives on an anhydrous basis in water);

5-30% (particularly 20%) water; and

1-3% (particularly 2%) fragrance.

[0039] Soft solids—Soft solids may be made with formulations described in U.S. Pat. No. 6,682,749. A sample formulation is as follows:

40-70% (particularly 50%) elastomer in cyclomethicone (KSG-15 from Shin-Etsu);

5-15% (particularly 6%) polyethylene (for example, beads having a density in the range of 0.91-0.98 g/cm³ and an average particle size in the range of 5-40 microns);

10-20% (particularly 15%) C 12-15 alkylbenzoate (FINSOLVTM TN from Finetex);

0.1-25% (particularly 22%) antiperspirant active of the invention in powder form;

1-15% (particularly 5%) dimethicone (particularly with a viscosity of 100 centistokes); and

1-3% (particularly 2%) fragrance.

[0040] Gels—Gels may be made with a variety of formulations such as:

5-50% (particularly 29%) cyclomethicone (particularly D5);

0.1-10% (particularly 3%) cyclomethicone/dimethicone copolyol (such as Dow Corning 2-5185C);

0-10% (particularly 5%) hydrogenated polyisobutene 250;

0-10% (particularly 5%) C12-15 alkylbenzoate (FINSOLVTM TN from Finetex);

0-10% (particularly 5%) dimethicone (particularly with a viscosity of 100 centistokes);

0.1-25% (particularly 20%) antiperspirant active of the invention in powder form or 10-25% (particularly 20%) of active in solution (25-45% actives on an anhydrous basis);

5-50% (particularly 30%) water; and

1-3% (particularly 2%) fragrance.

[0041] Note that in the explanation of the invention, where water is listed it is intended to count the contribution of the water present in the antiperspirant solution as part of the overall water content. Thus, water is sometimes listed as part of the actives solution or sometimes listed separately.

[0042] In one embodiment the refractive indices of the external and internal phases are matched within 0.005 to obtain a clear product.

[0043] Other formulations of interest include:

[0044] Formulation A:

0.5-2.5% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));

55-65% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));

1-10% PPG-3 myristyl ether;

10-25% antiperspirant active of the invention;

10-25% water; and

0.5-1.5% fragrance.

[0045] Formulation B:

1.0-3.0% dimethicone copolyol (for example, Dow Corning 2-5185C (48%)) 40-60% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 1-5% cyclomethicone (in addition to that found in the elastomer);
 4-12% PPG-3 myristyl ether;
 15-30% antiperspirant active of the invention;
 15-35% water; and
 0.5-1.5% fragrance.

[0046] Formulation C:

1.0-3.0% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 1-10% hydrogenated polyisobutene (for example, FancoI™, Polyiso 250);
 40-55% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 3-8% PPG-3 myristyl ether;
 15-20% antiperspirant active of the invention;
 20-30% water; and
 1.0-3.0% fragrance.

[0047] Formulation D:

1.0-3.0% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 40-60% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 3-8% PPG-3 myristyl ether;
 15-30% antiperspirant active of the invention;
 15-30% water;
 0.5-1.5% fragrance; and
 1-10% diethylhexyl naphthalate

[0048] Formulation E:

0.5-2.5% dimethicone copolyol (for example, Dow Corning 2-5185C (48%));
 60-70% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 7-10% antiperspirant active of the invention;
 25-35% water;
 1-10% methylpropylene diol (MPDiol); and
 0.5-1.5% fragrance

[0049] Formulation F:

1.0-3.0% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 6-10% hydrogenated polyisobutene (for example, FANCOL™ Polyiso 250);
 35-45% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 6-10% PPG-3 myristyl ether;
 40-50% antiperspirant active of the invention as 43% active in water no additional water; and
 0.5-1.0% fragrance.

[0050] Formulation G

0.1-0.6% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 4-7% hydrogenated polyisobutene (for example, FANCOL™ Polyiso 250);

40-50% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 4-7% PPG-3 myristyl ether;
 40-50% antiperspirant active of the invention as 43% active in water no additional water; and
 0.5-1.0% fragrance.

[0051] Formulation H:

0.5-2.0% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 1-7% hydrogenated polyisobutene (for example, FANCOL™ Polyiso 250);
 40-50% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 45-55% antiperspirant active as 43% active of the invention in water no additional water; and
 0.5-1.5% fragrance.

[0052] Formulation I:

2-7% dimethicone copolyol (for example, Dow Corning 2-5185 C (48%));
 0.1-1% Oleath-20 1-5% C12-15 alkyl benzoate (FINSOLV™ TN);
 15-25% elastomer in cyclomethicone (for example, DC-9040 from Dow Corning Corporation (Midland, Mich.) or KSG-15 from Shin-Etsu Silicones of America (Akron, Ohio));
 15-25% antiperspirant active of the present invention;
 15-30% water; and
 0.5-1.5% fragrance

[0053] The cosmetic composition according to the present invention can be packaged in conventional containers, using conventional techniques. Where a gel, cream or soft-solid cosmetic composition is produced, the composition can be introduced into a dispensing package (for example, conventional packages for gels with glide on applicators, jars where the gel or cream is applied by hand, and newer style packages having a top surface with pores) as conventionally done in the art. Thereafter, the product can be dispensed from the dispensing package as conventionally done in the art, to deposit the active material, for example, on the skin. For sticks, sprays, aerosols and roll-ons the compositions can be placed in a conventional types of container (with the inclusion of propellants in aerosols). This provides good deposition of the active material on the skin.

[0054] Compositions of the present invention can be formulated as clear, translucent or opaque products. A desired feature of the present invention is that a clear, or transparent, cosmetic composition, (for example, a clear or transparent deodorant or antiperspirant composition) can be provided. The term clear or transparent according to the present invention is intended to connote its usual dictionary definition; thus, a clear liquid or gel antiperspirant composition of the present invention allows ready viewing of objects behind it. By contrast, a translucent composition, although allowing light to pass through, causes the light to be scattered so that it will be impossible to see clearly objects behind the translucent composition. An opaque composition does not allow light to pass there through. Within the context of the present invention, a gel or stick is deemed to be transparent or clear if the maximum transmittance of light of any wavelength in the range 400-800 nm through a sample 1 cm thick is at least

35%, or at least 50%. The gel or liquid is deemed translucent if the maximum transmittance of such light through the sample is between 2% and less than about 35%. A gel or liquid is deemed opaque if the maximum transmittance of light is less than about 2%. The transmittance can be measured by placing a sample of the aforementioned thickness into a light beam of a spectrophotometer whose working range includes the visible spectrum, such as a Bausch & Lomb Spectronic 88 Spectrophotometer. As to this definition of clear, see European Patent Application Publication No. 291,334 A2. Thus, according to the present invention, there are differences between transparent (clear), translucent and opaque compositions.

EXAMPLES

Comparative Example

[0055] A 0.72 M $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (18 mmol) is held at 90° C. and stirred vigorously. To this solution, a 4 N $\text{Ca}(\text{OH})_2$ (20 mmol) is added dropwise over a 1 hour 30 minute period. A ratio of OH:Al of 2.22 is employed in an attempt to prevent the formation of larger unwanted Al species. The pH after the reaction was 2.36 due to the low OH:Al ratio. The SEC chromatogram, illustrated in FIG. 1, exhibits multiple peaks including, SEC-Peak 4, and SEC-Peak 5 indicating multiple Al species are present in solution. At a retention time of approximately 15.5 minutes, SEC-Peak 3 is observed.

[0056] Also for comparison, 10% solutions are prepared from commercially available antiperspirants. The solutions are prepared by adding 1 g of antiperspirant to 9 g of water and mixing. The antiperspirant salts were Reach™ 103, Reach™ 301 from Reheis, and Summit™ Z576 from Summit Research Labs.

Example 1

[0057] A 0.72M $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (18 mmol) is buffered with 20 mmol betaine monohydrate, held at 90° C., and stirred vigorously. To this solution, a 4 N $\text{Ca}(\text{OH})_2$ (20 mmol) is added dropwise over a 1 hour 30 minute period. A ratio of OH:Al of 2.22 is employed in an attempt to prevent the formulation of large Al species. The pH after the reaction is 2.56 due to the low OH:Al ratio. As illustrated in FIG. 2, the SEC chromatogram shows exclusively SEC-Peak 4 and SEC-Peak 5, which are known to represent active anti-perspirant species. Substantially no SEC-Peak 3 species is observed at a retention time of approximately 15.5 minutes.

Example 2

[0058] A 0.72M $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (16.26 mmol) was buffered with 20 mmol anhydrous betaine, held at 90° C., and stirred vigorously. To this solution, a 4 N $\text{Ca}(\text{OH})_2$ (20 mmol) was added dropwise over a 2 hour period. A ratio of OH:Al of 2.46 was employed in an attempt increase the final pH and to reduce SEC-Peak 5 species. Because a higher OH:Al ratio was used, the addition of the base was extended over a 2 hour period. The pH after the reaction was 4.8. As illustrated in FIG. 3, the SEC chromatogram indicated that the solution contained exclusively SEC-Peak 4 310 antiperspirant active

species. Substantially no SEC-Peak 3 species was observed at a retention time of approximately 15.5 minutes.

Example 3

[0059] A small portion of the solution from Example 2 is taken to determine the effects of Zr on the peak distribution. $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ is added to achieve a molar ratio of 8:1, Al:Zr. The pH after adding Zr reduces to 3.7. As shown in FIG. 4, the SEC chromatogram of this zirconium, aluminum solution shows two notable features. Firstly, the SEC-Peak 4 remains the predominate peak, and the SEC-Peak 5 intensity increased to 1%—as expected by the reduced pH. Secondly, the SEC chromatogram does not show any peaks with retention time of 12.5 minute indicating the absence of undesirable Zr polymer species. The absence of this SEC-Peak indicates that the pure SEC-Peak 4 solution of Example 2 did not promote the aggregation of Zr into larger, less efficacious species. Also substantially no SEC-Peak 3 species is observed at a retention time of approximately 15.5 minutes.

Example 4

[0060] A 0.5M $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (25 mmol) is buffered with 31.25 mmol glycine, held at 95° C., and stirred vigorously. To this buffered solution, a 1.0 N $\text{Ca}(\text{OH})_2$ (31.25 mmol) is added dropwise over a 1 hour period. A ratio of OH:Al of 2.5 is employed in an attempt to increase the final pH and to reduce SEC-Peak 5 species. The pH after the reaction is 4.52. The SEC chromatogram shown in FIG. 5 exhibits primarily SEC-Peak 4 and a smaller SEC-Peak 5 (520). Substantially no SEC-Peak 3 species is observed at a retention time of approximately 15.5 minutes.

Example 5

[0061] A 0.5M $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (25 mmol) is buffered with 62.5 mmol glycine, held at 95° C., and stirred vigorously. To this buffered solution a 1.0 N $\text{Ca}(\text{OH})_2$ (31.25 mmol) is added dropwise over a 1 hour period. A ratio of OH:Al of 2.5 is employed in an attempt to increase the final pH and to reduce the SEC-Peak 5 species. The pH after the reaction is 4.52. The SEC chromatogram shown in FIG. 6 exhibits exclusively SEC-Peak 4 and no SEC-Peak 5. Substantially no SEC-Peak 3 species is observed at a retention time of approximately 15.5 minutes.

Example 6

[0062] A small portion of the solution from Example 5 is taken to determine the effects of Zr on the peak distribution. $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ is added to achieve an Al:Zr molar ratio of 8:1. The pH after adding Zr reduces to 3.3. The SEC chromatogram shown in FIG. 7 exhibits primarily SEC-Peak 4 and substantially no SEC-Peak 5 (720). This data indicates that the pure SEC-Peak 4 solution of Example 5 does not promote the aggregation of Zr into larger, less efficacious species. Also substantially no SEC-Peak 3 species is observed at a retention time of approximately 15.5 minutes.

[0063] Comparison of the Examples

Example	Ratio OH:Al	pH	Comparable ACH Solution	Relative Peak Distribution after Reaction (%)				
				Peak 2	Peak 3	Peak 4	Peak 5	Peak 4/ Peak 3
Summit™ Z576				3.1	34.1	40	22.6	1.2
Reach™ 103	n/a		10% ACH		63	34	3	0.54
Reach™ 301	n/a		10% ACH	7	65	12	16	0.18
Comparative	2.2	2.36	21% ACH	0	42	42	16	1.0
Example 1	2.2	2.56	21% ACH	0	0	75	25	∞
Example 2	2.5	4.8	22% ACH	0	0	100	0	∞
Example 3	2.5	3.7	22% ACH	0	0	99	1	∞
Example 4	2.5	4.5	4% ACH	0	0	93	7	∞
Example 5	2.5	4.52	4% ACH	0	0	100	0	∞
Example 6		3.32	4% ACH	0	0	98	2	∞

What is claimed:

1. An antiperspirant active composition comprising an aluminum salt having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, exhibiting a SEC chromatogram having a SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution.

2. The antiperspirant active of claim 1, wherein the composition further comprises zirconium.

3. The antiperspirant active of claim 1, wherein the aluminum salt is buffer free and has an OH to Al ratio of about 2:1 to about 2.6:1.

4. The antiperspirant active of claim 1 further comprising a buffer, wherein a molar ratio of buffer to aluminum is about 0.1:1 to about 3:1.

5. The antiperspirant active of claim 4, wherein the buffer is at least one buffer chosen from an amino acid, glycine, and betaine.

6. The antiperspirant active of claim 1, wherein the zirconium is present and a molar ratio of aluminum to zirconium is about 5:1 to about 10:1.

7. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 4 area of at least 50% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

8. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 4 area of 95 to 100% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

9. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 3 area of less than about 10% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

10. The antiperspirant active composition of claim 1, wherein the composition has no SEC Peak 3 area in the SEC chromatogram.

11. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 5 area of less than about 30% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

12. The antiperspirant active composition of claim 1, wherein the composition has no SEC Peak 5 area in the SEC chromatogram.

13. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 1 area of less than

about 10% and a SEC Peak 2 area of less than about 10% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

14. The antiperspirant active composition of claim 1, wherein the composition has a SEC Peak 4 area of 95 to 100%, no SEC Peak 3 area, and no SEC Peak 5 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

15. A method of making an antiperspirant active composition comprising:

I) heating an aqueous solution containing an aluminum salt having an aluminum to chloride molar ratio of about 0.3:1 to about 3:1, optionally with a buffer agent, at a temperature of about 50° C. to about 95° C. to reflux for a period of time of about 1 hour to about 5 hours to obtain an aluminum salt solution:

II) adding an aqueous solution of an inorganic base to obtain an aluminum salt solution having an OH:Al molar ratio of about 2:1 to about 2.6:1 to obtain a pH adjusted aluminum salt solution having a pH of about 2 to about 5; and

III) optionally adding an aqueous solution containing a zirconium compound to the pH adjusted aluminum salt solution to thereby obtain an aluminum-zirconium salt solution having a molar ratio of aluminum to zirconium of about 5:1 to about 10:1.

16. The method of claim 15, wherein the buffer is present in a molar ratio of buffer to aluminum is about 0.1:1 to about 3:1.

17. The method of claim 15, wherein the buffer agent is at least one buffer chosen from an amino acid, glycine, and betaine.

18. The method of claim 15, wherein the inorganic base includes at least one member chosen from metal hydroxides, calcium hydroxide, strontium hydroxide, barium hydroxide, metal oxides, calcium oxide, strontium oxide, and barium oxide.

19. The method of claim 15, wherein the aluminum chloride compound is chosen from aluminum trichloride, aluminum chlorohexahydrate, and aluminum dichlorohydrate.

20. The method of claim 15, wherein the composition further comprises zirconium.

21. The method of claim 20, wherein the zirconium is $ZrOCl_2 \cdot 8H_2O$.

22. The method of claim 15, wherein the antiperspirant active composition exhibits a SEC chromatogram having a

SEC Peak 4 to Peak 3 intensity ratio of at least 7 and a Peak 4 intensity greater than a Peak 5 intensity in aqueous solution.

23. The method of claim **22**, wherein the antiperspirant active composition has a SEC Peak 4 area of at least 50% of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

24. The method of claim **22**, wherein the antiperspirant active composition has a SEC Peak 4 area of 95 to 100% of the total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

25. The method of claim **22**, wherein the antiperspirant active composition has a SEC Peak 3 area of less than about 10% of the total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

26. The method of claim **22**, wherein the antiperspirant active composition has no SEC Peak 3 area.

27. The method of claim **22**, wherein the antiperspirant active composition has a SEC Peak 5 area of less than about 30% of the total area of Peaks 1, 2, 3, 4, 5, and 6.

28. The method of claim **22**, wherein the antiperspirant active composition has no SEC Peak 5 area.

29. The method of claim **22**, wherein the antiperspirant active composition has a SEC Peak 1 area of less than about 10% and a SEC Peak 2 area of less than about 10% of the total area of Peaks 1, 2, 3, 4, 5, and 6.

30. The method of claim **22**, wherein the composition has a SEC Peak 4 area of 95 to 100%, no SEC Peak 3 area, and no SEC Peak 5 area of a total area of Peaks 1, 2, 3, 4, 5, and 6 in the SEC chromatogram.

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