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OXIDATIVE FIXING COMPOSITION

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This invention relates to an improved fixing composition suitable for use in processes involving changing the configuration of keratin-containing substances. More particularly the invention concerns the provision of an improved composition for use as a fixing agent in the cold permanent waving of hair.

It is known that alkali metal bromates in aqueous solution are effective fixing agents for use in reestablishing disulfide linkages in reduced keratin and that their effectiveness in permanently fixing a wave imparted to hair is increased when they are applied to the hair in aqueous solution with acid buffers adapted to counteract the alkalinity of residual waving lotion remaining on the hair. It is to be borne in mind that commercially available alkali metal bromates are not buffers and have but little power to neutralize residual alkaline waving lotion. Acid reacting buffers such as sodium dihydrogen orthophosphate, acid reacting mixtures thereof with disodium hydrogen orthophosphate, disodium acid citrate, acid reacting salts of tartaric acid and other buffering agents, 2% solutions of which at 25° C. exhibit pH values from about 4 to not substantially more than 6.5, have been considered in the preparation of such buffered fixing compositions. Since the alkali metal bromates are oxidizing agents, inorganic buffering agents such as sodium dihydrogen orthophosphate and the acid reacting mixtures thereof with disodium hydrogen orthophosphate are preferred for reasons of safety in storage and handling.

It is also known that ordinary commercial forms of alkali metal bromate and acid buffering agent contain moisture and that when the ingredients are mixed the bromate is unstable in the presence of moisture and the acid reacting buffer, unpleasantly acrid and corrosive fumes of bromine being evolved. Obviously the packaging and storage of such mixtures in powdered form presents serious problems, especially to the manufacturers of present-day hair waving kits which customarily contain an envelope of powdered fixing agent.

It is an object of the present invention to provide a composition of alkali metal bromate and acid buffering agent which has improved stability towards decomposition. A further object is to provide a composition of said ingredients which tolerates the presence of moisture in moderate amounts without undesirable caking or decomposition. Another object is to provide a free-flowing fixing agent of improved stability for use in reestablishing disulfide linkages in reduced

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keratin. Other objects will be apparent from the following description taken in conjunction with the illustrative examples.

I have found that compositions of desirable stability and effectiveness are obtainable by producing the mechanical mixture of bromate and acid buffering constituents from materials which are in particulate form and which have been dried to substantially bone dryness, that is to a moisture content not substantially in excess of 0.01%. Thus an undried mixture of 100 parts potassium bromate and 75 parts sodium dihydrogen orthophosphate (pH of mixture in 2% aqueous solution=4.6) having an average moisture content of about 0.06% evolved a bromine odor after 7 hours' storage in a closed container at 100° F. In the case of a comparable mixture dried to bone dryness, no bromine odor was detected after 20 days' storage.

While such bone dry mixtures possess the desired stability under normal conditions of storage, they have been found to possess sufficient hygroscopic character to render them more or less undesirable for distribution in localities where normal atmospheric humidity is relatively high and absorption of moisture from the atmosphere is likely.

I have found that even such disadvantages can be overcome, that the homogeneous incorporation in the bromate and acid buffer mixture of finely divided alkali metal hydroxide or carbonate, such as sodium and potassium hydroxides and carbonates, as well as corresponding alkalis of other alkali metals, herein referred to generically as "alkali," markedly stabilizes the bromate against decomposition, and that moderate amounts of moisture not substantially greater than 2 per cent of the composition can be tolerated without undesirable decomposition of the bromate. I prefer that the alkali be 60 mesh or smaller in particle size (Standard Sieve Series) and more finely divided than either the bromate or buffer to insure contact of alkali with each particle of other constituent of the composition.

The amount of finely divided alkali which must be used to achieve the advantages of my invention can vary, the minimum amount depending in large measure on the moisture content of the system. Thus in a bone dry mixture of bromate and acid buffer, no alkali is necessary, provided substantial bone dryness is maintained. In a corresponding mixture in which the moisture content is about 1%, the incorporation of 1/4% of finely divided alkali, based on the bromate, will

result in a marked increase in the stability of the bromate against decomposition during storage. If the moisture content of the mixture is of the order of 0.7 to 1%, the incorporation of 1-2% or more of the alkali, based on bromate, is preferable. While the addition of larger amounts of alkali up to 5%, for example, has some beneficial effect on the stability of the bromate in the mixture of ingredients, especially in the upper range of permissible moisture, care should be taken to avoid the incorporation of an amount of alkali which will impart to a 2% aqueous solution of the composition a pH value in excess of 7, thereby avoiding reduction in the efficiency of the solution of the composition as a fixing agent. Obviously the maximum tolerance for alkali will vary depending on the acid properties of the buffer employed.

The relative proportions of bromate and acid buffer are not critical and wide variations of these ingredients are permissible in the compositions of my invention. Compositions containing from about 25% to about 150% by weight of acid buffer, based on the bromate, are operative, but optimum effectiveness in fixing performance is achieved when the usage of acid buffer amounts to about 50 to about 100% by weight of the bromate.

The most practical physical form in which the composition of my invention can be marketed is as a free flowing mechanical mixture of constituents in particulate form. However, the homogeneous compositions can be compressed into suitable tablet form and marketed as such, if desired.

The invention will be more fully understood from the following detailed examples which are submitted as exemplary of the compositions embraced in the appended claims.

Example 1.—A mechanical mixture of 100 parts bone dry powdered potassium bromate and 75 parts bone dry powdered potassium dihydrogen orthophosphate was prepared. This mixture, when dissolved in water to form a 2% aqueous solution, had a pH value of 4.6. The bone dry mixture was stable after 20 days' storage in a closed container at 80° F.

Example 2.—A homogeneous mechanical mixture was prepared from 100 parts powdered commercial potassium bromate, 50 parts powdered commercial sodium dihydrogen orthophosphate, and 1 part powdered commercial sodium hydroxide. The average moisture of the composition was 0.7% and after storage in a closed container at 80° F. for 29 days no bromine odor could be detected.

Example 3.—A homogeneous mechanical mixture was prepared from 98 parts sodium bromate, 75 parts sodium dihydrogen orthophosphate, and 2 parts of sodium carbonate. The mixture had an average moisture content of 0.3%, and no bromine odor due to decomposition of bromate was evident after storage for 54 days in a closed container at 100° F. A 2% aqueous solution of the mixture at 25° C. had a pH of 5.8.

Example 4.—A homogeneous mechanical mixture was prepared from 100 parts powdered potassium bromate, 75 parts powdered sodium dihydrogen orthophosphate and 0.5 part powdered potassium carbonate. By analysis the mixture had an average moisture content of 0.3%, and no bromine odor due to decomposition of bromate could be detected after 152 days' storage in a closed container at 100° F.

Example 5.—A homogeneous mechanical mix-

ture was prepared from 100 parts powdered potassium bromate, 75 parts powdered sodium dihydrogen orthophosphate, and 1 part powdered sodium carbonate. The mixture had an average moisture content of 0.5%, and did not evolve bromine vapor after 105 days' storage in a closed container within a temperature range of 100 to 120° F.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

1. A fixing composition adapted for reestablishing disulfide linkages in the treatment of reduced keratin, comprising essentially a homogeneous mechanical mixture of finely divided particles of alkali metal bromate oxidizing agent, acid reacting buffering agent and not more than 5% finely divided alkali, based on bromate, the average particle size of the alkali being less than the average particle size of the other ingredients, the amount of alkali being sufficient to stabilize said bromate against decomposition in the presence of moisture and acid reacting substances, but not more than an amount which will impart a pH value of 7 to a 2% aqueous solution of the mixture, the average moisture content of the mechanical mixture being not substantially greater than 2%.

2. A fixing composition adapted for reestablishing disulfide linkages in the treatment of reduced keratin, comprising essentially a homogeneous mechanical mixture of finely divided particles of alkali metal bromate oxidizing agent, acid reacting buffering agent and an amount of finely divided alkali which is at least ¼% by weight of said bromate, the average particle size of the alkali being less than the average particle size of the other ingredients, and in amount sufficient to stabilize said bromate against decomposition in the presence of moisture and acid reacting substances but not more than an amount which will impart a pH value of 7 to a 2% aqueous solution of said mixture, the average moisture content of said mechanical mixture being not substantially greater than 2%.

3. A fixing composition adapted for reestablishing disulfide linkages in the treatment of reduced keratin, comprising essentially a mechanical mixture in fine particulate form of alkali metal bromate oxidizing agent, an acid reacting buffering agent which in 2% aqueous solution exhibits a pH value from about 4 to about 6.5 and an amount of finely divided alkali which is at least ¼% by weight of said bromate, the average particle size of the alkali being less than the average particle size of the other ingredients, and in amount sufficient to stabilize said bromate against decomposition in the presence of moisture and acid reacting substances but not more than an amount which will impart a pH value of 7 to a 2% aqueous solution of said mixture, the average moisture content of said mechanical mixture being not substantially greater than 2%.

4. A fixing composition adapted for reestablishing disulfide linkages in the treatment of reduced keratin, comprising essentially a mechanical mixture in fine particulate form of alkali metal bromate oxidizing agent, an inorganic acid reacting buffering agent which in 2% aqueous solution exhibits a pH value from about 4 to about 6.5 and an amount of finely divided alkali which is at least ¼% by weight of said bromate, the average particle size of the alkali being less than the average particle size of the other ingredients, and in amount sufficient to stabilize said bromate against decomposition in

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the presence of moisture and acid reacting substances but not more than an amount which will impart a pH value of 7 to a 2% aqueous solution of said mixture, the average moisture content of said mechanical mixture being not substantially greater than 2%.

5. The composition of claim 4 in which the alkali is alkali metal hydroxide.

6. The composition of claim 4 in which the alkali is alkali metal carbonate.

7. The composition of claim 4 in which the inorganic acid reacting buffering agent is sodium dihydrogen orthophosphate and in which its amount in the composition is from about 50% to about 100%, based on the alkali metal bromate oxidizing agent.

8. The composition of claim 4 in which the alkali metal bromate oxidizing agent is potassium bromate, the inorganic acid reacting buffering agent is alkali metal dihydrogen orthophosphate, and the alkali is sodium carbonate.

9. The composition of claim 4 in which the alkali metal bromate oxidizing agent is potassium bromate, the inorganic acid reacting buffering agent is sodium dihydrogen orthophosphate in an amount from about 50% to about 100% based on the potassium bromate, and in which the alkali is sodium carbonate in an amount from 1 to 2% based on the potassium bromate, the average moisture content of the mixture being not substantially greater than 1.0%.

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