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<b>(21) International Application Number:</b> PCT/US94/09185		<b>(74) Agents:</b> KANAGY, James, M. et al.; SmithKline Beecham Corporation, Corporate Intellectual Property, UW2220, 709 Swedeland Road, P.O. Box 1539, King of Prussia, PA 19406-0939 (US).	
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<b>(71) Applicant (for all designated States except US):</b> SMITHKLINE BEECHAM CORPORATION [US/US]; Corporate Intellectual Property, UW2220, 709 Swedeland Road, P.O. Box 1539, King of Prussia, PA 19406-0939 (US).			
<b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> GALLOPO, Andrew, Robert [US/US]; 133 Wessington Avenue, Garfield, NJ 07026 (US). MAZZANOBILE, Salvatore [US/US]; 264 Crocker Place, Haworth, NJ 07641 (US).			

**(54) Title:** TOOTH WHITENING PREPARATIONS

**(57) Abstract**

This invention relates to compositions for whitening teeth and dental prostheses using a sequestering agent and a reducing agent.

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## Tooth Whitening Preparations

### Scope of the Invention

This is a continuation-in-part of application Serial No. 08/107,477 filed August 13, 1993.

5 This invention relates to a system for whitening natural teeth and dental prostheses. The whitening is achieved through the use of a sequestering agent and a reducing agent such as vitamin C. Any orally acceptable presentation can be utilized in this invention.

### Area of the Invention

10 Several factors contribute to enamel discoloration but the three main factors are believed to be: i) formation of plaque and tartar matrices on the tooth surface which then entrapment stains, ii) ingestion of certain drugs during tooth formation, and iii) discoloration due to oral cavity traumatization following which blood break-down products seep into the mineralized area of the teeth during enamel formation. This 15 invention is primarily concerned with the first factor or cause of tooth discoloration, that is the natural stain which accumulates on teeth.

Over-the-counter teeth whitening preparations have been developed to address the cosmetic preference of many to restore luster to tooth enamel discolored by surface entrapped materials; the term lightening may also be used in conjunction with the 20 advertising and sale of these products. While all dentifrices and mouthwashes contain some cleaning and polishing agents some enamel deposits may become to intractable to be fully removed by these agents under normal use conditions. Also these preparations may not be formulated with the amount or type of agent required to fully remove the amount of stains and discoloration which build up due to excessive exposure to the 25 staining agent. For example, smokers often develop discolored enamel because the tars and particulate in exhaled cigarette smoke collect on the teeth. And a number of comestibles can stain or discolor tooth enamel, tea being one example of a beverage where the tannins in the tea deposit on the tooth enamel. Some medicinal agents may cause staining or discoloration via entrapment, though this is not a usual common cause 30 of this type of staining.

Three approaches to enamel whitening are currently in general use. They are based on using abrasives, employ oxidizing agents or utilize a hydrolytic entity to break down the staining material, e.g. enzyme-based products.

One approach is basically a physical abrading of the stain to effect removal. 35 Harsher abrasives, which might also be called polishing agents, than what are normally used in tooth paste preparations are employed in this approach. Most if not all of these

preparations are toothpastes, gels or powder dentifrices as they require close contact with the teeth. And brushing or similar scrubbing or polishing action is required as a complement to successful stain removal. Examples of such products are Smokers Topol made by Topol-Dep Corporation and marketed to smokers and tea drinkers as a means 5 for removing stains caused by smoking and drink tea or similar beverages.

Oxidizing agent represent the most widely distributed and utilized agent in oral preparations marketed as enamel whiteners in the U.S. All of these products are pastes or gels. Urea peroxide, hydrogen peroxide or calcium peroxide are most often found in these products. Currently there are more than thirty such products marketed over the 10 counter in the U.S. How these oxidizing agents work remains a mystery but speculation is that they the an hydroxyl radical is released in the breakdown of the peroxide, which radical breaks down the plaque/stain complex to a product which can be flushed away or removed by an abrasive. These treatments require quite a bit of time to achieve good results; one-and-a-half to eight days, or 2-3 months depending on the peroxide source 15 and its concentration.

Recently catalytic systems have come back into favor and have been packaged and market through retail outlets in parallel with other oral care products. Proteolytic enzymes are the catalyst of choice, particularly papain. A second active such as a citric acid salt has been used by at least one manufacturer. These products are presented in a 20 paste or gel. They claim to whiten teeth by removing the plaque and calculus which have entrapped the stain.

This invention provides a unique alternative. A sequestering agent is combined with a reducing agent to effect stain removal and whiten tooth enamel.

### Summary of the Invention

25 This invention comprises a composition for reducing or removing surface deposited stains from natural teeth and dental prostheses comprising an effective amount of a sequestering agent and an orally acceptable reducing agent, particularly vitamin C or a pharmaceutical acceptable salt thereof in a carrier.

In addition this invention relates to a means for reducing or removing surface 30 deposited stains from natural teeth and dental prostheses which method comprises contacting teeth or dental prostheses which have surface stains with a composition comprising an effective amount of a sequestering agent and an orally acceptable reducing agent, particularly vitamin C or a pharmaceutical acceptable salt thereof, in a carrier for a time sufficient to remove at least a major amount of said stain.

35 This invention also relates to an article of manufacture comprising a first article which contains an orally acceptable reducing agent suitable and a second article which contains a sequestering agent in a form suitable for combining with first article for use in

whitening teeth. Also included is the product prepared by combining the first and second article.

### Detailed Embodiments of the Invention

The focus of this invention is on that of removing stains which are adhered to, or entrapped in materials on, the surface of teeth. Native teeth and dental prostheses, e.g., false teeth, can be treated with this invention. It is not directed to those situations where discoloration is due to materials integrated into the teeth such as happens when blood break-down products are incorporated into enamel during the mineralization process or where the likes of tetracycline cause developing tooth enamel to become discolored. The presumption behind this invention is that the native tooth material is white but that this whiteness has become covered up with surface debris which, when removed, will reveal the underlying natural color of the tooth. One can use this invention in those situations where there is also staining integral to the teeth but where surface deposits have further stained the teeth. Put another way, it is not contemplated that this preparation and process will increase teeth whiteness over what it already was before stains became deposited on them but will remove surface debris to reveal the underlying native color of the teeth.

This invention can also be used to prevent build-up of surface attached stains. In essence this is a matter of removing periodically small amounts of materials which are in fact stains but are so small or so thinly layered on the teeth as to be imperceptible under normal day-to-day conditions such as are represented by one's usual business or social endeavors. Deposit prevention per se may not be involved here but that has not been ruled out. That is, treating teeth with these preparations may prevent the attachment of stains or the entrapment of stains in some fashion. Whether one or both of these phenomena is going on is not so important as the fact that regular use of these preparations can achieve a state where the user does not perceive that her or his teeth are stained. And regular use can prevent a reoccurrence of that condition.

The term stain or staining is used interchangeable with discoloration and generally means that the surface of the enamel has taken on some unwanted or unnatural coloration distinct from the color of the underlying enamel. These words are intended to be given the same meaning as here as would be accorded to them in their contemporary usage in the oral and dental care arts.

Without being limited in any fashion, it is believed this invention works by somehow acting on the matrices entrapping the stain, the plaque, tartar or calculus, rather than acting on the stain itself. It is believed the matrices are modified or broken down in some fashion so as to permit the staining elements to be physically removed by abrasive or washing means. It may be that the reducing agent also has an affect on the stain's

coloration in some fashion or to some degree such that it is not perceived as being present, and thus the teeth appear to be lighter in color though the overlay of material is still in part or in whole covering the enamel. While this is possible, it is speculative.

Use in humans is primarily what is contemplated by this invention. However, 5 these preparations can be used in other species as well, for example in pet care products.

The active components of this invention comprise one or more sequesterants and an orally acceptable reducing agent.

Sequestering agent or sequesterant is used here in its normal and usual definition. Chelating agent is often used interchangeably with sequestering agent. And for the 10 purposes of this invention it is intended that they be interchangeable. Since these preparations will be used in the oral cavity they must not be toxic in the oral cavity or cause untoward or deleterious affects if consumed, e.g. swallowed, in the normal course of use. A partial list of sequestering agents which can be used in the context of this invention are: alkali metal salts of tripolyphosphate, pyrophosphate, and 15 hexametaphosphate; ethylenediamine tetracetic acid and its alkali metal salts; or citric acid and its alkali metal salts. Sodium tripolyphosphate is the most preferred sequesterant. Numerous manufacturers offer these compounds for sale, or they can be prepared by published synthetic methods.

One or more sequesterants can be used, though using just one is the preferred 20 means contemplated herein. The amount used can vary between about 1 to 20% by weight in a solid formulation or flowable dentifrice and between about 0.1 and 10% in a mouthwash.

An orally acceptable reducing agent is the second active component. In its broadest sense, this term is intended to include those compound which by a classical 25 chemistry definition can donate an electron to another molecule in the environment of the oral cavity without having a deleterious or unacceptably harmful affect on the oral cavity in normal and accepted use contemplated for preparations of this type. Synonyms for this term are preservatives or antioxidantizing agents. There are numerous compounds which have been proven to be useful as reducing agents. A list of such compounds 30 currently recognized for this purpose can be found in reference manuals and compendia covering pharmaceutical and oral care products. For example, see the work Martindale the Extra Pharmacopoeia, The Pharmaceutical Press, (London), Thirtieth Ed. James E. F. Reynolds Ed. (1993) beginning at page 1132-1139. In so far as they meet the standards of oral acceptability, any one of the compounds mentioned there have the potential to be 35 used in this invention. Certain reducing agents are to be preferred over others in this invention. For example vitamin C and its esters, vitamin E, the benzoates and hydroxybenzoates, butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA) and other reducing phenols, derivatives of dihydroxyquinoline, derivatives of

polymerized 2,2,4-trimethyl-1,2-dihydroquinoline and alkyl gallate such as dodecyl gallate, ethyl gallate, octyl gallate, propyl gallate. Vitamin C, vitamin E, BHA, BHt, and propyl gallate are more preferred. Vitamin C preferred above the others. All of these compounds are available from commercial sources.

5 As noted vitamin C is the preferred active ingredient. The acid form can be used as well as its monovalent metal salts; the sodium salt is widely available. Also, the esters of ascorbic acid may be used. The palmitic acid ester, ascorbyl palmitate, is readily available under the tradenames Neocutis and Ondascora. Vitamin C and its salts can be purchased from any number of manufacturers. Chemistries for making vitamin C are  
10 available in the chemical literature as well.

These preparations will be presented in a form which is safe for use in the oral cavity and which will not have a deleterious effect if accidentally swallowed. The oral care art has developed a substantial body of formulation types and has identified and tested a large listing of ingredients which can be used in these preparations in a safe and  
15 efficacious manner. Confecting or manufacturing these preparations, and their safe packaging and storage is also well documented in these arts. It is anticipated that the sequestering agents and the vitamin C-based whitening compositions contemplated by this invention can be formulated, manufactured and provided to the consumer using the technologies which are contemporary with this invention and that future developments in  
20 the dental formulation arts will be applicable as well. If modifications are needed to accommodate the actives, it is expected that routine investigations will be all that is necessary to accomplish these changes.

While this unique combination of materials can be presented in any form, it is contemplated that certain physical preparation, as compared with ingredient mixes, will  
25 provide better and faster results. By example, one can provide the two actives in the form of an oral rinse, a gum, a dentifrice such as a paste, gel or powder, or one can impregnate these materials into a toothpick or dental floss, or coat them with a preparation containing the sequesterant and reducing agent. Appliqués and mouth pieces can also be used, that is preparations which are applied to the teeth and left in place for  
30 several hours with or without a covering membrane to secure them in place. While this combination of actives can be used in a professional setting, it is intended primarily as a self-treatment product.

In addition, a course of care may include using two or more of these presentations. Say for example a mouth wash can be used in conjunction with a tooth  
35 paste where both contained the two ingredients. Such a regimen can contribute to optimizing the affect of this oral care regimen, particularly in those consumers who normally use both products. Flossing in conjunction with using tooth paste can also comprise an effective treatment regimen.

It is contemplated that these preparations would be formulated in a way which permitted them to be used in the fashion and for the time the consumer would normally associate with the use of that presentation. For example, if the actives were presented as a mouth wash, it would be formulated so that the directions for use would be consonant

5 with the normal and accepted practice of using mouth washes. The same applies for dentifrice's, toothpicks, or dental floss and chewing or bubble gums. No special time requirements are contemplated for the practice of this invention. If an appliqu  or mouth piece approach is used, that necessarily entails keeping the materials on the teeth for up to several hours, or more. That is the only context where extended treatment is the norm.

10 Preferably the two actives will be formulated into a tooth paste or gel. So for example an effective amount of a sequesterant and the reducing agent will be mixed other orally acceptable ingredients to make a paste or gel which will be packages in a usual fashion. The consumer will then put some of this gel or paste on a tooth brush and brush her teeth for a discrete time, just as if she was using any other tooth paste. Most

15 dentists and researchers recommend brushing ones teeth for at least three minutes per brushing to achieve maximum results, though compliance with this standard is not universal. A similar standards is recommended for the instant pastes and gels, though it is expected that non-compliance will still provide the desired results with regular use, i.e. daily use.

20 An effective amount of sequesterant and reducing agent will vary with depending on the vehicle and whether or not some other form of mechanical energy is put into the process (brushing or irrigating). Toothpastes and gels will have between about 1 and 25% of the sequestering agent and 0.1 to 20% of the reducing agent. These figures apply to dentifrices, mouth washes and the like. Appliqu s and mouth pieces will

25 contain essentially the same concentrations on a weight/weight basis. Gums will contain between about the same of each active.

Any orally acceptable carrier or carriers can be used, with the limitation that multivalent metal ions should be eliminated or minimized, otherwise these ions will form a complex with the sequestering agent in the product and prematurely render in partially

30 or wholly ineffective. In addition, oxidizing agents should not be used lest they react with the reducing agent. Otherwise, the two actives can be formulated with any compatible excipients which are also acceptable for use in the oral cavity.

Formulation pH may be a factor in optimizing the efficacy of some or all of the sequestering agents. Although the preferred pH of a dentifrice, for example, is

35 approximately 7, a given sequesterant may be most active as a whitening agent at another pH. Thus the preferred specie, STP, is more effective at alkaline pH as the dissociation constant is higher. STP in some preparations was most effective at a pH of about 8. Other sequesterants may be optimized by adjusting the pH to a given point. What that

might be will vary with the sequesterant and may well be influenced by the carrier. Because there may be several variables, not all of which are known or defined with precision, which come into play in selecting an optimum pH for a dentifrice, it is recommended to test this factor as per the examples given below. Other delivery forms 5 such as a mouth wash or gums may have unique pH requirements as well. They have not been investigated but such investigation is within the routine skill of the artisan or can be readily carried out using routine skills and the disclosures presented herein.

These preparations can be presented in two or more separately formulated and 10 separately stored forms if the essential actives are not compatible or the optimum conditions for a given active would be such that the other would be degraded during storage. Thus it is contemplated that STP, for example, would be formulated at an optimum pH range of between about 8 and 10 while a separate preparation of vitamin C, for example, would be prepared at an acidic pH. Each formulation would then be filled 15 into separate tubes or other containers, for example, which are jointly packaged and sold with instructions to dispense the two separate preparations at or around the time they are to be applied to the teeth or prostheses. An illustration of this article is that of a tube with two separate compartments, one containing STP at a pH of 8.0 and the other containing a vitamin C preparation at pH of about 5.0. The tube would have a common dispensing nozzle which may or may not be divided so that when the dispensing 20 mechanism was activated, a portion of the material in each tube would be dispensed onto a device of some sort, say for example a toothbrush or a mouth piece.

## Oral Preparations

### Dentifrices

Foundation formulations for toothpastes, gels and toothpowders which can be 25 used in this inventions will have the usual carriers, binders, surfactants, humectants, coloring agents, pigments, antiplaque agents, anti-bacterial agents, bioadhesive-type agents, abrasives, anticaries agents, flavorings, sweeteners, bulking agents and the like which can be used in preparing pastes and powders. Gels and pastes contain water.

Dental abrasives are useful with these actives as a means for providing physical 30 removal of materials which have been acted on by the sequestering agent and vitamin C. Classic examples of dental abrasives are calcium pyrophosphate, silica abrasives, alumina, insoluble metaphosphates, particulate thermosetting polymerized resins, and sodium bicarbonate. The patent and scientific literature is replete with examples of such abrasives. One such example is U.S. patent 4,822,599 which list a series of dentifrice 35 abrasives and references commercial sources and literature references on their preparation. Most if not all of dental abrasives are available from commercial sources.

A selected abrasive should be compatible with two active ingredients as well as any additives which may be actives in their own right, such as fluoride ions and antibacterial agents. But as with any other paste, gel or powder, the selection of an abrasive can be influenced by the consequence of combining a particular abrasive with 5 another additive. For example if fluoride ions and calcium pyrophosphate ions are to be included in these preparations the pyrophosphate should be converted from its  $\gamma$ -phase to its  $\beta$ -phase by heating the  $\gamma$ -phase to 700°-900° C as per the teachings of U.S. patent 3,112,247. Also certain quaternary ammonium-based antibacterial agents may not be compatible with some silica abrasives. Silica is a preferred abrasive for this work.

10 Abrasive concentrations can cover a very broad range. Preparations are described with abrasive ranging in concentration from 5 to 80% by weight depending on the abrasive. A secondary concentration range is that of 10 to 50% depending on the abrasive selected. Herein the preferred abrasive, silica, is employed in amounts between 10 and 20% by weight.

15 A source of fluoride ion may be included in these preparations. Fluoride ion sources are numerous. For example see U.S. patent 3,535,421 which lists numerous salts which can be used in the dental arts. While any one of these sources could be used sodium fluoride, stannous fluoride and sodium monofluorophosphate have emerged as the preferred ion sources in most dentifrices.

20 Fluoride ions are routinely added into dentifrices in an amount sufficient to provide about 1000 ppm of the fluoride ion. Where a preparation is formulated such that the fluoride ion is confined to one component of the preparation, but is mixed with the other components at the time of use, the fluoride ion source should be adjusted upward in an amount sufficient to provide a concentration of about 1000 pp. in the product as used.

25 As for other components, flavorings, coloring agents, sweeteners, humectants, thickening agents, binders and surfactants are most commonly used in dentifrices.

30 Taste is provided by adding a small amount of a flavoring agent; this component also leaves a perception of mouth freshness. Numerous minty flavored agents are available for use in dentifrices and it is well known in the art how to go about selecting a flavoring and testing its consumer acceptability. Flavoring agents are routinely used at levels of between about 0.1 to 5% by weight.

35 Dyes, lakes and titanium dioxide are routinely used in the dentifrice arts for imparting color; in the case of titanium dioxide a white paste or powder is obtained. These materials are widely available, have been oft used in such formulations and are well known to the dental artisan. Coloring agents are usually present in concentrations ranging between 01. and 5%.

Sweeteners are routinely added to increase consumer acceptability. So called artificial sweeteners are used today to avoid the cariogenic potential of most sugars and

other sweetening agents. Examples of non-cariogenic sweeteners now in routine use are saccharin, aspartame, D-tryptophan, dihydrochalcones, cyclamates, and acesulfame. Sweeteners comprise about 0.1 to 5% of the formulation.

Humectants are added to gels and pastes to prevent their drying out on exposure to air, and they impart a "moist" feel to the mouth when brushing. Some humectant, eg. sorbitol, are perceived as sweet. Examples of compounds useful as humectants in dentifrices are the polyhydric alcohols such as glycerin, sorbitol, zylitol, and polyethylene glycols. Sorbitol (usually 70% sorbitol/water) and glycerin are preferred. In pastes and gels one or two humectants are usually used in amounts between about 20 and 40%.

Binders and thickening agents can be added to assure physical integrity. Examples of these are gums such as xanthan and acacia gum, carageenan, the celluloses such as carboxy methyl cellulose and certain polymers exemplified by the carboxyvinyl polymers (Gantrez and the like). These latter polymers, and perhaps some of the others, have an additional benefit in that their adhesive nature which makes them useful as binders also can serve the additional purpose of adhering to the teeth surface and thereby binding the active ingredients to the teeth for a longer period. Gantrez is a example of a polyacrylic carboxylate material which serves such a dual purpose.

Surfactants normally are added to dentifrices to assist with removing debris. All classes of surfactants, anionics, cationics, amphoteric, nonionics and zwitterion-based surfactants, can be used in these preparations. These compounds, and those which are most useful in the dental arts, are well documented in the literature. Reference is made to U.S. patent 4,822,599 for a detailed listing of useful surfactants. Surfactants are available through any number of commercial manufacturers or can be make by well documented processes.

Surfactants are normally used in amounts between about 0.5 and 5% in pastes and gels but may be used at higher concentrations in some dental powders.

Appliqués can provide an effective means for removing stains as per this invention. These can be prepared in the form of a doughy or tacky material which can be readily molded to conform to the teeth. It can then be manually compressed on the teeth as is or placed in a plastic retainer, inserted into the mouth and bitten into, and left in place of a some time, for example 15 to 30 minutes. When the appliquéd is removed the debris causing the stain will be removed. The appliquéd is then discarded.

The two actives can be formulated as a mouthwash or mouth rinse as well. A mouth wash or rinse will contain up to water, up to 30 % alcohol, flavor, polyhydric alcohols, anti-caries agents, plaque removing agents, sweeteners, dyes and lakes, and a

preservative in some instances. The two actives could also be incorporated into currently existing formulations such as Cepacol (Lakeside Pharmaceuticals), Plax, (Pfizer), Listerine (Warner-Lambert), Scope (Procter & Gamble), and the like.

Concentrations of the actives in these products would be in the range of about 1  
5 to 5% (weight/volume) for the sequestering agent and reducing agent.

A soaking and cleaning solution for dental pieces can also be prepared with these  
two active ingredients. It is contemplated that such preparations would contain water, a  
surfactant, an effervescing agent, and other optional ingredients. Dental prostheses  
would be removed and placed in a solution containing the sequestering agent and vitamin  
10 C and soaked for several hours, then either brushed with a recommended dentifrice or  
simply rinsed and reinserted into the mouth. The concentration of actives here 1-20% of  
the sequestering agent and 0.1 to 15% of the reducing agent.

The following examples are provided by way of illustration and are not intended  
to limit the scope of the invention.

15

Example 1

Formulation of Tooth Whitening Toothpaste

A toothpaste representative of what may be prepared for the practice of this  
invention was prepared as per the ingredient profile and percentages in Table I.

Ingredient	Concentration (W/W%)
Water, DI	24.477%
Sorbitol, 70%	24.53
Abrasive silica	14.0
Sodium TriPhosphate	10.0
Glycerin	10.0
Ascorbic Acid	5.0
Thickening silica	4.0
Polyethylene glycol 400, NF	3.0
Sodium Lauryl Sulfate	1.15
Titanium Dioxide	1.0
Sodium Saccarin	1.0
Xanthan Gum	0.8
Flavor	0.8
Sodium Fluoride	0.243
Total	100.00

Example 2

Tooth whitening was measured by the following process:

Whitening was determined using a modification to a system normally employed in the dental arts for determining the abrasivity of dentifrices.

5        Bovine jaws were obtained at a local abattoir and the teeth were extracted in their native state; no precleaning was done. An initial L value was determined on a colorimeter (Hunter). This was designated as the initial whiteness value. Teeth were then mounted in the trays of a wear tester device manufactured by Ramè-Hart, of Mountain Lakes, New Jersey. This machine is often used to test the abrasivity of dental  
10      preparations. Tooth brushed were mounted and the teeth and brushes aligned. A 1:3 slurry of paste and water was poured into the trays and brushing commenced for a total of six hours. The slurry was replaced hourly. At 3 and 6 hours L value determinations were made. This was done by first rinsing the teeth with deionized water, then placing them in a closed container at 100% humidity for 1 hours, then taking a reading on the  
15      colorimeter to obtain a 3 hour and 6 hour L value.

Four types of preparations were tested. A placebo was prepared by replacing the tripolyphosphate and vitamin C content in the Example 1 formulation with water. In a second preparation the polyphosphate was replaced with water but the vitamin C was retained. Thirdly the vitamin C was replaced with water, but the polyphosphate was  
20      retained. Fourthly the paste describe in Example 1 was tested. This latter formulation provided whitening results superior to the other 3 preparations.

What is claimed is:

1. A composition for reducing or removing surface deposited stains from natural teeth and dental prostheses comprising an effective amount of an orally acceptable sequestering agent and an orally acceptable reducing agent in an orally acceptable carrier.
- 5 2. The composition of claim 1 in which the sequestering agent is sodium tripolyphosphate and vitamin C.
3. The composition of claim 2 which is in the form of a paste or gel.
4. The composition of claim 3 wherein the phosphate is present in an amount 10 of 10% and the vitamin C is present in an amount of 5%.
- 15 5. A means for removing surface deposited stains from natural teeth and dental prostheses which method comprises contacting teeth or dental prostheses which have surface stains with a composition comprising an effective amount of a sequestering agent and an orally acceptable reducing agent in a carrier for a time sufficient to remove at least a major amount of said stain.
6. An article of manufacture comprising a first article which contains an orally acceptable reducing agent suitable and a second article which contains a sequestering agent in a form suitable for combining with first article for use in whitening teeth.
- 20 7. A product for whitening natural teeth or dental prostheses prepared by combining the first and second articles of claim 6.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US94/09185

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :A61K 7/20

US CL :424/49, 53

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)

U.S. : 424/49, 53

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)

APS, CASONLINE, DERWENT

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 2,470,906 (TAYLOR) 24 MAY 1949.	1-7
A	US, A, 3,888,976 (MLKVY et al.) 10 JUNE 1975.	1 to 7
A	US, A, 4,115,293, (SCHOENHOLZ et al.) 19 SEPTEMBER 1978.	1-7
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Further documents are listed in the continuation of Box C.



See patent family annex.

• Special categories of cited documents:	"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
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Date of the actual completion of the international search

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Date of mailing of the international search report

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Name and mailing address of the ISA/US  
Commissioner of Patents and Trademarks  
Box PCT  
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

SHEP ROSE /jd

Telephone No. (703) 308-1235

**INTERNATIONAL SEARCH REPORT**

International application No.

PCT/US94/09185

**C (Continuation) - DOCUMENTS CONSIDERED TO BE RELEVANT**

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