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Schrader

[11] **3,775,044**

[45] **Nov. 27, 1973**

[54] **THIOCARBAMIDE DIOXIDES, COMPLEXING AGENTS AND OXIDATION DYES AND HAIR DYEING THEREWITH**

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[21] Appl. No.: **244,862**

[52] **U.S. Cl.** ..... **8/11,** 8/10.2, 8/86, 45/9 R

[51] **Int. Cl.** **D06p 1/32**

[58] **Field of Search** ..... 8/10.2, 11, 86, 32; 45/9 R

[56] **References Cited**

UNITED STATES PATENTS

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[57] **ABSTRACT**

In a process for dyeing human hair by means of an aqueous composition containing oxidation dyes, the improvement which comprises including in said composition a thiocarbamide dioxide in combination with a complexing agent.

15 Claims, No Drawings

THIOCARBAMIDE DIOXIDES, COMPLEXING AGENTS AND OXIDATION DYES AND HAIR DYEING THEREWITH

PRIOR ART

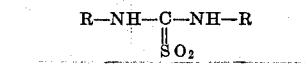
Oxidation dyes are used to a considerable extent for the purpose of dyeing human hair. Reducing agents are generally added in a smaller quantity to the oxidation dyes in order to avoid oxidation of the dyes during storage and before mixing with an oxidizing agent. However, these additives are insufficient to prevent oxidation of the dye in the mixing container after adding H_2O_2 and before or during application of the dye solution or cream to the hair. This reduces the coloring power of the mixture and renders it extremely difficult to obtain uniform coloration of the hair. Increasing the content of reducing agent in the dyeing cream does not provide a solution to this problem, since, as a result of reduction, a corresponding portion of the required oxidizing agent which has been added is rendered ineffective.

OBJECTS OF THE INVENTION

It is therefore an object of the present invention to overcome the above-stated disadvantages of the prior art by providing a process for dyeing human hair with an aqueous composition containing oxidation dyes and including thiocarbamide derivative additives with a complexing agent in said composition.

It is a further object of the present invention to provide a process and composition for dyeing human air that prevents premature oxidation of the oxidation hair dyes, so as to maintain the coloring power of the hair dye in order to obtain uniform coloring of the hair.

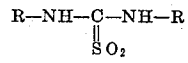
It is another object of the present invention to provide a development in the process of dyeing human hair which comprises applying to the hair at a temperature of from 10°C to 40°C for a time sufficient to effect dyeing an effective amount of an aqueous composition for dyeing human hair comprising (a) an oxidation dyestuffs combination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence of an oxidizing agent, wherein the improvement consists of including in said aqueous composition (1) from 0.05 to 2 percent by weight based upon the total composition weight of a thiocarbamide derivative of the formula



wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total composition weight of an organic complexing agent.

It is a further object of the present invention to provide a development in an aqueous composition for dyeing human hair comprising (a) an oxidation dyestuffs combination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence or absence of an oxidizing agent, wherein the improvement consists of including in said aqueous composition (1) from 0.05 to 2 percent by

weight based upon the total composition weight of a thiocarbamide derivative of the formula

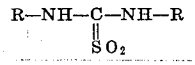


wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total weight of an organic complexing agent.

Other and further objects of the present invention will become apparent as the description thereof proceeds.

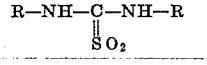
DESCRIPTION OF THE INVENTION

The present invention is directed to a development in the process of dyeing human hair which comprises applying to the hair at a temperature of from 10°C to 40°C for a time sufficient to effect dyeing an effective amount of an aqueous composition for dyeing human hair comprising (a) an oxidation dyestuffs combination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence of an oxidizing agent, wherein the improvement consists of including in said aqueous composition (1) from 0.05 to 2 percent by weight based upon the total composition weight of a thiocarbamide derivative of the formula



wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total composition weight of an organic complexing agent.

The present invention is also directed to a development in an aqueous composition for dyeing human hair comprising (a) an oxidation dyestuffs combination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence or absence of an oxidizing agent, wherein the improvement consists of including in said aqueous composition (1) from 0.05 to 2 percent by weight based upon the total composition weight of a thiocarbamide derivative of the formula



wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total weight of an organic complexing agent.

In essence, therefore, the present invention is directed to an improvement in the conventional oxidation dyestuff formulations and methods of dyeing human hair wherein a type of antioxidant is employed. The said improvement comprises the addition to the aqueous dyestuff emulsion of (a) the above thiocarbamide derivative, (b) the above organic complexing agent and (c) small amounts of a reducing agent as said antioxidant.

Suitable thiocarbamide derivatives are compounds such as N,N'-dimethylthiocarbamide dioxide, N,N'-diethylthiocarbamide dioxide, N,N'-dipropylthiocarbamide dioxide and N,N'-dibutylthiocarbamide dioxide, as well as N-methylthiocarbamide dioxide, N-ethylthiocarbamide dioxide, N-propylthiocarbamide dioxide and N-butylthiocarbamide dioxide. Preferably, thiocarbamide dioxide is used. Alternatively, mixtures of the said compounds may be used if desired.

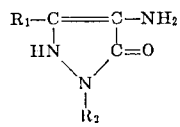
The reducing agents used are compounds known for this purpose, such as sodium sulfite, thioglycollic acid or ascorbic acid. Alternatively, water-soluble salts of these compounds, such as the sodium,potassium, ammonium or ethanolamine salts may be used instead of the free acids. Suitable organic complexing agents are: hydroxy-loweralkanediphosphonic acids such as 1-hydroxyethane-1, 1-diphosphonic acid and 1-hydroxypropane-1,1-diphosphonic acid, amino-loweralkanediphosphonic acids such as 1-aminoethane-1,1-diphosphonic acid and 1-aminopropane-1,1-diphosphonic acid, aminotrimethylenephosphonic acid, 8-hydroxyquinoline, nitrilotriacetic acid, diethylenetriaminepentaacetic acid and preferably ethylenediaminetetraacetic acid. Alternatively, water-soluble salts of these acids such as the alkali metal salts, for example sodium, potassium, or ammonium or ethanolamine salts may be used instead of the acids.

The reducing agent is added in quantities of from 0.1 to 3 percent, preferably 1 to 2 percent by weight based upon the total composition. The complexing agent is added in quantities of from 0.1 to 2 percent, preferably 0.1 to 0.5 percent by weight based upon the total composition, while the thiocarbamide derivative is added in quantities of from 0.05 to 2 percent, preferably 0.1 to 0.5 percent by weight based upon the total composition.

The oxidation dyes used are preferably the aromatic diamines, the phenols and/or the aminophenols known for this purpose. Particularly suitable developer components are substituted ortho- and paraphenylenediamines, N-alkylaminoanilines, N,N-dialkylaminoanilines and aminophenols such as p-toluylenediamine, o-toluylenediamine, 2,5-diaminoanisole, p-aminodiphenylamine, N,N-dimethylaminoaniline, N,N-diethylaminoaniline, N-methylaminoaniline, N-ethylaminoaniline, and N,N-β-hydroxyethylaminoaniline.

Coupling components include aromatic diamines, phenols, and aminophenols in which, in particular, a functional group such as a hydroxyl group, an amino group, or an alkoxy group is in the meta position relative to the amino- or hydroxyl group. Suitable compounds are, for example, resorcinol, m-toluylenediamine, 2,4-diaminoanisole, m-aminophenol, m-methylaminophenol and α-napthol.

Pyrazolone derivatives, such as are described in U.K. Patent Specification No. 1,213,697, and U.S. Patent application Ser. No. 167,855, filed July 30, 1971, may also be used as oxidation dyes. The latter specifications describe 4-aminopyrazolones of the general formula:



as a developer component, wherein R_1 and R_2 each represent a hydrogen atom or an organic radical having one to 10 carbon atoms.

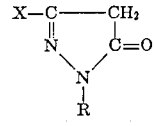
The organic radical R_1 of the 4-aminopyrazolone may, for example, comprise an alkyl having one to 10 carbon atoms, or an aryl or heterocyclic radical. Furthermore, functional groups such as —OH, —NH_2, —$NHCH_3$, —$N(CH_3)_2$ or halogen atoms, particularly chlorine, may also be present. The groups —COOH, —COOR', —CONHR' —CONR'R'' may be present as organic radicals, wherein R' and R'' represent a lower alkyl or hydroxyalkyl radical having one to four carbon atoms.

The organic radical R_2 of the 4-aminopyrazolone may comprise an alkyl having one to 10 carbom atoms or, alternatively, an aryl or heterocyclic radical. Furthermore, functional groups may also be present. Hydrocarbon radicals which have one to 10 carbon atoms, and which contain —OH, —NH_2, —COOH, —$CONH_2$, —SO_3H and —SO_2NH_2 as functional groups, are particularly suitable.

A phenyl radical is particularly suitable as the aryl radical. The phenyl radical may contain alkyl or hydroxyalkyl groups having one to four carbom atoms or other substituents such as an —NH_2, —OH, —COOH, —$CONH_2$, —SO_3H and —SO_2NH_2 group.

It is frequently advantageous to use the 4-aminopyrazolones in the form of their salts, preferably the hydrochloride salts, since this reduces their sensitivity to air.

3-aminopyrazolones of the general formula:



are also suitable as the coupling component, wherein R represents a hydrogen atom, an alkyl or phenyl radical and X represents an NH_2—, NHR_1—, NR_1R_2—group (R_1 and R_2 being alkyl radicals having one to four carbon atoms) or an OH group.

When using these oxidation dyes, the coloring of the hair is substantially intensified by the addition of thiocarbamide dioxide in combination with a reducing agent and a complexing agent.

Likewise, the 4-aminopyrazolones mentioned as developer components may be combined with the aromatic diamines, phenols or aminophenols already mentioned above as coupling components.

The concentration of the dyes or dye components is dependent to a certain extent upon the intended use, and particularly upon the degree of the desired coloration. The concentration is usually from 0.1 to 5 percent by weight, preferably 0.5 to 2 percent by weight, based upon the total composition.

When using oxidation dyes, the colors are developed by atmospheric oxygen, or, preferably, by chemical oxidizing agents. Preferably, hydrogen peroxide or its products of addition, such as percarbamide, melamine-perhydrate, or sodium perborate, are used as chemical oxidizing agents.

The hair is dyed at a temperature of from 10° to 40°C, but preferably at room temperature. The hair dyes may be used in the form of aqueous solutions, or especially in the form of creams or emulsions. For this purpose, the dyes or dye components may be mixed with wetting agents or washing agents, particularly anionic or nonionic. Alkylbenzene sulfonates, fatty alcohol sulfates, alkyl sulfonates, fatty acid ethanolamides, products of addition of ethylene oxide to fatty acids and fatty alcohols, and fatty alcohol ether sulfates, are particularly suitable as wetting and washing agents. The hair dyes may also be manufactured in the form of shampoos, particularly cream-like shampoos.

Other known additives which may be present are thickeners such as methyl cellulose, starch, higher fatty alcohols, vaselines, paraffin oil, and fatty acids, as well as perfume oil or hair lotion such as pantothenic acid or cholesterol.

The additives are used in the conventional quantities used for these purposes. Suitable quantities of the wetting agent additive are particularly from 0.5 to 30 percent by weight related to the total composition, and suitable quantities of the thickener additives are particularly from 0.1 to 25 percent by weight based upon the total composition.

The pH-value of the hair dyes may lie in the conventional slightly acid to alkaline range. Preferably, the hair dyes are adjusted to a pH-value of from 8 to 10.

An unexpected result of the present process according to the invention is that by employing these novel hair dyes, it is possible, to a considerable extent, to prevent oxidation of the oxidation dyes, based on diamines, phenols or aminophenols, in the mixing container after adding the oxidizing agent, either before or during application of the dye solution to the hair. The coloring power of the mixture is thus maintained and it is possible to obtain uniform coloring of the hair. In this manner, the hair can be colored in considerably darker tints with light-colored dyeing creams. When oxidation dyes based on pyrazolone derivatives are used, the novel process effects a more intensive dyeing of the hair.

The invention will be illustrated with reference to the following Examples which are not to be deemed limitative in any manner thereof.

EXAMPLE 1

1.75 parts by weight of resorcinol, 1.6 parts by weight of p-toluylenediamine, 0.45 parts by weight of m-diaminoanisole, 0.38 parts by weight of naphthol and 3 parts by weight of 1,2-propylene glycol were dissolved in 30 parts by weight of water. 0.1 parts by weight of thiocarbamide dioxide, 0.9 parts by weight of thioglycollic acid and 2 parts by weight of ethylenediaminetetraacetic acid were then added thereto. The mixture thus obtained was incorporated into 50 parts by weight of an emulsion containing 10 parts by weight of fatty alcohol (carbon chain length $C_{16} - C_{18}$), 26 parts by weight of sodium lauryl ether sulfate and 34 parts by weight of water. The pH-value was adjusted to 9.5 by means of ammonia, and the emulsion was made up to 100 parts by weight with water.

50 g of 9 percent hydrogen peroxide were added to 50 g of this light-beige colored cream, thoroughly stirred, and applied to strands of grey hair which had not been pre-treated. During the entire period of application of about 30 minutes, the cream retains a light color which gradually changes to a light grey tint, whereas the hair is dyed jet-black.

The above described procedure was repeated for the purpose of comparison, but with the difference that the cream used did not contain an additive of thiocarbamide dioxide, thioglycollic acid and ethylenediaminetetraacetic acid. Immediately after the hydrogen peroxide solution was added, this cream exhibited a dark grey color which rapidly became more intense.

When as described above, one of the said additives, i.e. thiocarbamide dioxide or thioglycollic acid or ethylenediaminetetraacetic acid, was omitted in the cream used, and the same procedure was followed, the creams darkened to a deep black by way of grey within a few minutes after adding the hydrogen peroxide solution.

EXAMPLE 2

A cream as described in Example 1, paragraph 1, was prepared, except that the thioglycollic acid was replaced by 1 part by weight of sodium sulfite. 50 g of 9 percent hydrogen peroxide was added to 50 g of this light-colored cream, and the cream was applied to grey strands of hair after mixing. The period of treatment was about 25 minutes at a temperature of 28°C. During the treatment the cream exhibited only a light grey tint, while the hair is dyed jet-black.

EXAMPLE 3

A cream as described in Example 1, paragraph 1, was prepared, except that 1.5 parts by weight of 8-hydroxyquinoline were added instead of ethylenediaminetetraacetic acid.

50 g of 9 percent hydrogen peroxide were added to 50 g of the light-colored cream thus obtained, and strands of grey hair were treated therewith for 25 minutes at 28°C. The color of the dyeing cream changed only slightly during the entire period of application, while the hair was dyed black.

EXAMPLE 4

A dyeing cream as described in Example 1, paragraph 1, was prepared, except that 1 part by weight of sodium sulfite was added instead of the thioglycollic acid, and 1.5 parts by weight of 1-hydroxyethane-1, 1-diphosphonic acid were added instead of the ethylenediaminetetraacetic acid.

50 g of the light-colored cream thus obtained were mixed with 50 g of 9 percent hydrogen peroxide solution and applied to strands of grey hair which had not been pre-treated. The period of treatment was about 30 minutes at about 30°C. The hair was dyed jet-black after this period of time, while the dyeing cream had assumed only a light gray color.

Comparable results were obtained if, in a modification of the procedures according to Examples 1 to 4, an equivalent quantity of aromatic diamines, phenols or aminophenols of the kind described in the foregoing specification were used as developer components and coupling components. The development of color in the cream is inhibited to a considerable extent in all cases.

EXAMPLE 5

A dyeing cream was prepared as in Example 1, paragraph 1, except that 1 part by weight of 1-phenyl-3-carbamoyl-4-aminopyrazolone-5 and 0.9 parts by weight of 1-phenyl-3-methylpyrazolone-5 were used as dye components.

50 g of this cream were mixed with 50 g of 9% H_2O_2, and grey hair was treated therewith for 25 minutes at room temperature. A deep violet color was obtained. When the hair was treated in a similar manner, but with a dyeing cream which contained no thioglycollic acid, no thiocarbamide dioxide or no ethylenediaminetetraacetic acid, individually or mixed, only lilac colors were obtained whose intensity was considerably less.

Comparable results were obtained when, in a modification of the procedure described above, an equivalent quantity of 4-aminopyrazolones or 3-aminopyrazolones of the kind described above in the specification were used as developer and coupling components.

EXAMPLE 6

A composition was prepared by dissolving 0.85 parts by weight of p-toluylenediamine sulfate, 0.25 parts by weight of p-aminophenol hydrochloride, 0.08 parts by weight of resorcinol, and 0.1 parts by weight of m-aminophenol in 20 parts by weight of water to which ammonia was added. 0.1 parts by weight of N,N'-dimethylthiocarbamide dioxide, 1.5 parts by weight of ascorbic acid and 2 parts by weight of ethylenediaminetetraacetic acid were also added thereto.

The mixture thus obtained was incorporated in 50 parts by weight of an emulsion containing 10 parts by weight of fatty alcohol (carbon chain length C_{16} to C_{18}), 26 parts by weight of sodium lauryl ether sulfate and 34 parts by weight of water. The pH-value was adjusted to 9.5 with ammonia and the mixture was made up to 100 parts by weight with water. 50 g of 6 percent hydrogen peroxide were added to 50 g of this almost white-colored dyeing cream, which was then well stirred, and the cream was applied to dark-blond hair. The reaction time was about 10 minutes. The hair was tinted a light brown. The dyeing cream changed color to only a slight extent within the stated time.

Virtually the same results were obtained when, in a modification of the procedure described above, N,N'-diethylthiocarbamide dioxide, N,N'-dipropylthiocarbamide dioxide or N,N'-dibutylthiocarbamide dioxide were used as the thiocarbamide derivative.

The above described procedure was repeated for the purpose of comparison, but with the difference that the cream used contained no N,N'-dimethylthiocarbamide dioxide, no ascorbic acid or no ethylenediaminetetraacetic acid. This cream exhibited a brownish color immediately after adding the hydrogen peroxide solution.

EXAMPLE 7

0.001 mol 1-phenyl-3-dimethylamide-4-aminopyrazolone-5 and 0.001 mol α-naphthol were dissolved in 20 parts by weight of water to which ammonia was added. 0.1 parts by weight of N-methylthiocarbamide dioxide, 1 part by weight of sodium sulfite, and 0.2 parts by weight of diethylenetriaminepentaacetic acid were also added.

The mixture thus obtained was incorporated in 50 parts by weight of an emulsion containing 10 parts by weight of fatty alcohol (carbon chain length C_{16} to C_{18}), 26 parts by weight of sodium lauryl ether sulfate, and 34 parts by weight of water. The pH-value was adjusted to 9.5 with ammonia and the mixture was made up to 100 parts by weight with water. 50 g of 6 percent hydrogen peroxide were added to 50 g of this almost white-colored dyeing cream, which was then well stirred; and the cream was applied to white hair. The reaction period was about 10 minutes. The hair was dyed an intense blue color.

Virtually the same results were obtained when in a modification of the procedure according to the above two paragraphs N-ethylthiocarbamide dioxide, N-propylthiocarbamide dioxide or N-butylthiocarbamide dioxide was used as the thiocarbamide derivative.

The above described procedure was repeated for the purpose of comparison, but with the difference that the cream used contained no N-methylthiocarbamide dioxide, no sodium sulfite or no diethylenetriaminepentaacetic acid. The color obtained on the white hair was only slightly blue.

EXAMPLE 8

Dyeing creams as described in Example 1, paragraphs 1 and 2, were prepared, except that a corresponding quantity of the sodium, potassium, ammonium and/or ethanolamine salt of thioglycollic acid was used instead of the thioglycollic acid in each case. The same results were obtained as with the thioglycollic acid.

EXAMPLE 9

Dyeing creams as described in Example 6, paragraphs 1 and 2, were prepared, except that a corresponding quantity of the sodium, potassium, ammonium and/or ethanolamine salt of ascorbic acid was used instead of the ascorbic acid in each case. The same results were obtained as with ascorbic acid.

EXAMPLE 10

Dyeing creams as described in Example 1, paragraphs 1 and 2, were prepared, except that a corresponding quantity of 1-hydroxyethane-1, 1-diphosphonic acid, 1-hydroxypropane-1, 1-diphosphonic acid, 1-aminoethane-1, 1-diphosphonic acid, 1-aminopropane-1, 1-diphosphonic acid, aminotrimethylene-phosphonic acid and nitrilotriacetic acid was used as a complexing agent instead of ethylenediaminetetraacetic acid.

When dyeing the hair, virtually the same results were obtained as with ethylenediaminetetraacetic acid.

EXAMPLE 11

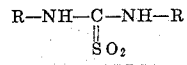
The complexing agents present in the form of the free acids in Examples 1 to 10 were replaced by the corresponding sodium, potassium, ammonium and/or ethanolamine salts. The same results were obtained as with the free acids.

Although the present invention has been disclosed in connection with a few preferred embodiments thereof, variations and modifications may be resorted to by those skilled in the art without departing from the principles of the new invention. All of these variations and modifications are considered to be within the true spirit and scope of the present invention as disclosed in the foregoing description and defined by the appended claims.

I claim:

1. In the process of dyeing human hair which comprises applying to the hair at a temperature of from 10°C to 40°C for a time sufficient to effect dyeing an effective amount of an aqueous composition for dyeing human hair comprising (a) an oxidation dyestuffs com-

bination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence of an oxidizing agent, the improvement which consists of including in said aqueous composition (1) from 0.05 to 2 percent by weight based upon the total composition weight of a thiocarbamide derivative of the formula



wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total composition weight of an organic complexing agent.

2. The process as claimed in claim **1** in which the thiocarbamide derivative is thiocarbamide dioxide.

3. The process as claimed in claim **2,** in which the reducing agent is selected from the group consisting of thioglycollic acid, a water-soluble salt of thioglycollic acid, and the mixtures thereof.

4. The process as claimed in claim **2** in which the organic complexing agent is selected from the group consisting of ethylenediaminetetraacetic acid, a water-soluble salt of ethylenediaminetetraacetic acid, and the mixtures thereof.

5. The process as claimed in claim **1,** in which the thiocarbamide derivative is selected from the group consisting of N-alkylthiocarbamide dioxide and N,N'-dialkylthiocarbamide dioxide, said alkyls having from one to four carbon atoms.

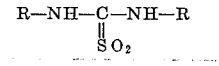
6. The process as claimed in claim **5,** in which the reducing agent is selected from the group consisting of thioglycollic acid, a water-soluble salt of thioglycollic acid, and the mixtures thereof.

7. The process as claimed in claim **5** in which the organic complexing agent is selected from the group consisting of ethylenediaminetetraacetic acid, a water-soluble salt of ethylenediaminetetraacetic acid, and the mixtures thereof.

8. The process of claim **1** wherein said aqueous solution has a further content of from 0.5 to 30 percent by weight of a surfactant selected from the group consisting of alkylbenzenesulfonates, higher fatty alcohol sulfates, higher alkylsulfonates, higher fatty acid ethanolamides and ethylene oxide adducts of higher fatty alcohols and alkylphenols.

9. The process of claim **1** wherein said aqueous solution has a further content of from 0.1 to 25 percent by weight of thickeners selected from the group consisting of methylcellulose, hydroxymethylcellulose, starch, higher fatty alcohol, vaselin, paraffin oils and fatty acids.

10. In an aqueous composition for dyeing human hair comprising (a) an oxidiation dyestuffs combination of a developer component and a coupling component, (b) from 0.1 to 3 percent by weight based upon the total composition weight of a reducing agent, (c) in the presence of an oxidizing agent, the improvement which consists of including in said aqueous composition (1) from 0.05 to 2 percent by weight based upon the total composition weight of a thiocarbamide derivative of the formula



wherein R is a member selected from the group consisting of hydrogen and alkyl having one to four carbon atoms, and (2) from 0.1 to 2 percent by weight based upon the total weight of an organic complexing agent.

11. The composition as claimed in claim **8,** in which the thiocarbamide derivative is thiocarbamide dioxide.

12. The composition as claimed in claim **9,** in which the reducing agent is thioglycollic acid.

13. The composition as claimed in claim **9** in which the organic complexing agent is ethylenediaminetetraacetic acid.

14. The composition as claimed in claim **8,** in which the thiocarbamide derivative is selected from the group consisting of N-alkylthiocarbamide dioxide and N,N'-dialkylthiocarbamide dioxide.

15. The composition as claimed in claim **8** in which there is (1) from 0.1 to 0.5 percent by weight of thiocarbamide derivative, (2) from 0.1 to 0.5 percent by weight of complexing agent, and (b) from 1 to 2 percent by weight of reducing agent.

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