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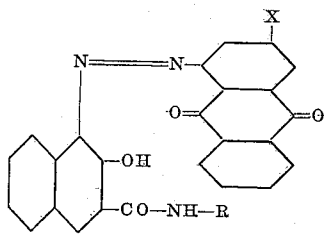
AZO DYESTUFFS INSOLUBLE IN WATER, AND FIBER DYED THEREWITH

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12 Claims. (Cl. 260—192)

The present invention relates to azo-dyestuffs insoluble in water and to fiber dyed therewith; more particularly it relates to dyestuffs corresponding with the following general formula:



wherein R means a radical of the benzene or naphthalene series and X stands for a substituent, such as halogen, an alkyl, alkoxy or trifluoromethyl-group.

We have found that valuable azo-dyestuffs, insoluble in water, may be obtained by coupling in substance, on the fiber or on a substratum adapted for the production of lakes a diazo-compound of a 1-aminoanthraquinone containing in 3-position a substituent, such as, for instance, halogen, an alkyl-, alkoxy- or trifluoromethyl-group, with an arylide of 2.3-hydroxynaphthoic acid, neither of the components containing any group imparting solubility, such as, for instance, sulfonic acid or carboxylic acid groups.

In this manner vivid scarlet and red shades of good properties of fastness are obtained. In comparison with the known azo-dyestuffs from unsubstituted 1-diazo-anthraquinone the new dyestuffs represent a considerable technical advance; they have generally a better fastness to light and to soaping and throughout an essentially clearer shade. A special advantage of the invention resides in the fact that the diazo-compounds used are rather soluble in water. This behavior of the substituted 1-diazo-anthraquinones could not be foreseen, since it is known that amino-compounds carrying substituents, especially those of negative character, in general yield diazonium compounds which are more sparingly soluble in water than those of the unsubstituted amino-compounds.

The following examples serve to illustrate the invention, but they are not intended to limit it thereto, the parts being by weight unless otherwise stated; the parts by weight and the parts by volume are related to each other as are kilograms to liters:

1. *Grounding solution*

6 parts of 1-(2'.3'-hydroxynaphthoylamino)-benzene are dissolved, while heating, in
6 parts by volume of alcohol,
3 parts by volume of formaldehyde solution of 30 per cent strength and
3 parts by volume of caustic soda solution of 34° Bé. and the solution is introduced into a solution of
5 parts by volume of Turkey red oil and
10 parts by volume of caustic soda solution of 34° Bé. in
1000 parts by volume of water.

Developing solution

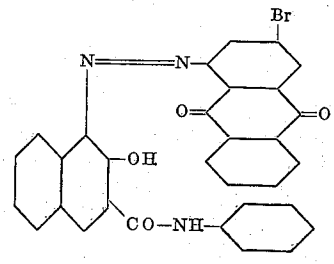
2.055 parts of the acid diazonium sulfate obtainable by diazotization of 1.51 parts of 1-amino-3-bromanthraquinone, are dissolved at room temperature in 1000 parts by volume of water with addition of 1 part of acetic acid of 50 per cent. strength.

Dyeing process

50 parts of cotton yarn or mixed fabric from cotton and viscose staple fiber are treated in the grounding solution at 20° C. for half an hour. The material is then well squeezed and introduced into the developing solution. After half an hour the coupling is finished. The material is well rinsed, soaped at boiling temperature, rinsed again and dried.

A bright scarlet of very good fastness to light is obtained.

The dyestuff corresponds with the following formula:



2. 15 parts of 1-(2'.3'-hydroxynaphthoylamino)-benzene,
15 parts by volume of Monopol brilliant oil and
20 parts by volume of caustic soda solution of 38° Bé.

are dissolved in boiling water and the whole is made up to 1000 parts by volume. Fabrics from cotton or viscose staple fiber are padded with this solution. The material is then dried and printed with a printing paste which has been obtained by dissolving 26 parts of the acid diazonium sulfate from 1-amino-3-bromanthraquinone in

10 parts of acetic acid of 50 per cent strength
515 parts of water and
450 parts of starch tragacanth thickening.

After printing, the material is dried and treated with a sodium carbonate solution of 1 per cent strength, rinsed and soaped at boiling temperature.

Very uniform scarlet-red prints are obtained which are distinguished by their clarity and good properties of fastness.

By using as grounding solution instead of 1-(2'.3'-hydroxynaphthoylamino)-benzene the same quantity of 1-(2'.3'-hydroxynaphthoylamino)-2-methylbenzene or 12 parts of 1-(2'.3'-hydroxynaphthoylamino)-2-methoxybenzene, very clear, fast scarlet tints are likewise obtained which, in contradistinction to the known prints obtained by means of 1-diazo-anthaquinone, are very uniform also when produced with a printing paste reduced 1:5 or 1:8.

3. Fabric made from cotton is grounded on the foulard as described in Example 2 with 1-(2'.3'-hydroxynaphthoylamino)-benzene, dried and then developed on the foulard with a solution containing per 1000 parts by volume of water 30 parts of the acid diazonium sulfate from 1-amino-3-chloroanthraquinone. The material is then passed through the air for ½ to ¾ minute, then rinsed and soaped at boiling temperature; a very clear scarlet red tint of very good properties of fastness is obtained.

The material dyed on the foulard is dried and printed with the following white discharge:

150 grams of sodium formaldehyde sulfoxylate
50 grams of potash
20 grams of sodium benzylsulfanilate
160 grams of water
450 grams of starch tragacanth thickening
75 grams of titanium dioxide 1:1
75 grams of zinc white 1:1
20 grams of anthraquinone paste of 30 per cent strength

1 kilo.

The printed material is steamed for 5 minutes in a rapid ager and after-treated at boiling temperature with a solution containing per liter of water 1 gram of sodium carbonate and 1 gram of the product obtained by the action of ethylene oxide on isooctylphenol.

A pure white is obtained on a scarlet-red ground.

4. 27 parts of 1-(2'.3'-hydroxynaphthoylamino)-benzene are dissolved in hot dilute caustic soda solution, cooled to room temperature and 10 grams of calcined sodium carbonate are added thereto. A solution of 36.6 parts of the acid diazonium sulfate from 1-amino-3-chloroanthraquinone in water is then caused to run in. As soon as diazonium compound can no longer be detected, the solid matter is filtered with suction, washed and dried. A scarlet-red powder is obtained which dissolves in concentrated sulfuric acid to a dark red solution. The coupling may also be conducted in an acid, for instance acetic acid medium. The dyestuff may also be prepared in the presence of a substratum. It has a very good fastness to light, to oils and to overstriping.

The following table indicates a number of other azo-dyestuffs and tints obtainable by this invention:

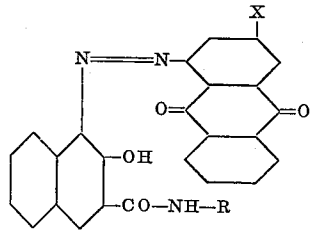
	Diazonium compound from—	Coupled with—	Tint
1	1-amino-3-bromanthraquinone	1-(2'.3'-hydroxynaphthoylamino)-4-chlorobenzene	Scarlet-red.
2	do	4-methoxybenzene	Do.
3	do	2-methyl-4-chlorobenzene	Scarlet.
4	do	2-(2'.3'-hydroxynaphthoylamino)-Naphthalene	Scarlet-red.
5	1-amino-3-chloranthraquinone	1-(2'.3'-hydroxynaphthoylamino)-2-methoxybenzene	Do.
6	do	Naphthalene	Yellowish red.
7	do	2-methyl-4-chlorobenzene	Scarlet.
8	do	4-methoxybenzene	Red.
9	do	4-chlorobenzene	Do.
10	do	2-methylbenzene	Do.
11	1-amino-3-trifluoromethylanthraquinone	Benzene	Scarlet.
12	do	2-methoxybenzene	Do.
13	do	2-methyl-4-chlorobenzene	Do.
14	1-amino-3-methoxyanthraquinone	2-methoxybenzene	Do.
15	do	4-methoxybenzene	Do.
16	do	2-methyl-4-chlorobenzene	Yellowish red.
17	do	2-methylbenzene	Scarlet.
18	do	Benzene	Yellowish red.
19	1-amino-3-methoxyanthraquinone	2-(2'.3'-hydroxynaphthoylamino)-Naphthalene	Do.
20	1-amino-3-methylanthraquinone	1-(2'.3'-hydroxynaphthoylamino)-Benzene	Red.
21	do	2-methoxybenzene	Do.
22	do	2-methylbenzene	Do.
23	do	4-chlorobenzene	Do.
24	do	2-(2'.3'-hydroxynaphthoylamino)-Naphthalene	Do.

Since an object of the present invention is to provide dyestuffs of good fastness properties which dyestuffs are insoluble in water and alkalies, it is to be understood that the aromatic nuclei of the general formulae appearing in the appended claims do not contain any substituents

which are known to render organic compounds soluble in water or alkalies and to tend to depreciate the fastness of the dyestuffs to alkalies. Substituents of this kind are, for instance, the sulfonic acid and the carboxylic acid group.

We claim:

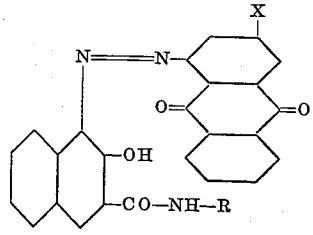
1. The water-insoluble azo-dyestuffs corresponding with the following general formula:



wherein R means a member of the group consisting of radicals of the benzene and naphthalene series and X stands for a member of the group consisting of chlorine, bromine, methoxy, methyl and trifluoromethyl,

being scarlet to red powders which have a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, vivid scarlet and red shades of good properties of fastness, especially of very good fastness to light.

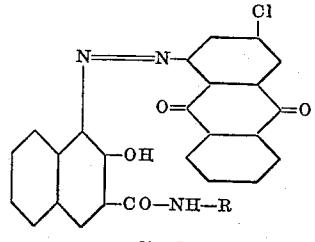
2. The water-insoluble azo-dyestuffs corresponding with the following general formula:



wherein R means a radical of the benzene series and X stands for a member of the group consisting of chlorine, bromine, methoxy, methyl and trifluoromethyl,

being scarlet to red powders which have a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, vivid scarlet and red shades of good properties of fastness, especially of very good fastness to light.

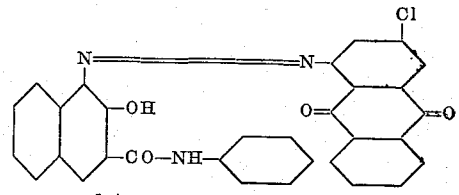
3. The water-insoluble azo-dyestuffs corresponding with the following general formula:



wherein R means a radical of the benzene series,

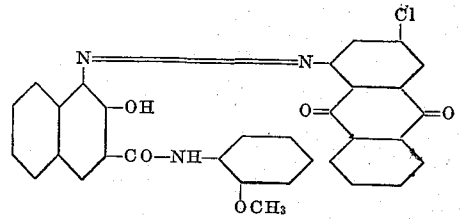
being scarlet to red powders which have a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, vivid scarlet and red shades of good properties of fastness, especially of very good fastness to light.

4. The water-insoluble azo-dyestuff of the following formula:



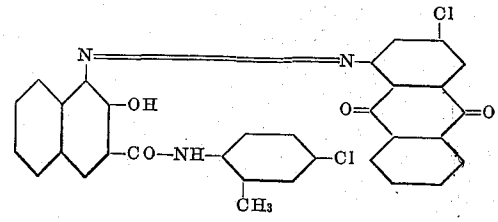
being a scarlet-red powder which has a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, very clear scarlet-red shades of good properties of fastness, especially of very good fastness to light.

5. The water-insoluble azo-dyestuff of the following formula:



being a scarlet-red powder which has a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, very clear scarlet-red shades of good properties of fastness, especially of very good fastness to light.

6. The water-insoluble azo-dyestuff of the following formula:



being a scarlet-red powder which has a very good fastness to light, to oils and to overstriping, and yielding, when produced on the fiber, very clear scarlet shades of good properties of fastness, especially of very good fastness to light.

7. Fiber dyed with the water-insoluble azo-dyestuffs as claimed in claim 1.

8. Fiber dyed with the water-insoluble azo-dyestuffs as claimed in claim 2.

9. Fiber dyed with the water-insoluble azo-dyestuffs as claimed in claim 3.

10. Fiber dyed with the water-insoluble azo-dyestuff as claimed in claim 4.

11. Fiber dyed with the water-insoluble azo-dyestuff as claimed in claim 5.

12. Fiber dyed with the water-insoluble azo-dyestuff as claimed in claim 6.

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