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2,312,398

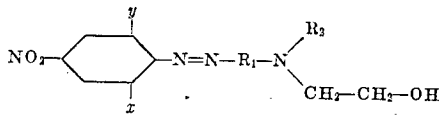
DYESTUFF PREPARATIONS

Friedrich Felix, Basel, Switzerland, assignor to the firm of Society of Chemical Industry in Basle, Basel, Switzerland

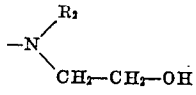
No Drawing. Application May 24, 1940, Serial No. 337,081. In Switzerland June 8, 1939

3 Claims. (Cl. 8—26)

It has been found that acetate rayon may be dyed in shades of blue of surprising tinctorial strength if, for the dyeing process, those dyestuff preparations be used which consist of mixtures of two or more blue dyestuffs, and correspond to the general formula



in which x represents cyanogen, halogen or an SO_2-alkyl group, y represents halogen or a nitro group, R_1 is an aromatic nucleus of the benzene series in which the —N=N-group and the

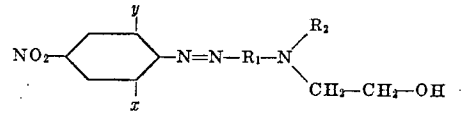


-group stand in the para position to one another, and R_2 stands for hydrogen or an alkyl group.

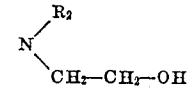
These mixtures of dyestuffs possess a better power of exhaustion when compared with that of the individual components of the mixtures. But those dyestuffs which have, as individuals, a relatively good power of exhaustion may have this property improved quite considerably by incorporating them into mixtures in the manner explained above. The manufacture of the preparations can take place either in the manufacture of the dyestuffs, by making use of a mixture of intermediates, the choice of which is governed by consideration of the general formula mentioned above, or the finished dyestuffs may be dissolved in a common solvent, from which the mixture of dyestuffs is precipitated in a fine state of subdivision, or the finished dyestuffs may be ground together into a finely subdivided state, or the dyestuffs, which are already in a finely dispersed state, may be added to the same dyebath.

Dyestuffs which find application by the present process are described, for example, in U. S. A. Patents Nos. 2,173,417; 2,053,817;2,053,818; 2,082,-156 and 2,155,755. They are prepared according to the usual methods, by use of diazo compounds derived from 1-amino- 2-cyano-6-chloro-4-nitrobenzene, 1 - amino - 2:4-dinitrobenzene-6-methyl sulfone, 1-amino-6-chloro-2:4-dinitrobenzene, 1-amino - 6 - cyano - 2:4 - dinitrobenzene, etc., and coupling components, such as N-ethylhydroxyethylamino-3-methyl-benzene, N - dihydroxyethylamino-3-methylbenzene, N - hydroxyethylamino-3-methylbenzene, N - dihydroxyethylamino-3-methyl-6-methoxybenzene, N - hydroxyethylamino-3-methyl-6-methoxybenzene, as well as N-dihydroxyethylamino - 3 - acetylamino-6-methoxybenzene, etc.

Among these preparations, those are of particular value which contain at least two dyestuffs dyeing blue shades and possessing the general formula



wherein x stands for a member of the group consisting of cyanogen, halogen and SO_2-alkyl, y stands for a member of the group consisting of halogen and NO_2, provided that only one of the two symbols y and x stands for halogen, R_1 stands for an aromatic nucleus of the benzene series in which —N=N-group and the



-group stand in para position to one another and in which a substituent of the group consisting of alkyl, O-alkyl and NH-acyl stands in ortho position to the —N=N-group, and R_2 stands for a member of the group consisting of hydrogen and alkyl. These preparations dye acetate rayon in shades which are of much greater tinctorial strength than those obtained with either of the individual components alone. They may contain 2, 3 or still more single components. Those dyestuffs are particularly valuable in which the radical R_2 stands for methyl, ethyl or hydroxyethyl, and in the manufacture of which compounds such as 1-amino-2-chloro-4-nitro - 6 - cyanobenzene or 1 - amino-2-methyl-sulfone-4:6 - dinitrobenzene are used as diazo components. Dyestuffs of this nature have been described in U. S. A. Patent Nos. 2,173,417; 2,053,817; 2,053,-818; and 2,082,156. These mixtures form the most valuable blue dyestuffs for acetate rayon which have been produced to date.

Example 1

Equal parts of the dyestuff obtained by the union of diazotized 1-amino-2:4-dinitrobenzene-6-methyl sulfone with N-dihydroxyethylamino-3-methylbenzene and of the dyestuff obtained by the union of the same diazo compound with N-hydroxyethylamino-3-methyl-6-methoxybenzene are ground with about 10 parts of sulfite cellulose waste liquor or of another dispersing agent of similar action. The paste thus obtained may be used for the dyeing of acetate rayon. Pastes of this nature can also be converted into preparations of similar behaviour in powder form by careful drying and subsequent grinding.

The blue dyeings obtained with these preparations are considerably more intensive than the dyeings of similar shade which are obtained by use of the individual components.

Example 2

8 parts of a paste containing 10 per cent of its weight of the dyestuff derived from diazotized 1-amino-2:4-dinitrobenzene - 6 - methyl sulfone and N-hydroxyethylamino-3-methyl-6-methoxybenzene and 8 parts of a paste containing 10 per cent of its weight of the dyestuff obtained from the same diazo compound coupled with N-dihydroxyethylamino - 3 - methylbenzene are stirred at 50° C. with about an equal quantity of a soap solution of 6 per cent strength, or preferably with about an equal quantity of a solution of 6 per cent strength of a soap-like substance, and the suspension is diluted with water to 300 parts. 10 parts of acetate rayon are entered into this bath, the temperature is raised during ¾ hour to 75–80° C. and the rayon is worked at this temperature for about ¾ hour further, after which it is rinsed and dried. The acetate rayon is dyed a deep blue shade.

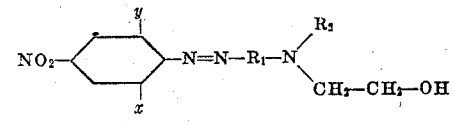
The following dyestuffs may be mentioned as being examples selected from the group of blue-dyeing dyestuffs which can give valuable dyeings when used in mixtures:

	Diazo component	Coupling component	Shade
(1)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-hydroxyethylamino-3-methyl-6-methoxybenzene	Blue.
(2)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-ethoxyethylamino-3-methyl-6-methoxybenzene and N-methylhydroxyethylamino-3-methyl-6-methoxybenzene	Do.
(3)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3-methylbenzene and N-ethylhydroxyethylamino-3-methyl sulfone	Do.
(4)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3-acetylamino-6-methoxybenzene and N-dihydroxyethylamino-3-methyl-6-methoxybenzene	Do.
(5)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-dihydroxyethylamino-3:6-diethoxybenzene	Do.
(6)	(a) 1-amino-2:4-dinitrobenzene-6-methyl sulfone (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3:6-dimethoxybenzene and N-dihydroxyethylamino-3:6-diethoxybenzene	Do.
(7)	(a) 1-amino-2:4-dinitro-6-chlorobenzene (b) 1-amino-2:4-dinitro-6-chlorobenzene	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-hydroxyethylamino-3-methyl-6-methoxybenzene	Do.
(8)	(a) 1-amino-2:4-dinitro-6-chlorobenzene (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-dihydroxyethylamino-3-methyl-6-methoxybenzene	Do.
(9)	(a) 1-amino-2:4-dinitro-6-chlorobenzene (b) 1-amino-2:4-dinitrobenzene-6-methyl sulfone	N-butyl-hydroxyethylamino-3-methylbenzene and N-dihydroxyethylamino-3-methylbenzene	Do.
(10)	(a) 1-amino-2-cyano-6-chloro-4-nitrobenzene (b) 1-amino-2-cyano-6-chloro-4-nitrobenzene	N-dihydroxyethylamino-3-acetylamino-6-methoxy-benzene and N-dihydroxyethylamino-3-methyl-6-methoxybenzene	Do.
(11)	(a) 1-amino-2-cyano-6-chloro-4-nitrobenzene (b) 1-amino-2-cyano-6-chloro-4-nitrobenzene	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-butyl-hydroxyethylamino-3-methylbenzene	Do.
(12)	(a) 1-amino-2:4-dinitrobenzene-6-methylsulfone (b) 1-amino-2:4-dinitrobenzene-6-methylsulfone (c) 1-amino-2:4-dinitrobenzene-6-methylsulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-hydroxyethylamino-3-methyl-6-methoxybenzene and N-ethoxyethylamino-3-methyl-6-methoxybenzene	Do.
(13)	(a) 1-amino-2:4-dinitrobenzene-6-methylsulfone (b) 1-amino-2:4-dinitrobenzene-6-methylsulfone (c) 1-amino-2:4-dinitrobenzene-6-methylsulfone (d) 1-amino-2:4-dinitrobenzene-6-methylsulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-hydroxyethylamino-3-methyl-6-methoxybenzene and N-ethoxyethylamino-3-methyl-6-methoxybenzene and N-methylhydroxyethyl-amino-3-methyl-6-methoxybenzene	Do.
(14)	(a) 1-amino-2:4-dinitrobenzene-6-methylsulfone (b) 1-amino-2:4-dinitrobenzene-6-methylsulfone (c) 1-amino-2:4-dinitrobenzene-6-ethylsulfone	N-dihydroxyethylamino-3-methyl-6-methoxybenzene and N-hydroxyethylamino-3-methyl-6-methoxybenzene and N-ethoxyethylamino-3-methyl-6-methoxybenzene	Do.

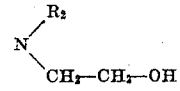
It is naturally also possible to mix the individual dyestuffs within the confines of the said group as desired, that is to say, they need not be mixed only in the mixtures given in the above table.

What I claim is:

1. Dyestuff preparations containing at least two dyestuffs dyeing blue shades of the general formula



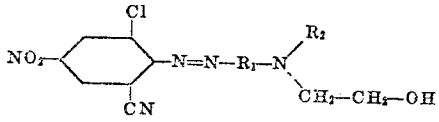
wherein x stands for a member of the group consisting of cyanogen, halogen, and SO_2-alkyl, y stands for a member of the group consisting of halogen and NO_2, provided that only one of the two symbols y and x stands for halogen, R_1 stands for an aromatic nucleus of the benzene series in which the —N=N-group and the



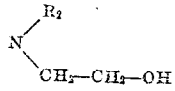
-group stand in para position to one another and in which a substituent of the group consisting of alkyl, O-alkyl and NH-acyl stands in ortho position to the —N=N-group, and R_2 stands for a member of the group consisting of CH_3, C_2H_5

and CH_2CH_2OH, which dyestuff preparations dye acetate rayon in shades of a more increased tinctorial power than the individual components.

2. Dyestuff preparations containing at least two dyestuffs dyeing blue shades of the general formula

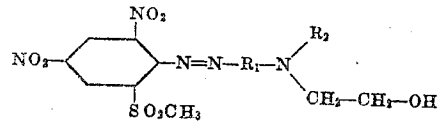


wherein R_1 stands for an aromatic nucleus of the benzene series in which the —N=N-group and the

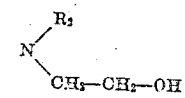


-group stand in para position to one another and in which a substituent of the group consisting of alkyl, O-alkyl and NH-acyl stands in ortho position to the —N=N-group, and R_2 stands for a member of the group consisting of CH_3, C_2H_5 and CH_2—CH_2—OH, which dyestuff preparations dye acetate rayon in shades of a more increased tinctorial power than the individual components.

3. Dyestuff preparations containing at least two dyestuffs dyeing blue shades of the general formula



wherein R_1 stands for an aromatic nucleus of the benzene series in which the —N=N-group and the



-group stand in para position to one another and in which a substituent of the group consisting of alkyl, O-alkyl and NH-acyl stands in ortho position to the —N=N-group, and R_2 stands for a member of the group consisting of CH_3, C_2H_5 and CH_2—CH_2—OH, which dyestuff preparations dye acetate rayon in shades of a more increased tinctorial power than the individual components.

FRIEDRICH FELIX.