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(54) **Monoazo dyes of the benzothiazole series, their preparation and use in dyeing or printing hydrophobic fibres.**

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Monoazo dyes of the benzothiazole series, their preparation and use in dyeing or printing hydrophobic fibres.

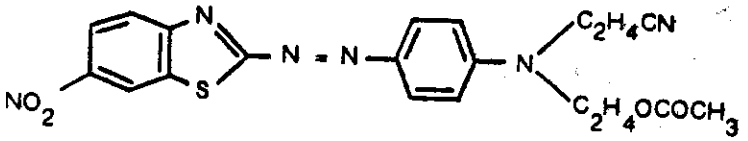
The present invention relates to novel benzothiazole monoazo dyes suitable useful for dyeing or printing hydrophobic fibers.

With the recent tendency to higher quality, more expensive and fashionable hydrophobic fibers, particularly polyester fibers, the colored discharge printing system has come to assume greater prominence among other dyeing techniques. This system is characterized by its ability to produce delicate and bold expressions of printing patterns and its uniqueness in application of such patterns with vivid and fresh colors on various types of textile fabrics.

The dyes usable in this system can be divided into two types: dischargeable dyes for ground-dyeing and non-dischargeable dyes for effect color of printing. Among them, the latter type of dyes pose no serious problem in their use because of good color fastness of the existing dyes of this type. As for the dischargeable ground-dyeing dyes, they should meet requirements such that they are perfectly decomposed to become colorless by a discharging treatment or even if the decomposed matter after the discharging treatment should retain color, such color should be easily eliminated, and they should be superior in various fastnesses.

It is difficult to select one which can meet such requirements from existing dyes, and it has been proposed to develop a new dye, but so far, no satisfactory result has been reached in these attempts.

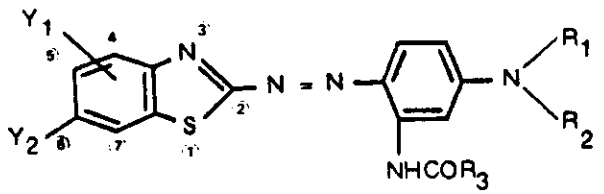
For example, a dye of the following formula:



proposed in Japanese Patent Publication No. 16039/61 is used as red dye, but this dye is still unsatisfactory in the aspects of hue and color fastness. Thus, request has been voiced for the development of a dischargeable red or violet dyestuff which has excellent dyeability as well as various good fastness properties, particularly wet color fastness after resin finishing.

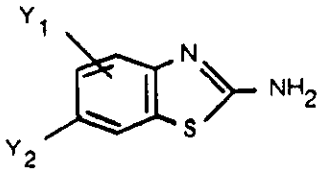
As a result of more extensive studies for the development of a dischargeable red or violet dyestuff usable for ground dyeing, the present inventors found novel dyes having very excellent dischargeability, dyeing affinity, hue, build-up property, color fastnesses to light, sublimation and potting fastness, as well as wet fastness to washing after resin finishing, domestic laundering, water.

The present invention provides a benzothiazole monoazo dye of the formula (I),



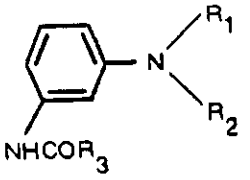
[I]

wherein R_1 is an n- or iso-pentyl or n-hexyl group, R_2 is a cyanoethyl group, R_3 is a hydrogen atom or an alkyl group having 1 to 4 carbon atoms, amino or phenyl group, and Y_1 and Y_2 represent independently a hydrogen atom, an alkyl group having 1 to 4 carbon atoms, an alkoxy group having 1 to 4 carbon atoms, a halogen atom, a nitro group, a cyano group, a thiocyanate group or an alkylsulfonyl group, provided that both Y_1 and Y_2 are not hydrogen atom at the same time and Y_1 is located at the 4- or 5-position; a process for preparing the same comprising diazotizing a 2-aminobenzothiazole compound represented by the formula (II):



[II]

wherein Y_1 and Y_2 are as defined above, and coupling resulting diazotized compound with a compound represented by the formula (III):



[III]

wherein R_1, R_2 and R_3 are as defined above; and a process for dyeing or printing hydrophobic fibers comprising using the dye of the formula (I).

In the present invention, R_1 is an n- or iso-pentyl or n-hexyl group, and of these the most preferred is n-pentyl. Preferred examples for R_3 are hydrogen, methyl, ethyl and phenyl, and of these the most preferred is methyl. Preferred examples for Y_1 include hydrogen, and halogens (e.g. chlorine or bromine) and for Y_2 include methoxy, halogens (e.g. chlorine, bromine), nitro, cyano and alkylsulfonyls (e.g. methylsulfonyl, ethylsulfonyl), and the most preferred for Y_1 and Y_2 is hydrogen and chlorine, respectively.

In the production of the dye of the formula (I), diazotization of the 2-aminobenzothiazole compounds represented by the formula (II) in this invention can be accomplished at a relatively low temperature, preferably at −10° to 10°C within 2 to 8 hours by using sodium nitrite or nitrosylsulfuric acid in an amount of 1.0 to 1.1 mole per mole of the compound (II) in a mineral acid (such as hydrochloric acid, sulfuric acid, phosphoric acid) or an organic acid (such as acetic acid, propionic acid).

Listed below are some typical examples of said 2-aminobenzothiazole compounds:

2-amino-6-nitrobenzothiazole
2-amino-6-methylsulfonylbenzothiazole
2-amino-6-methoxybenzothiazole
2-amino-6-ethoxybenzothiazole
2-amino-6-chlorobenzothiazole
2-amino-6-bromobenzothiazole
2-amino-6-iodobenzothiazole
2-amino-6-cyanobenzothiazole
2-amino-6-thiocyanobenzothiazole
2-amino-6-methylbenzothiazole
2-amino-4,6-dichlorobenzothiazole
2-amino-4,6-dibromobenzothiazole
2-amino-5,6-dichlorobenzothiazole
2-amino-5,6-dibromobenzothiazole
2-amino-4-chloro-6-nitrobenzothiazole
2-amino-4-bromo-6-nitrobenzothiazole
2-amino-4-methyl-6-nitrobenzothiazole
2-amino-4-methoxy-6-nitrobenzothiazole
2-amino-4-cyano-6-nitrobenzothiazole
2-amino-4-nitro-6-chlorobenzothiazole
2-amino-4-cyano-6-chlorobenzothiazole

Coupling of the diazotized 2-aminobenzothiazole compound of the formula (II) and the compound of the formula (III) is performed in an aqueous medium. In order to elevate solubility of the compound of the formula (III), there may be used an alcoholic organic solvent such as methanol, ethanol, propanol, butanol, or an organic acid such as acetic acid, propionic acid, or a mineral acid such as hydrochloric acid, sulfuric acid, phosphoric acid. The coupling can be accomplished at a relatively low temperature, preferably at −10° to 10°C, within 2 to 10 hours using the compound (III) in an amount of 1.0 to 1.1 mole per mole of the diazotized compound (II).

As examples of the compounds represented by the formula (III), there may be cited the following:

3-(N-n-pentyl-N-ß-cyanoethyl)aminoacetoanilide.

The dye produced in the manner described above is pulverized into fine particles usually in an aqueous medium together with a suitable dispersant and used in the form of a paste or in the form of a powder prepared by spray drying.

Among the examples of the dyes represented by the formula (I) are such as those listed in Table I below.

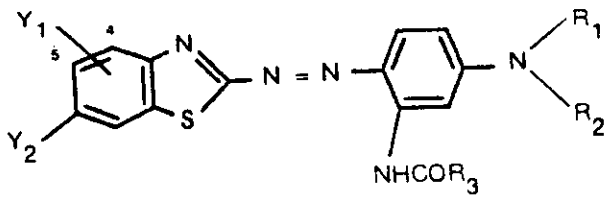


TABLE 1

No.	Y_1	Y_2	R_1	R_2	R_3	DMF λ max (nm)
1	H	Cl	$n\text{-}C_5H_{11}$	C_2H_4CN	CH_3	530
2	H	Br	,,	,,	,,	530
3	4-Cl	Cl	,,	,,	,,	532
4	4-Br	Br	,,	,,	,,	532

The dyes of the formula (I) obtained according to the above-described process can be advantageously used for dyeing or printing hydrophobic fibers, particularly polyester fibers to give a brilliant red to violet color with excellent color fastnesses to light, sublimation, as well as excellent color fastness to domestic laundering, water, after resin finishing. What is especially noteworthy is their splendid availability as ground-dyeing dyes for the colored discharge printing system. An additional advantage of the dyes of the formula (I) is their extremely high molar absorbance.

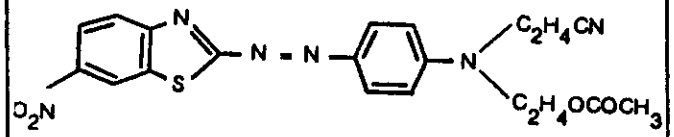
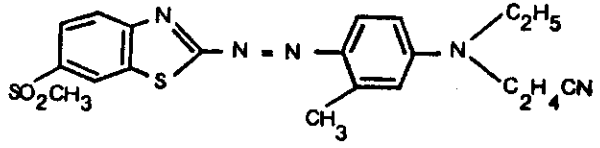
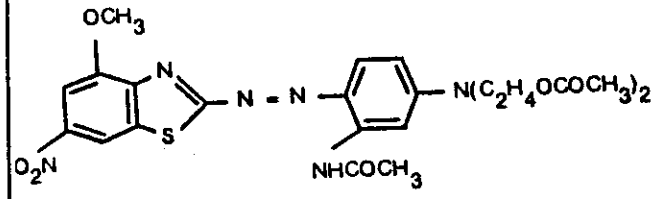
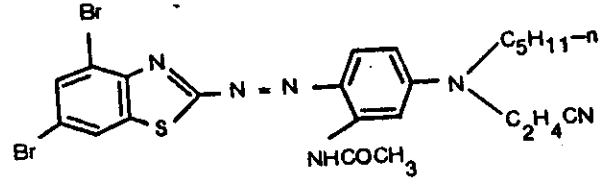
In carrying out the dyeing of hydrophobic fibers using the dyes of the present invention, the dye is first pulverized into fine particles in an aqueous medium together with a suitable dispersant to prepare a paste-like dye agent or a powdery dye agent by spray drying of the paste or other means. The fibers are dipped in an aqueous medium containing the dye agent, and the dyeing is carried out at a temperature of 105°C or higher, preferably 110°—140°C under increased pressure, or the dyeing may be carried out in the presence of a carrier such as o-phenylphenol or trichlorobenzene at a relatively high temperature, for example under a boiling water condition.

Further, the so-called thermosol method may also be applied, that is, hydrophobic fiber cloth may be dyed by padding the cloth with a dye disperse liquor followed by dry-heat treatment at 150° to 230°C for 30 to 60 seconds.

Still further, the dye (I) of the present invention may effectively be applied to printing methods which comprise printing the cloth with a color paste produced from a dye (I)-containing dispersion and a suitable thickening agent, followed by steaming or thermosol treatment. Also, the dye (I) may be applied to solvent dyeing methods using an organic solvent (e.g. trichloroethylene, perchloroethylene) as a dyeing medium.

Shown below are the results of comparative tests conducted using the dyes of the present invention and the known dyes of similar type. It will be understood from Table 2 that the dyes of this invention are superior to the known dyes in hue, dischargeability and color fastness after resin finishing.

TABLE 2

	Formula	Hue (Brilliancy)	Discharge-ability (white discharge-ability)	Color fastness after resin finishing					
				Washing			Water		
				Discolor-ation	Nylon staining	Silk staining	Discolor-ation	Nylon staining	Silk staining
Known dye	 Dye of Japanese Pat. Pub. No. 16039/61	Δ	Δ	5	4	3	5	2–3	2–3
	 Dye of Japanese Pat. Pub. No. 5949/66	Δ	Δ	5	4–5	3–4	5	3	2–3
	 Dye of Japanese Pat. Pub. No. 7712/70	Δ	Δ	5	3–4	3–4	5	3	2–3
Dye of this invention	 (No. 14)	O	◎	5	4–5	4–5	5	4–5	4–5

Notes:

(1) Dyeing method: High-temperature dyeing

Material to be dyed: Polyester fabric

Dye concentration: 3.0% owf

Dyeing conditions: pH 5.0 (acetic acid-sodium acetate buffer), 130°C, 60 min.

Hue was judged according to visual test: (◎: excellent; O: good; Δ: rather poor; x: poor)

(2) Resin treatment (finishing)

Sumistat F-1 (antistatic agent mfd. by Sumitomo Chemical Co.) 10 g/l

Sumitex Softener LK-1 (softener, manufactured by Sumitomo Chemical Co.) 10 g/l

Procedure of immersion and squeezing was repeated twice.

Heat setting:

Intermediate drying conditions: 80°C, 2 min.

Heat-setting conditions: 160°C, 2 min.

(3) Fastness

Color fastness to washing: AATCC No. II-A

Color fastness to water: JIS L0846-1967 A

(4) White discharging

White discharging paste composition (stannous chloride method)

Stannous chloride		10 parts by weight	
Urea		3 parts ,,	
Tetorocin p-300	(↓)	3 parts ,,	(carrier mfd. by Yamakura Chemicals Co.)
Glyecin A	(↓)	3 parts ,,	(Relubitizing additive agent mfd. by BASF AG)
12% Maypro gum NP 60	(↓)	60 parts ,,	(thickening agent mfd. by Meyahll Co.)
Hot water		21 parts ,,	
Total		100 parts ,,	

White discharging process:

Printing → drying → steaming (130°C, 30 min.) → washing with water →

reduction clearing → washing with water → drying

Judgment:

The degree of tinting at the white-discharged parts was determined according to the gray scale for staining. (◎: excellent; O: good; Δ; rather poor; X: poor)

The invention is now described in further detail with reference to the following Examples, which are only illustrative and are not intended to limit the scope of the present invention. All the "parts" appearing in the following Examples are by weight.

Example 1

9.2 Parts of 2-amino-6-chlorobenzothiazole was added and dispersed in 100 parts of 40% sulfuric acid, and the mixture was cooled below 0°C by external cooling and further added dropwise with 14.8 parts of 43% nitrosylsulfuric acid at less than 0°C over the period of 1 hour. The mixture was kept below 0°C for 2 hours to complete the diazotization. In the meantime, 13.7 parts of 3-(N-n-pentyl-N-ß-cyanoethyl)aminoacetoanilide was added and dissolved in 50 parts of acetic acid, followed by addition of 100 parts of ice to cool the solution below 0°C, and the above-said diazo solution was poured thereinto over the period of 1 hour while cooling below 0°C to effect coupling. The mixture was kept below 0°C for 2 hours to complete coupling.

The precipitated dye was separated by filtration, washed with 200 parts of water and dried, whereby there was obtained 21.1 parts of dark red crystals.

The thus obtained crystals had the structure of No. 1 in Table 1, melting point of 162—163°C and molar absorption coefficient of as high as 5.9×10^4 $1 \cdot cm^{-1} \cdot mol^{-1}$.

Example 2

The process of Example 1 was repeated but by using 11.0 parts of 2-amino-4,6-dichlorobenzothiazole instead of 9.2 parts of 2-amino-6-chlorobenzothiazole, obtaining the dark red crystals having the structure of No. 3 in Table 1, melting point of 165—167°C and molar absorption coefficient of as high as 6.0×10^4 $1 \cdot cm^{-1} \cdot mol^{-1}$.

Example 3

2.0 Parts of sodium lignosulfonate was added to 1.0 part of the powdery dyestuff of No. 1 in Table 1, and the mixture was finely pulverized and dispersed in an aqueous medium, and this dispersion was dried to obtain a dyeing agent. Then 0.3 part of this dyeing agent was added into a dye bath containing 1.0 part of O-phenylphenol as carrier, and 10 parts of polyethylene terephthalate fibers was immersed in this dye bath and subjected to 90-minute dyeing at 98—100°C. Upon soaping of these fibers at 90°C, there were obtained polyester fibers which have been dyed to clear deep violet with high dye bath stability and had excellent color fastnesses to daylight and sublimation, as well as high wet fastnesses to water and washing after resin finishing.

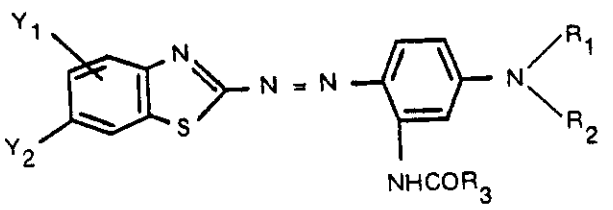
Example 4

2.0 Parts of a condensate of naphthalene-sulfonic acid and formaldehyde was added to 1.0 part of the powdery dyestuff of No. 3 in Table 1, and the mixture was finely pulverized and dispersed in an aqueous medium. The obtained dispersion was kneaded with a suitable paste and water, then padded on a polyester fabric and, after drying, subjected to 7-minute steaming at 175°C. This fabric was then subjected to soaping at 90°C to obtain with high dye bath stability a clear red dyed product having excellent color fastnesses to daylight and sublimation, as well as high wet fastnesses to water and washing after resin finishing.

When the above process was repeated by using No. 4 dyestuff instead of No. 3 dyestuff, there was obtained a polyester fabric dyed in brilliant red.

Claims

1. A compound characterised in that it has the formula (I):

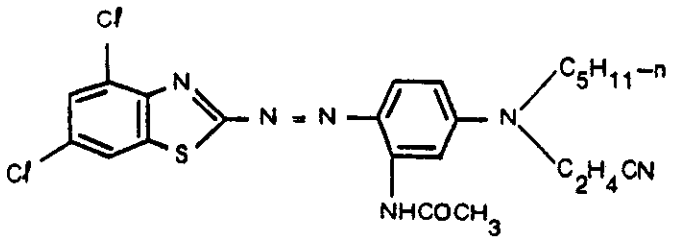


(I)

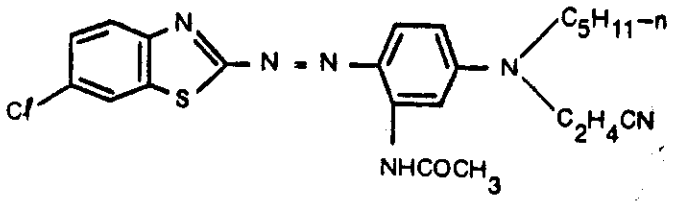
wherein R_1 is an n- or iso-pentyl or n-hexyl group, R_2 is a cyano ethyl group, R_3 is a hydrogen atom or an alkyl group having 1 to 4 carbon atoms, amino or phenyl group, and Y_1 and Y_2 represent independently a hydrogen atom, an alkyl group having 1 to 4 carbon atoms, an alkoxy group having 1 to 4 carbon atoms, a halogen atom, a nitro group, a cyano group, a thiocyanate group or an alkylsulfonyl group, provided that both Y_1 and Y_2 are not hydrogen at the same time and Y_1 is located at the 4- or 5-position.

2. A compound according to claim 1 characterised in that R_1 and R_2 in the formula (I) are an n- or iso-pentyl group and a cyanoethyl group, respectively, and R_3 is a methyl group.

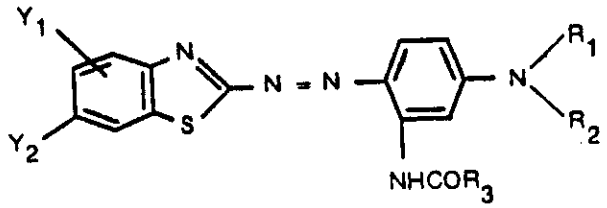
3. A compound according to claim 1 characterised in that it has the formula:



4. A compound according to claim 1 characterised in that it has the formula:

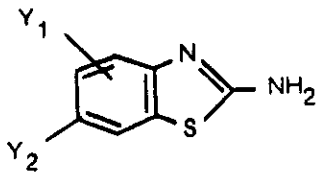


5. A process for preparation of a compound of the formula (I):



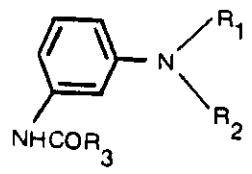
(I)

wherein R_1 is an n- or iso-pentyl or n-hexyl group, R_2 is a cyanoethyl group, R_3 is a hydrogen atom or an alkyl group having 1 to 4 carbon atoms, amino or phenyl group, and Y_1 and Y_2 represent independently a hydrogen atom, an alkyl group having 1 to 4 carbon atoms an alkoxy group having 1 to 4 carbon atoms, a halogen atom, a nitro group, a cyano group, a thiocyanate group or an alkylsulfonyl group, provided that both Y_1 and Y_2 are not hydrogen at the same time and Y_1 is located at the 4- or 5-position, the process characterised in that it comprises coupling a diazotized 2-aminobenzothiazole compound of the formula (II):



(II)

wherein Y_1 and Y_2 are as defined above with a compound of the formula (III):



(III)

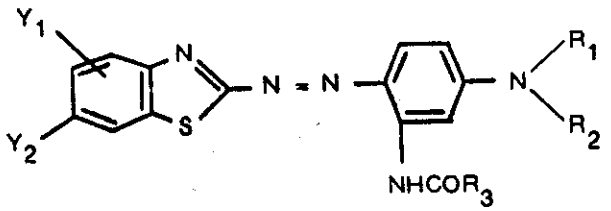
wherein R_1, R_2 and R_3 are as defined above.

6. A process for dyeing or printing hydrophobic fibers characterised in that the process uses a dye as claimed in any of claims 1 to 4.

7. Hydrophobic fibers characterised in that they have been dyed or printed by the process of claim 6.

Patentansprüche

1. Verbindung der Formel:

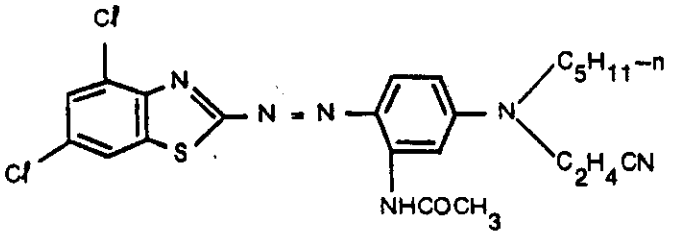


(I)

worin bedeuten: R_1 eine n- oder iso-Pentyl- oder n-Hexylgruppe; R_2 eine Cyanoethylgruppe; R_3 ein Wasserstoffatom, eine Alkylgruppe mit 1—4 Kohlenstoffatom(en), eine Aminogruppe oder eine Phenylgruppe und Y_1 und Y_2 jeweils ein Wasserstoff- oder Halogen atom, eine Alkylgruppe mit 1—4 Kohlenstoffatom(en), eine Alkoxygruppe mit 1—4 Kohlenstoffatom(en), eine Nitrogruppe, eine Cyanogruppe, eine Thiocyanatgruppe oder eine Alkylsulfonylgruppe, wobei gilt, daß nicht beide Reste Y_1 und Y_2 gleichzeitig Wasserstoffatome darstellen dürfen und der Rest Y_1 sich in 4- oder 5-Stellung befindet.

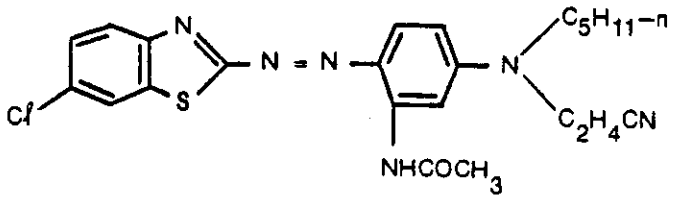
2. Verbindung nach Anspruch 1, dadurch gekennzeichnet, daß in der Formel (I) R_1 für eine n- oder iso-Pentylgruppe steht, R_2 eine Cyanoethylgruppe darstellt und R_3 eine Methylgruppe bedeutet.

3. Verbindung nach Anspruch 1, dadurch gekennzeichnet, daß sie der Formel:



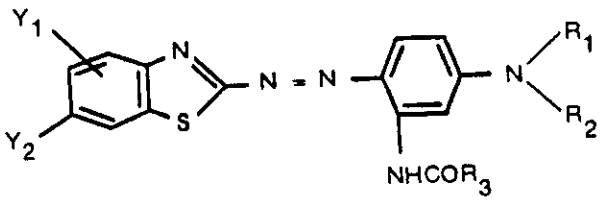
entspricht.

4. Verbindung nach Anspruch 1, dadurch gekennzeichnet, daß sie der Formel:



entspricht.

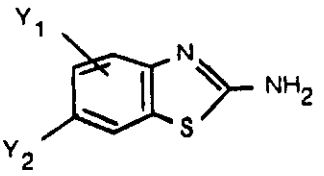
5. Verfahren zur Herstellung einer Verbindung der Formel



(I)

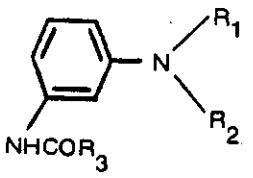
worin bedeuten: R_1 eine n- oder iso-Pentyl- oder n-Hexylgruppe; R_2 eine Cyanoethylgruppe; R_3 ein Wasserstoffatom, eine Alkylgruppe mit 1—4 Kohlenstoffatom(en), eine Aminogruppe oder eine Phenylgruppe und Y_1 und Y_2 jeweils ein Wasserstoff- oder Halogenatom, eine Alkylgruppe mit 1—4

Kohlenstoffatom(en), eine Alkoxygruppe mit 1—4 Kohlenstoffatom(en), eine Nitrogruppe, eine Cyanogruppe, eine Thiocyanatgruppe oder eine Alkylsulfonylgruppe, wobei gilt, daß nicht beide Reste Y_1 und Y_2 gleichzeitig Wasserstoffatome darstellen dürfen und der Rest Y_1 sich in 4- oder 5-Stellung befindet, dadurch gekennzeichnet, daß man eine diazotierte 2-Aminobenzothiazolverbindung der Formel:



[II]

worin Y_1 und Y_2 die angegebene Bedeutung besitzen, mit einer Verbindung der Formel:



[III]

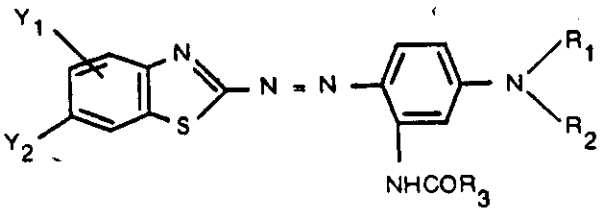
worin R_1, R_2 und R_3 die angegebene Bedeutung besitzen, kuppelt.

6. Zum Anfärben oder Bedrucken hydrophober Fasern, dadurch gekennzeichnet, daß man sich hierbei eines Farbstoffs nach einem der Ansprüche 1—4 bedient.

7. Hydrophobe Fasern, dadurch gekennzeichnet, daß sie nach dem Verfahren des Anspruchs 6 gefärbt oder bedruckt sind.

Revendications

1. Composé caractérisé en ce qu'il répond à la formule (I)

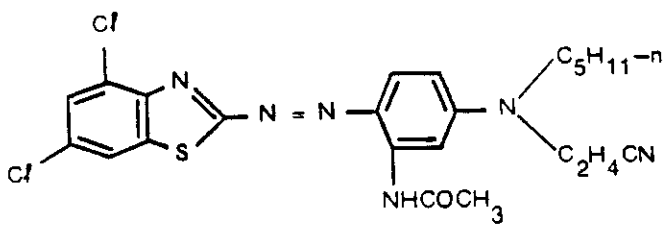


(I)

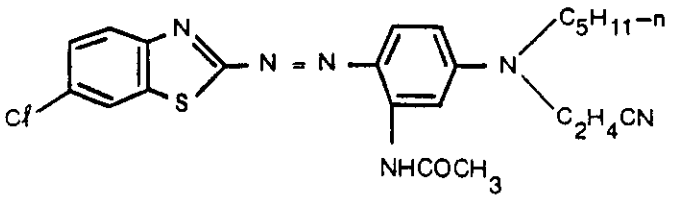
dans laquelle R_1 est un groupe n- ou iso-pentyle ou n-hexyle, R_2 est un groupe cyanéthyle, R_3 est un atome d'hydrogène ou un groupe alcoyle comportant 1 à 4 atomes de carbone, un groupe amino ou phényle et Y_1, Y_2 représentent, indépendamment l'un de l'autre, un atome d'hydrogène, un groupe alcoyle comportant 1 à 4 atomes de carbone, un groupe alcoxy comportant 1 à 4 atomes de carbone, un atome d'halogène, un groupe nitro, un groupe cyano, un groupe thiocyanate ou un groupe alcoylsulfonyle, avec cette condition que Y_1 et Y_2 ne soient pas tous les deux des atomes d'hydrogène en même temps et que Y_1 soit situé en position 4 ou 5.

2. Composé selon la revendication 1, caractérisé en ce que R_1 et R_2 dans la formule (I) sont respectivement un groupe n- ou iso-pentyle et un groupe cyanéthyle et en ce que R_3 est un groupe méthyle.

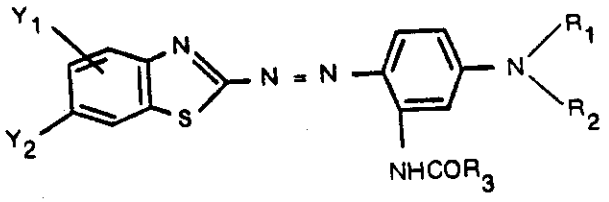
3. Composé selon la revendication 1, caractérisé en ce qu'il répond à la formule



4. Composé selon la revendication 1, caractérisé en ce qu'il répond à la formule

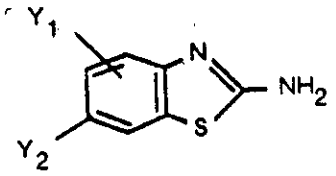


5. Procédé pour la préparation d'un composé de formule (I)



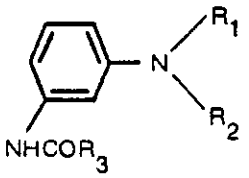
(I)

dans laquelle R_1 est un groupe n- ou iso-pentyle ou n-hexyle, R_2 est un groupe cyanéthyle, R_3 est un atome d'hydrogène ou un groupe alcoyle comportant 1 à 4 atomes de carbone, un groupe amino ou phényle et Y_1, Y_2 représentent, indépendamment l'un de l'autre, un atome d'hydrogène, un groupe alcoyle comportant 1 à 4 atomes de carbone, un groupe alcoxy comportant 1 à 4 atomes de carbone, un atome d'halogène, un groupe nitro, un groupe cyano, un groupe thiocyanate ou un groupe alcoylsulfonyle, avec cette condition que Y_1, Y_2 ne soient pas tous les deux des atomes d'hydrogène en même temps et que Y_1 soit situé en position 4 ou 5, caractérisé en ce qu'il comprend les opérations consistant à copuler un composé 2-aminobenzothiazolique diazoté de formule (II)



[II]

dans laquelle Y_1 et Y_2 ont les significations données ci-dessus, avec un composé de formule (III)



[III]

dans laquelle R_1, R_2 et R_3 ont les significations données ci-dessus.

6. Procédé pour la teinture ou l'impression de fibres hydrophobes, caractérisé en ce qu'il est fait usage d'un colorant selon l'une quelconque des revendications 1 à 4.

7. Fibres hydrophobes, caractérisées en ce qu'elles ont été teintes ou imprimées par le procédé selon la revendication 6.

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03.10.79/ JP 128308/79

(54)
Monoazofarbstoffe der Benzothiazolreihe, ihre Herstellung und Verwendung beim Färben und Bedrucken von hydrophoben Fasern
Monoazo dyes of the benzothiazole series, their preparation and use in dyeing or printing hydrophobic fibres
Colorants monoazoiques de la série des benzothiazoles, leur préparation et leur utilisation dans la teinture et l'impression de fibres hydrophobes

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frühere Anmeldung/earlier application/demande initiale :

Zeichen/ref/ref : PA9058-/MRH/PMH

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