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DYESTUFFS

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10 Claims. (Cl. 260—163)

This invention relates to new dyestuffs, and especially to new dyestuffs containing a long chain alkyl group.

According to our invention we make new dyestuffs containing a long chain alkyl group (C_8—C_{20}) by introducing this group, by means of an alkyl-aryl group. This introduction may be effected by known process for making dyestuffs containing tolyl and xylyl groups.

The new dyestuffs of our invention containing long-chain alkyl groups have improved properties, especially that of yielding dyeings which have improved fastness, including improved fastness to washing and milling. The new dyestuffs of our invention having the long alkyl chain directly attached to an aryl group, have this long alkyl chain very firmly attached and in consequence the dyestuffs have great chemical stability.

More specifically, according to the invention we make new dyestuffs containing a long chain alkyl (C_8—C_{20}) group by introducing into compounds already having the properties of dyestuffs, or using as an intermediate in the manufacture of dyestuffs a compound containing an alkylaryl group RR′ where R stands for a long chain alkyl (C_8—C_{20}) and R′ stands for phenylene, naphthylene, acenaphthylene or the like. Thus for example in carrying the invention into practical effect, we may use a long-chain alkylarylamine, such as an alkyl phenylamine or an alkyl naphthylamine, or we may use a long chain alkyl phenol, such as an alkyl phenol, or an alkyl naphthol, or we may use a long chain alkyl benzoic acid, a long chain alkyl benzyl alcohol or a long chain alkyl benzoyl halide; and we may carry the combination into effect according to the known methods of using similar intermediates, free from long chain alkyls, in making dyestuffs for instance, by diaotization and coupling, condensation, acylation, arylation and the like.

Thus with a long chain alkyl arylamine we may diazotize and couple the diazonium salt with a pyrazolone, a phenol, a naphthol, a phenolsulfonic acid, a naphthol sulfonic acid, an aniline, an aniline sulfonic acid, a naphthylamine, a naphthylaminesulfonic acid, an aminophenol, an aminophenolsulfonic acid, an aminonaphthol, an aminonaphtholsulfonic acid or other coupling component. Also with a long chain alkyl arylamine we may condense with an aminonaphthol a similar compound, for example H, S, Y and γ acids, under such conditions that the amino group is replaced by the alkylaryl group RR′ and then couple the so-produced long-chain alkyl arylaminonaphtholsulfonic acid or similar compound with a diazotised amine.

In employing a long chain alkyl benzoyl halide we may acylate an aminonaphthol sulfonic acid —H, J, S, J—or similar acid and couple the long chain alkylbenzoylamino-naphthol-sulfonic acid with a diazotised amine.

It will be understood that when necessary, one or more sulfonic or other water-solubilising groupings may be introduced at one stage or other in the manufacture.

The new dyestuffs when containing a sufficiency of sulfonic or carboxylic acid groups dissolve in water to give solutions showing a tendency to gel. When applied in the usual way as acid dyestuffs they show remarkable melting and potting fastness, when compared with analogous dyestuffs not containing the characteristic long chain alkyl groups. Because of this they are adapted for use in place of chrome dyestuffs.

The invention also includes the new compounds obtainable by the above process, including dyestuffs containing an alkyl aryl group RR′ hereinbefore defined, and various species belonging to this group as well as the individual dyestuffs described herein.

The invention also includes the application of the new dyestuff.

The following examples in which parts are by weight, illustrate, but do not limit the invention.

Example 1

26.1 parts of p-dodecylaniline (made in a similar way to that used for p-octylaniline by Beran, Ber. d. deutschen chem. Ges. 1885, 18, 132) are stirred for 15 minutes at the ordinary temperature with 250 parts of hydrochloric acid (36%) and 300 parts of water. The mixture is then warmed to 50° C. until a homogeneous paste is formed and then enough ice added to cool the whole to 5° C. Diazotisation is done at this temperature by adding slowly 69 parts of a 10% aqueous solution of sodium nitrite, whereby a pale greenish-yellow solution is obtained. When the diazotisation is complete the diazo compound is added with stirring to a solution of 25.4 parts of 1(4′-sulfophenyl)-3-methyl-5-pyrazolone in 200 parts of water, to which have been added 25 parts of sodium carbonate. Coupling is rapid and when finished, the new dyestuff, which is partly out of solution as a bright yellow suspension, is completely precipitated by adding common salt and making acid to litmus with hydrochloric acid. It is then filtered off, dried and

ground to a yellow powder. It is soluble in water with a yellow colour. It dyes wool in bright yellow shades of very good fastness to severe washing, rubbing and light.

Example 2

26.1 parts of p-dodecylaniline are diazotized as in Example 1 and the cooled solution of the diazo compound added at 5° C. to a solution of 34.5 parts of the sodium salt of 1-(2':5'-dichloro-4'-sulfo) phenyl-3-methyl-5-pyrazolone in 400 parts of water containing 21 parts of sodium carbonate. Coupling is rapid, the dyestuff being at first in the form of a gel. After stirring for some hours the dyestuff becomes granular and is filtered off, washed with 5% salt solution and dried. It dyes wool in greenish-yellow shades of very good fastness to washing, milling and light.

Example 3

In the above example, 33 parts of the disodium salt of 1-(4'-sulfo) phenyl-3-carboxy-5-pyrazolone are used instead of 1-(2':5'-dichloro-4'-sulfo phenyl-3-methyl-5-pyrazolone. A dyestuff is obtained which dyes wool in much redder shades of yellow and of very good fastness to severe washing, milling and light.

Example 4

A diazo solution obtained as in Example 1 from 26.1 parts of p-dodecylaniline is added to a solution of 11.0 parts of 1:3-dihydroxybenzene in 200 parts of water containing 8 parts of sodium hydroxide. On completion of coupling (about 2 hours), the diazo solution obtained in the usual way from 25.3 parts of 1-aminobenzene-2:5-disulfonic acid is added, followed by enough caustic soda to make alkaline to litmus paper again. The second coupling is allowed to complete in about 12 hours and the new disazo dyestuff then precipitated by adding enough hydrochloric acid to colour Congo red paper. The dyestuff is then filtered off, and dried. When ground it is a dark brown powder soluble in water with a brown colour.

It dyes wool in orange-brown shades of good fastness to severe washing, milling and light.

Example 5

26.1 parts of p-dodecylaniline are diazotised as described in Example 1 and the diazo solution added to a solution of 246 parts of the sodium salt of 2-hydroxy-naphthalene-8-sulfonic acid in 350 parts of water, containing 21 parts of sodium carbonate. Coupling is rapid and when complete 30 parts of salt are added and the dyestuff filtered off, washed with 5% salt solution and dried. The new dyestuff dyes wool from an acid bath in clear orange shades of very good fastness to severe washing and milling.

Example 6

The diazo solution obtained as in Example 1 from 26.1 parts of p-dodecylaniline is added with stirring to a solution of 30.4 parts of 2-naphthol-6:8-disulfonic acid in 200 parts of water containing 30 parts of sodium carbonate. On completion of the coupling the new dyestuff, which is practically all out of solution, is filtered off, pressed well and dried. When ground, it is a dark orange coloured powder soluble in water with an orange colour.

It dyes wool in orange shades of very good fastness to severe washing, milling and light.

Example 7

If in Example 5, 348 parts of the disodium salt of 1-hydroxynaphthalene-3:6-disulfonic acid are used instead of 2-hydroxynaphthalene-8-sulfonic acid, a dyestuff is obtained which dyes wool from an acid bath in redder shades of orange, which also have very good fastness to severe washing and milling.

Example 8

26.1 parts of p-dodecylaniline are diazotised as in Example 1 and the diazo solution added at 5° C. to a solution of 30.3 parts of 2-naphthylamine-6:8-disulfonic acid in 320 parts of water containing 8 parts of caustic soda and then adding 21 parts of anhydrous sodium carbonate. When coupling is complete the mixture is heated to 50° C. and the dyestuff precipitated with about 10% salt and filtered off. It dyes wool in reddish-yellow shades of very good fastness to severe washing and milling.

Example 9

27.5 parts of 5-dodecyl-2-aminotoluene (made in a similar way to that used by Beran, loc. cit.) are stirred for 15 minutes with 250 parts of 36% hydrochloric acid and 300 parts of water, to 50° C. until a homogeneous paste is formed, then enough ice added to cool the whole to 5° C. 69 parts of a 10% aqueous solution of sodium nitrite are then slowly added. The resulting pale greenish-yellow diazo solution is then added with stirring to a solution of 22.3 parts of 2-naphthylamine-8-sulfonic acid in 200 parts of water containing 25 parts of anhydrous sodium carbonate below 10° C. Coupling is rapid and when complete the new dyestuff is filtered off, dried and ground. The product is an orange powder, soluble in hot water to an orange solution which dyes in clear orange shades of very good fastness to severe washing and milling.

Example 10

If, in the above example, instead of 5-dodecyl-2-aminotoluene there are used 26.1 parts of p-dodecylaniline, a dyestuff is obtained which dyes wool in yellower shades of orange and possessing similar fastness to severe washing and milling.

Example 11

To a diazo solution obtained as in Example 1 from 26.1 parts of p-dodecylaniline, a neutral solution of 23.9 parts of 2-amino-8-naphthol-6-sulfonic acid in 250 parts of water containing 5.3 parts of anhydrous sodium carbonate is slowly added in about one hour. Coupling is allowed to complete during 12 hours, the mixture being kept acid to Congo red paper throughout.

The new dyestuff which is out of solution as a brownish-red suspension, is filtered off and dried.

It produces brownish-red shades on wool of good fastness to severe washing and milling and very good fastness to light.

Example 12

26.1 parts of p-dodecylaniline are diazotized as in Example 1 and coupled with 23.9 parts of 2-amino-5-naphthol-7-sulfonic acid in aqueous sodium carbonate. The new dyestuff so-obtained dyes wool from an acid bath in bright orange shades of very good fastness to severe washing and milling.

Example 13

If, in Example 12, 31.9 parts of 1:8-amino-naphthol-2:4-disulfonic acid are used instead of 2-amino-5-naphthol-7-sulfonic acid, a violet-red dyestuff is obtained which dyes wool in reddish-violet shades of very good fastness to severe washing and milling.

Example 14

A diazo solution obtained as in Example 1 from 26.1 parts of p-dodecylaniline is added with stirring to a solution of 36.1 parts of 1-acetamino-8-naphthol-3:6-di-sulfonic acid in 200 parts of water containing 30 parts of sodium carbonate. Coupling is rapid and gives a bright red suspension, which is filtered. When dried and ground the new dyestuff is a dark red powder readily soluble in water with a bluish-red colour. Wool is dyed by it in bright crimson shades of very good fastness to severe washing, milling and light.

Example 15

If in Example 14, 38.9 parts of 1-butyryl-amino-8-naphthol-4:6-disulfonic acid are used instead of 1-acetamino-8-naphthol-3:6-disulfonic acid, a dyestuff is obtained which dyes wool in clear red shades of very good fastness to severe washing, milling and light.

Example 16

If in Example 14, 28.1 parts of 2-acetamino-8-naphthol-6-sulfonic acid are used instead of 1-acetamino-8-naphthol-3:6-disulfonic acid, a dyestuff is obtained which dyes wool in yellowish-red shades of very good fastness to severe washing and milling.

Example 17

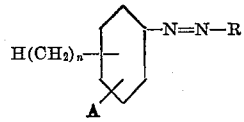
If in Example 14, 42.3 parts of 1-benzoylamino-8-naphthol-3:6-disulfonic acid are used instead of 1-acetamino-8-naphthol-3:6-disulfonic acid, a dyestuff is obtained which dyes wool from an acid bath in bluish-red shades of good fastness to severe washing and milling.

It is apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof and, therefore, it is not intended to be limited except as indicated in the appended claims.

This is a division of U. S. application Serial No. 28,352, filed June 25, 1935, U. S. Patent No. 2,100,-392, patented November 30, 1937.

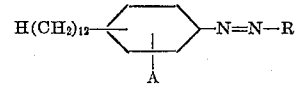
We claim:

1. Compounds of the general formula:



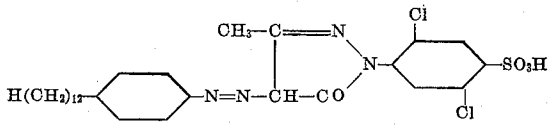
in which R stands for a coupling component devoid of long alkyl chains chosen from the group consisting of naphthol-sulfonic acids, naphthyl-amine-sulfonic acids, amino-naphthol-sulfonic acids, acyl-amino-naphthol-sulfonic acids, and pyrazolone-sulfonic acids, A stands for hydrogen or methyl, and *n* for a number between 10 and 20 inclusive.

2. Compounds of the general formula:

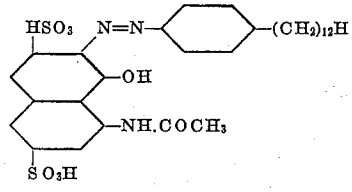


where R stands for a coupling component devoid of long alkyl chains chosen from the group consisting of naphthol-sulfonic acids, naphthyl-amine-sulfonic acids, amino-naphthol-sulfonic acids, acyl-amino-naphthol-sulfonic acids, and pyrazolone-sulfonic acids, A stands for hydrogen or methyl.

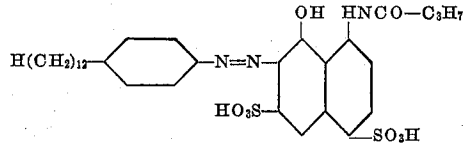
3. The compound of the formula:



4. The compound of the formula:



5. The compound of the formula:



6. Process for the manufacture of monoazo dyestuffs which comprises coupling a diazotized aniline, which carries as a nuclear substituent a primary normal alkyl group with not less than 10 and not more than 20 carbon atoms, with a coupling component devoid of long alkyl chains chosen from the group consisting of naphthol-sulfonic acids, naphthyl-amine-sulfonic acids, amino - naphthol - sulfonic acids, acyl - amino-naphthol-sulfonic acids and pyrazolone-sulfonic acids.

7. Process for the manufacture of monoazo dyestuffs which comprises coupling a diazotized dodecylaniline with a coupling component devoid of long alkyl chains chosen from the group consisting of naphthol-sulfonic acids, naphthyl-amine-sulfonic acids, amino-naphthol-sulfonic acids, acyl-amino-naphthol-sulfonic acids, and pyrazolone-sulfonic acids.

8. Process for the manufacture of a monoazo dyestuff which comprises coupling diazotized p-dodecyl-aniline with 1-(2':5'-dichloro-4'-sulfo) phenyl-3-methyl-5-pyrazolone.

9. Process for the manufacture of a monoazo dyestuff which comprises coupling diazotized p-dodecylaniline with 1-acetyl-amino-8-naphthol-3:6-disulfonic acid.

10. Process for the manufacture of a monoazo dyestuff which comprises coupling diazotized p-dodecyl-aniline with 1-butyryl-amino-8-hydroxy-4:6-disulfonic acid.

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