

# ALIEN PROPERTY CUSTODIAN

## TEXTILE GOODS

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My present invention relates to textile goods, and more particularly to textiles capable of fluorescing when met by ultra-violet light.

It is known to apply to fabrics derivatives of stilbene, especially 4,4'-diaminostilbene-2,2'-disulfonic acid or substitutive derivatives thereof such as benzoylamino- or aminobenzoylamino-derivatives to produce fabrics capable of fluorescing by means of ultra-violet light.

My invention is based on the observation that stilbene compounds colorless or slightly colored and particularly such stilbene derivatives as do not contain free amino groups are especially suitable for the improvement of textiles of any kind as, for instance, fibers, filaments, yarns, or fabrics, knit or felt goods or cloths from animal or vegetable and/or artificial filaments if they contain in their molecule at least one 1,3,5-triazine nucleus. The employment of amino stilbene compounds of this kind has already been proposed in the production of colorless or only slightly colored protective envelopes for goods capable of being affected by light and air.

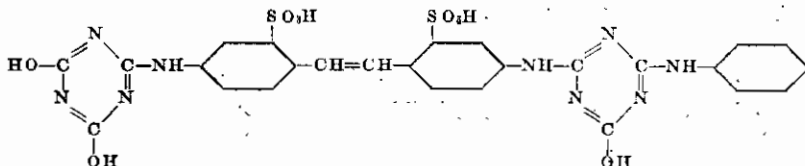
On a treatment with a solution of 0.001-0.2% strength of stilbene derivatives as defined above even strongly brownish-yellow unbleached textile goods obtain with ordinary daylight the appearance as if they be colored pure-white so that they are closely similar to goods well bleached. Moreover, the strength of the products treated with these stilbene derivatives is not reduced in contrast with the products bleached. The textiles

materials and have so strong an affinity to these materials that they cannot be removed by washing; they are also very resistant to alkaline washing liquids. The aminostilbene compounds are also absorbed by artificial textiles as, for instance, cellulose acetate, polymerized vinylchloride or superpolyamides. Finally, the textiles thus treated are more resistant to turning yellow produced by light and therefore keep the white color longer than the aminostilbene derivatives not containing 1,3,5-triazine rings.

The amino stilbene derivatives of the present invention are preferably applied to the textile materials in the form of water-soluble salts as, for instance, sodium, potassium, or triethanol-amino salts in an aqueous neutral or alkaline solution. It is sometimes of advantage to add dispersing or wetting agents to the solution in order to obtain a homogeneous liquid and effect a fast penetrating into the inner layers of the textile material. The stilbene derivatives may, if desired, be added to the solutions or melts of artificial spinning materials. Water-insoluble aminostilbene derivatives of the invention may successfully be employed in the form of an organic solution.

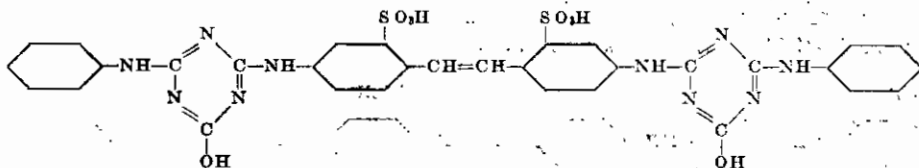
As examples of such aminostilbene derivatives may be mentioned the following compounds:

(1) 4-[2,4-dihydroxy-1,3,5-triazyl-(6)]-amino-4'-[2-hydroxy-4-phenyl-amino-1,3,5-triazyl-(6)]amino-stilbene disulfonic acid-(2,2') of the following formula:



of this invention are also protected from the noxious influence of the ultra-violet rays contained in the daylight. Furthermore, the

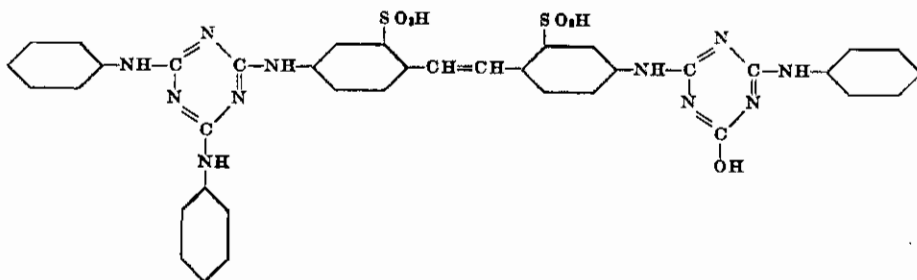
(2) 4,4'-(bis-[2-hydroxy-4-phenylamino-1,3,5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



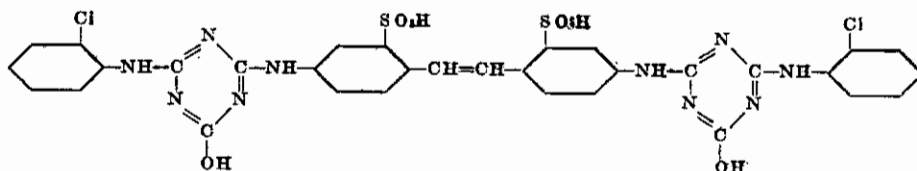
aminostilbene derivatives having 1,3,5-triazine nuclei are especially strongly absorbed by textile

(3) 4-[2,4-bis-phenylamino-1,3,5-triazyl-(6)]amino-4'-[2-hydroxy-4-phenylamino-

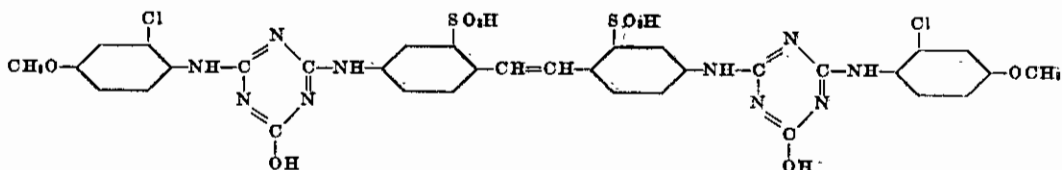
1.3.5-triazyl-(6) diamino-stilbene-disulfonic acid-(2.2') of the following formula:



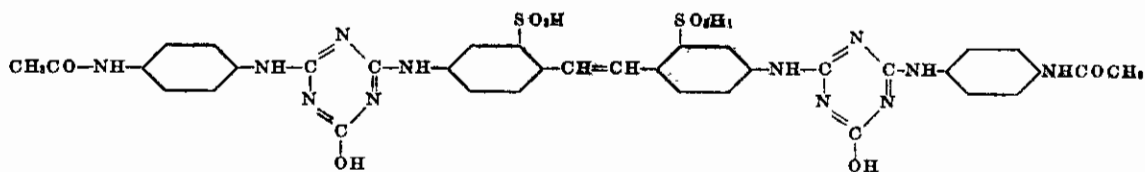
(4) 4.4' - (bis - [2 - hydroxy - 4-o-chlorophenyl-amino - 1.3.5 - triazol - (6) ]) diamino - stilbene - disulfonic acid-(2.2').



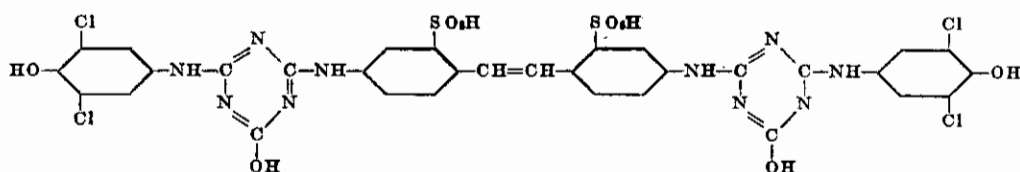
(5) 4.4' - (bis-[2-hydroxy-4-p-methoxy-phenyl-amino-1.3.5-triazyl-(6) ]) diamino-stilbene-disulfonic acid-(2.2').



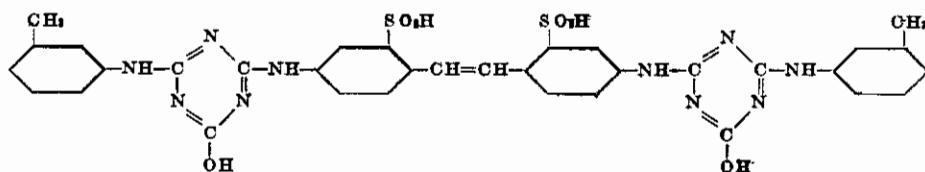
(6) 4.4' - (dis-[2 - hydroxy - 4 - p - acetylamino - phenylamino - 1.3.5 - triazol - (6) ]) diamino-stilbene-disulfonic acid-(2.2').



(7) 4.4' - (bis-[2-hydroxy-4-p-hydroxy-m,m-dichlorophenyl - amino - 1.3.5-triazyl - (6) ]) diamino-stilbene-disulfonic acid-(2.2').

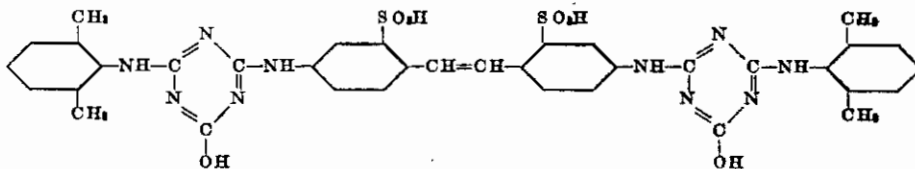


(8) 4.4' - bis - [2-hydroxy-4-m-methylphenyl-amino-1.3.5-triazyl-(6) ] diamino-stilbene - disulfonic acid-(2.2') of the following formula:



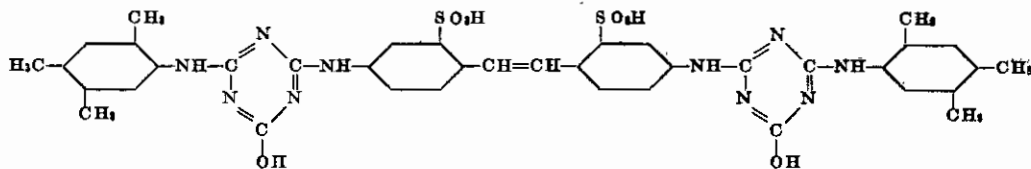
and the isomeric toluidine derivatives thereof.

(9) 4,4'-(bis-[2-hydroxy-4-o,o-dimethyl-phenylamino-1.3.5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:

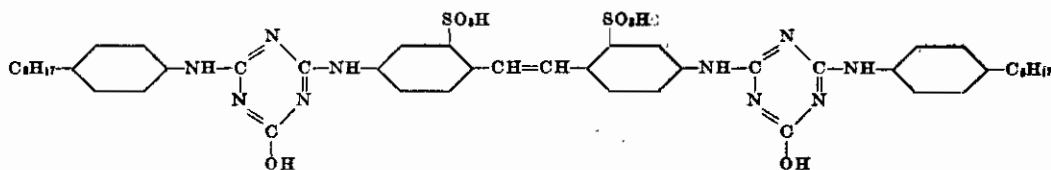


and the isomeric xylylene derivatives thereof.

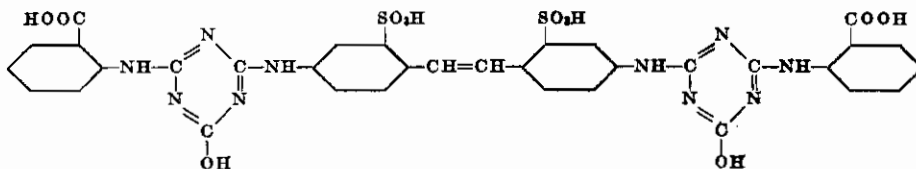
(10) 4,4'-(bis-[2-hydroxy-4-(1',3'',4''-trimethyl-phenyl-amino-(6''))-1.3.5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



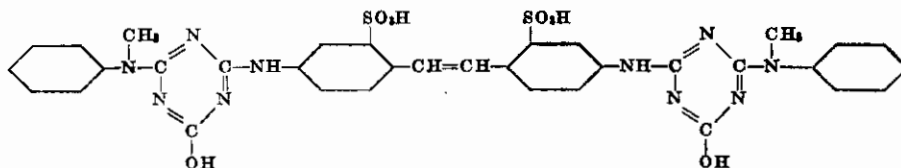
(11) 4,4'-(bis-[2-hydroxy-4-p-octyl-phenyl-amino-1.3.5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



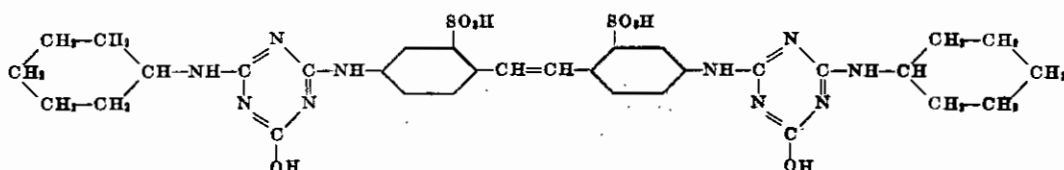
(12) 4,4'-(bis-[2-hydroxy-4-o-carboxy-phenyl-amino-1.3.5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



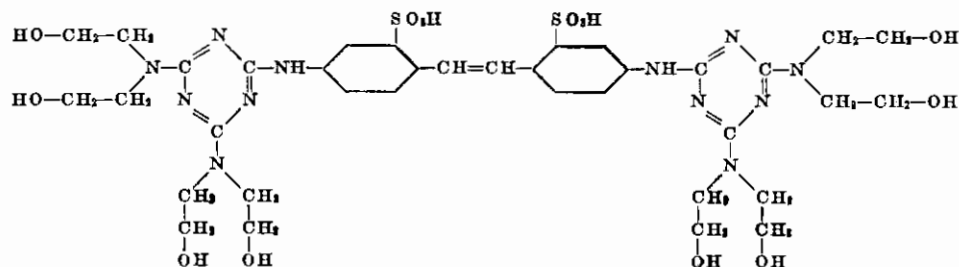
(13) 4,4'-(bis-[2-hydroxy-4-N-phenyl-N-methyl-amino-1.3.5-triazyl(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



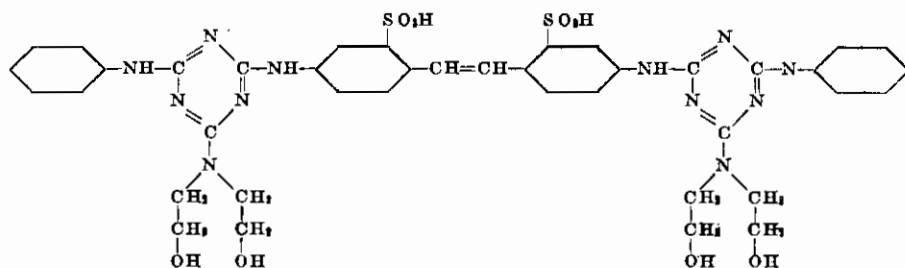
(14) 4,4'-(bis-[2-hydroxy-4-cyclohexyl-amino-1.3.5-triazyl-(6)])diamino-stilbene-disulfonic acid-(2,2') of the following formula:



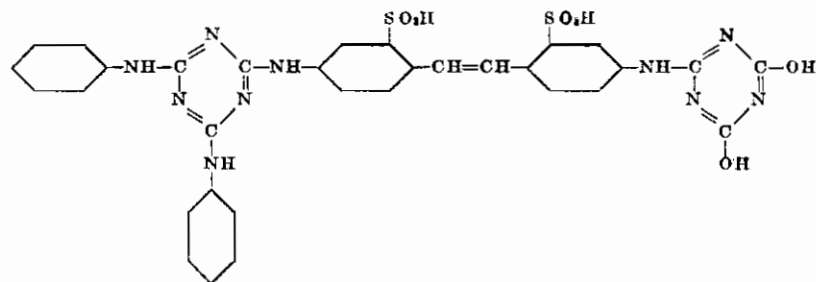
(15) 4,4'-(bis-[2,4-bis-diethanol-amino-1,3,5-triazyl-(6)]) diamino-stilbene-disulfonic acid-(2,2') of the following formula:



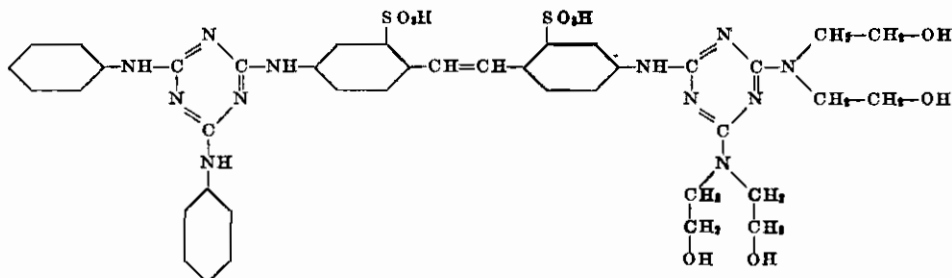
(16) 4,4'-(bis-[2-phenylamino-4-diethanol-amino-1,3,5-triazyl-(6)]) diamino-stilbene disulfonic acid-(2,2') of the following formula:



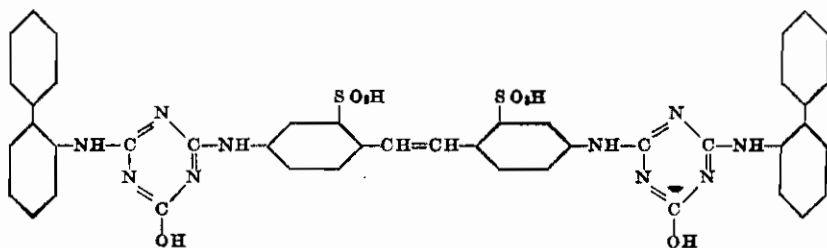
(17) 4-[2,4-bis-phenylamino-1,3,5-triazyl-(6)] amino-4'-[2,4-dihydroxy-1,3,5-triazyl-(6)] amino-stilbene disulfonic acid-(2,2') of the following formula:



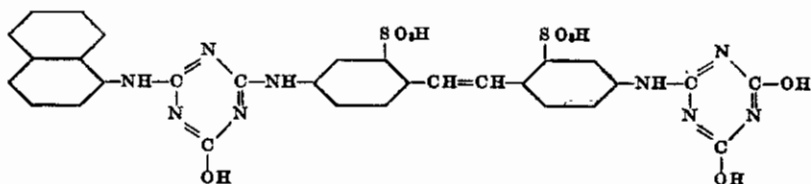
(18) 4-[2,4-bis-phenylamino-1,3,5-triazyl-(6)] amino-4'-[2,4-bis-ethanolamino-1,3,5-triazyl-(6)] amino-stilbene-disulfonic acid-(2,2') of the following formula:



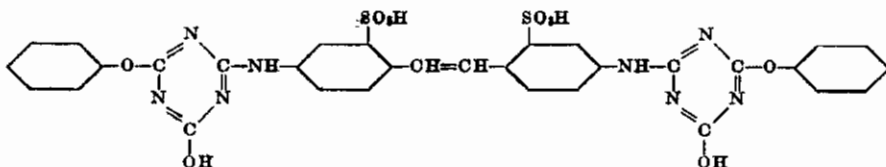
(19) 4,4'-(bis-[2-hydroxy-4-o-phenyl-phenyl-amino-1,3,5-triazyl-(6)]) diamino-stilbene-disulfonic acid-(2,2') of the following formula:



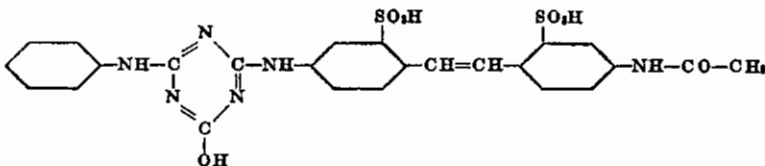
(20) 4-[2-hydroxy-4- $\alpha$ -naphthylamino-1,3,5-triazyl-(6)] amino-4'-[2,4-dihydroxy-1,3,5-triazyl-(6)] amino-stilbene disulfonic acid-(2,2') of the following formula:



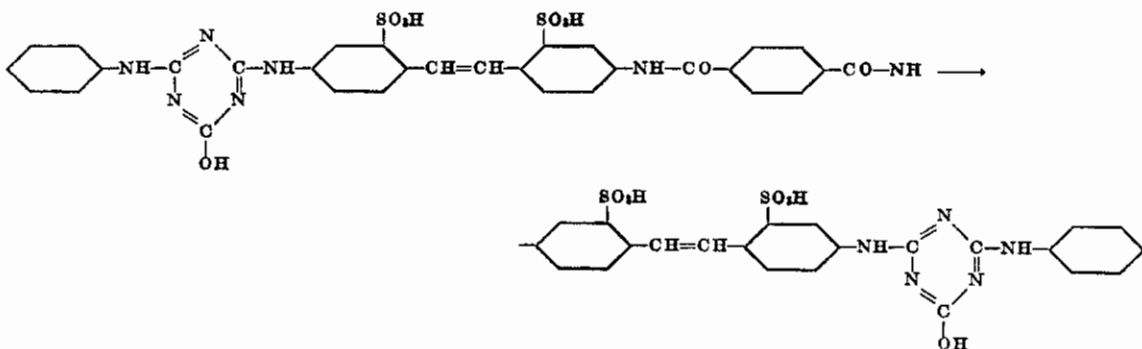
(21) 4,4'-(bis-[2-hydroxy-4-phenoxy-1,3,5-triazyl-(6)]) diamino-stilbene-disulfonic acid-(2,2') of the following formula:



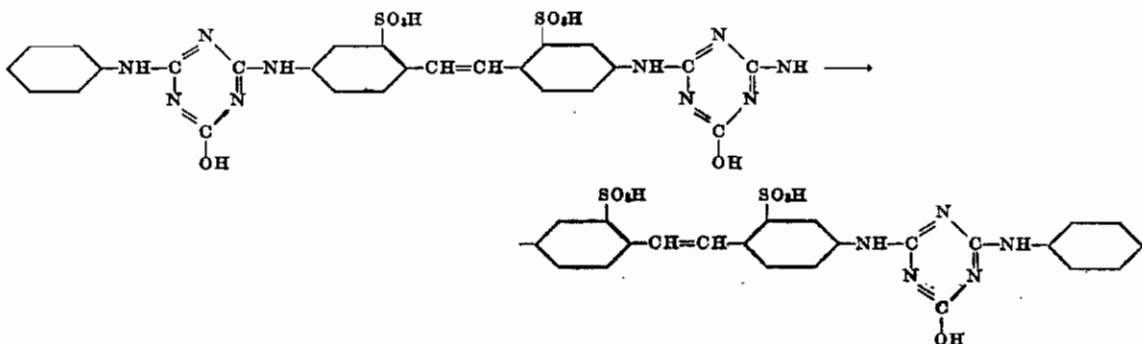
(22) 4-(2-hydroxy-4-phenylamino-1,3,5-triazyl-(6))-4'-acetyl-amino-stilbene-disulfonic acid-(2,2') of the following formula:



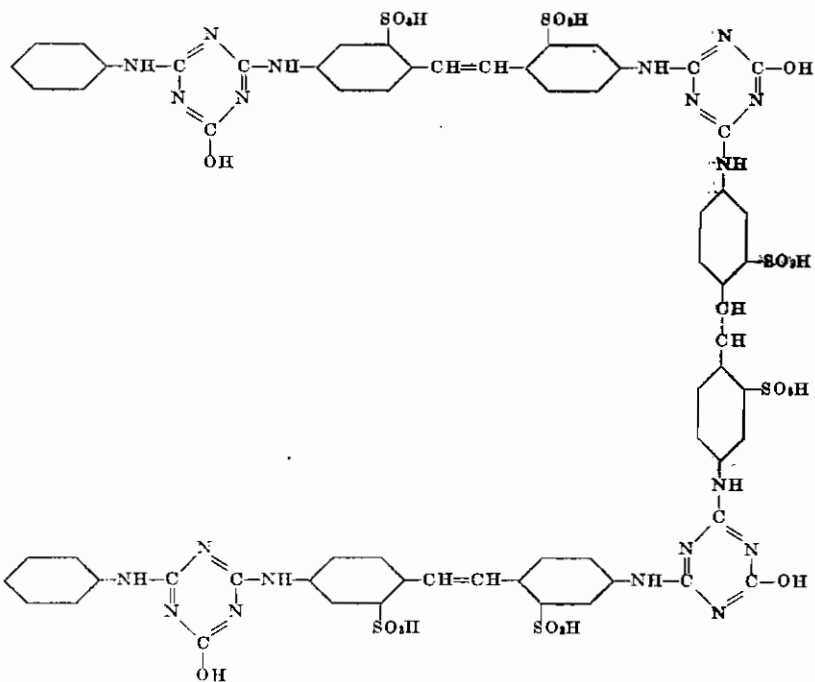
(23) Terephthaloyl-bis-(4-amino-[4'-(2-hydroxy-4-phenylamino-1,3,5-triazyl-(6)) amino] stilbene disulfonic acid (2,2')) of the following formula:



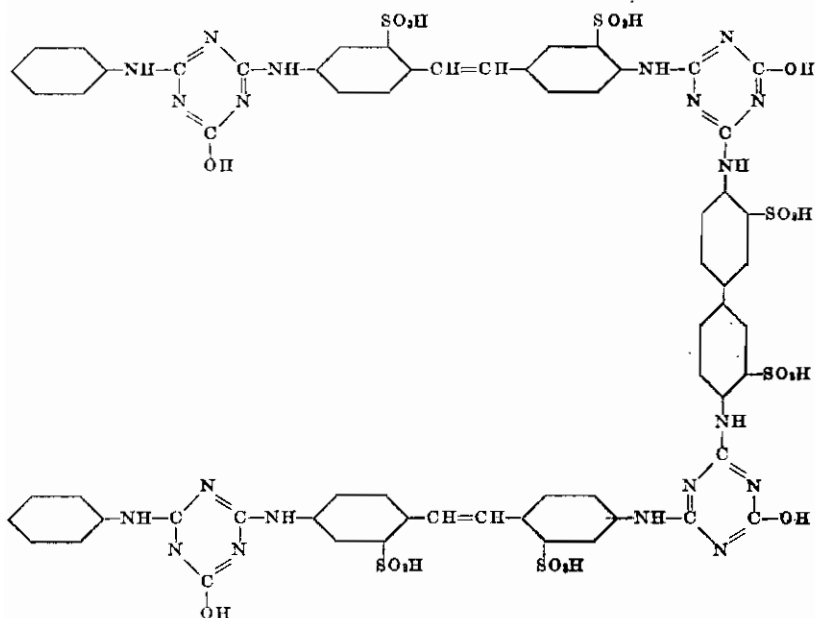
(24) The product prepared by reacting 2 mols of 4-amino-4'-[2-hydroxy-4-phenylamino-1,3,5-triazyl-(6)] amino-stilbene-disulfonic acid-(2,2') with 1 mol of cyanurichloride of the following constitution:



(25) The product prepared by reacting 1 mol of bis-[2,4-dichloro-1,3,5-triazyl-(6)] diaminostilbene-disulfonic acid-(2,2') with 2 mols of 4-amino-4'-[2-hydroxy-4-phenylamino-1,3,5-triazyl-(6)] amino-stilbene-disulfonic acid-(2,2') of the following constitution:



(26) The product prepared by reacting 1 mol of 4,4'-bis-[2,4-dichloro-1,3,5-triazyl-(6)] diamino-diphenyl-disulfonic acid (3,3') with two mols of 4-amino-4'-[2-hydroxy-4-phenylamino-1,3,5-triazyl-(6)] amino-stilbene disulfonic acid-(2,2') of the following constitution:

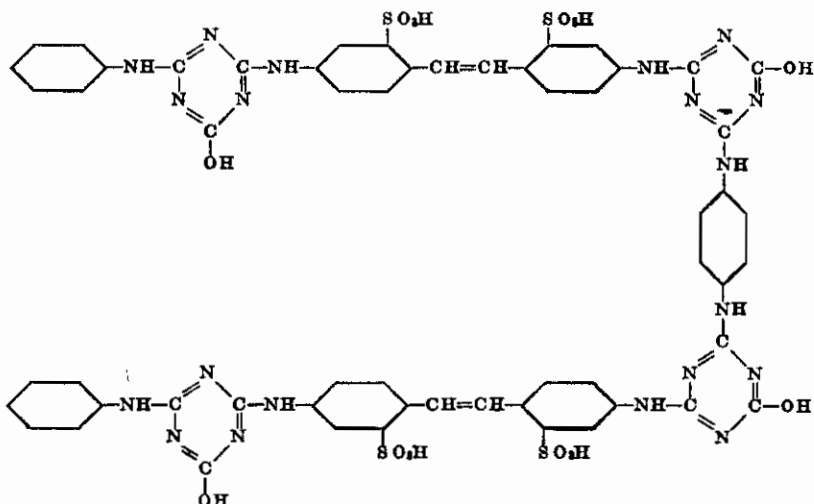


(27) The product prepared by reacting 2 mols of 4-[2-hydroxy-4-phenyl-amino-1,3,5-triazyl-(6)] amino-4'-[2,4-dichloro-1,3,5-triazyl-(6)]

amino-stilbene-disulfonic acid-(2,2') with 1 mol p-phenylenediamine of the following constitution:

chlorine atoms present in the 1.3.5-triazine nuclei.

The unsymmetrical compounds are correspond-



These examples are, of course, not to be considered as limitations of the invention. Instead of the 4,4'-diamino-stilbene-2,2'-disulfonic acids, for instance, the 3,3'-disulfonic acids or 2,2'-diamino-stilbene-4,4'-disulfonic acids or 4,4'-diamino-stilbene-3,3'-dicarboxylic acids may also be employed.

Substitutive derivatives of such amino-stilbene-sulfonic acids or amino-stilbene-carboxylic acids are also useful as contain only one sulfo or one carboxyl group. The corresponding aminostilbene derivatives having no carboxyl- or sulfonic acid radical are likewise suited if certain solvents as, for instance, organic solvents are used.

Instead of the aromatic, hydroaromatic or aliphatic substituents mentioned above other substituents of this kind may also be employed in the triazine nucleus such as hydrocarbon remainders or the radicals  $C_6H_5.S-$ ,  $C_6H_5.SO_2-$ . Moreover, heterocyclic substituents as, for instance, aminobenzimidazoles, aminobenzthiazoles, aminoquinolines, and the like and finally such substituents as are fluorescent themselves are useful.

The aminostilbene compounds symmetrical in structure are prepared by causing 1.3.5-triazylchloride dissolved in acetone to react with an aqueous solution of the sodium salt of diamino-stilbene-disulfonic acid at a low temperature (about 0-5° C). After the reaction in which the solution has been maintained essentially neutral is complete the amine or phenol is caused to flow slowly into the reaction solution, if necessary, at a somewhat higher temperature (10-30° C). Water-insoluble amines are conveniently dissolved in an organic solvent soluble in water or are used in the form of their water-soluble salts. Finally soda can be added to the mixture and the whole is boiled in order to saponify the

ingly prepared from nitroaminostilbene-disulfonic acid.

The following Examples illustrate the production of the new textile goods.

#### Example I

100 g of fabric from viscous staple fiber or artificial silk or 100 g of viscous staple fiber in the form of flakes are treated with 3000 g of water containing 1.5 g of the sodium salt of 4-[2-hydroxy-4- $\beta$ -naphthyl-amino-1.3.5-triazyl-(6)]-4'-benzoylamino-stilbene-disulfonic acid-(2,2') at 80° C for 45 minutes and subsequently dried.

#### Example II

100 g of a cotton fabric are treated in the same manner as described in Example I.

#### Example III

100 g of linen are treated in the same manner as described in Example I.

#### Example IV

A rope of viscous silk disulfurized and unbleached of 100 g is treated in a solution consisting of 3 liters of water and 0.6 g of the sodium salt of 4,4'-(bis-[2-hydroxy-4-phenyl-amino-1.3.5-triazyl-(6)])-diamino-stilbene-disulfonic acid (2,2') at 80° C for 45 minutes and then dried. To the bath 10 to 20 g of sodium sulfate free from water may, if necessary, be added in portions towards the end of the treatment.

#### Example V

1 kg of the molten polyamide prepared from amino-caproic acid mixed with 0.2 g of the sodium salt of 4,4'-(bis-[2-hydroxy-4-phenyl-amino-1.3.5-triazyl-(6)])-diamino-stilbene-disulfonic acid-(2,2') is worked up into filaments.

BRUNO WENDT.