## United States Patent Office.

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## TRISAZO DYE.

SPECIFICATION forming part of Letters Patent No. 572,723, dated December 8, 1896.

Application filed April 10, 1896. Serial No. 586,931. (Specimens.) Patented in England July 10, 1893, No. 13,402, and in Germany October 23, 1894, No. 86,009.

To all whom it may concern:

Be it known that I, Christian Rudolph, doctor of philosophy, chemist, residing at Offenbach - on - the - Main, Germany, have invented a new and useful Improvement in the Manufacture of New Brown-Red to Corinth Trisazo Dyes, (for which patents have been obtained in Germany, No. 86,009, dated October 23, 1894, and in England, No. 13,402, dated July 10, 1893,) of which the following is a specification.

 $SO_3H$   $NH_2$ 

Amidophenolsulfo-acid III.

My invention consists in the discovery that the so-called "paradiamins"—as, for instance, benzidin, toluidin, dianisidin, &c. — under suitable circumstances yield intermediate 15 compounds with those metaämidoöxysulfoacids of the benzene series which contain the OH group and NH<sub>2</sub> group in the so-called "meta" position. Such are, for instance, the amidophenolsulfo-acid III and the amido-20 cresolsulfo-acid III, as will be seen by comparing the formulæ of these acids.

 $ho_{H}$   $ho_{SO_3H}$   $ho_{NH_2}$   $ho_{Amidocresolsulfo-acid~III}$ 

Now if the intermediate products thus resulting from the action of one molecule of a paradiamin upon one molecule of a metaämido-oxysulfo-acid of the benzene series are combined with resorcin or metaphenylendiamin tetrazo dyestuffs result which are susceptible of acting upon one molecule diazonaphthionic acid, thus yielding new trisazo dyestuffs.

The above-mentioned acids, amidophenol-sulfo-acid III and amidocresolsulfo-acid III,

are obtained by melting anilindisulfo-acid or toluidindisulfo-acid, respectively, with alkalies.

The new trisazo dyestuffs dye unmordanted cotton from an alkaline bath brown-red to corinth.

As an example in which manner my invention may be carried out I shall hereinafter 40 describe the production of the dyestuff:

Azoamidophenolsulfo-acid III

Azometaphenylendiamin + diazonaphthionic-acid.

By "parts" are meant parts by weight. Convert nine and one-fifth (9.2) parts of benzidin into tetrazo-diphenyl chlorid in the usual way and pour it into an aqueous solution, which is cooled down to 0° centigrade, of ten and three-fifths (10.6) parts of the sodium salt of amidophenolsulfo-acid and the equal quantity of soda. After the formation 50 of the intermediate product add to the mass five and two-fifths (5.4) parts of phenylendiamin, and, finally, after the intermediate dyestuff has been formed, add eleven and seven-tenths (11.7) parts of diazonaphthionic 55 acid in aqueous emulsion. Allow to stand for twenty-four (24) hours. Then heat the mass, add common salt in order to precipitate the dyestuff, filter, press, and dry. The

coloring-matter thus obtained forms a black powder, which dissolves in water to a brown 60 and in concentrated sulfuric acid to a violet to a blue solution.

What I claim as my invention is—

1. The within-described process of making new trisazo dyestuffs consisting in first form- 65 ing intermediate products by combining the paradiamins, as for instance benzidin, with a metaämidoöxysulfo-acid of the benzene series which contains the OH group and the NH<sub>2</sub> group in the so-called "meta" position, then 70 combining these intermediate products with metaphenylendiamin or resorcin and finally causing diazonaphthionic acid to act upon the thus resulting intermediate dyestuffs.

2. As a new article of manufacture, the new 75

trisazo dyestuffs derived from the intermediate products formed by the combination of paradiamins and the metaämidoöxysulfoacids of the benzene series which contain the OH group and the NH<sub>2</sub> group in the so-called "meta" position, the said dyestuffs forming a black powder which is soluble in water giving brown to brown-red solutions and soluble in concentrated sulfuric acid giving violet to blue solutions, and dyeing unmordanted cot-

ton from an alkaline bath brown-red to corinth, substantially as herein described.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

## CHRISTIAN RUDOLPH.

Witnesses:
DEAN MASON,
JEAN GRUND.