## UNITED STATES PATENT OFFICE.

GUSTAV SCHULTZ, OF BERLIN, GERMANY, ASSIGNOR TO THE ACTIEN-GESELLSCHAFT FÜR ANILIN FABRIKATION, OF SAME PLACE.

## BLUE AZO DYE.

SPECIFICATION forming part of Letters Patent No. 462,824, dated November 10, 1891.

Application filed June 4, 1890. Serial No. 354,260. (Specimens.) Patented in Germany April 20, 1890, No. 57,444; in England May 5, 1890, No. 6,932, and in France May 12, 1890, No. 205,615.

To all whom it may concern:

Be it known that I, GUSTAV SCHULTZ, chemist, a subject of the King of Prussia, residing at Berlin, Prussia, German Empire, 5 have invented certain new and useful Improvements in the Production of New Blue Dye-Stuffs, (for which Letters Patent have been granted in Germany under date of April 20, 1890, No. 57,444; in France, dated May 12, 10 1890, No. 205,615, and in Great Britain, dated May 5, 1890, No. 6,932;) and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it ap-15 pertains to make and use the same.

The invention consists in a process of obtaining blue dye-stuffs from tetrazo-ditolyl and naphthylamine, as will now be fully de-

scribed.

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By combining one molecule of tetrazo-ditolyl with one molecule of naphthylamine a product having the following chemical formula is obtained:

$$\frac{C_{7}H_{6}-N=N-C_{10}H_{6}NH_{2}HCl}{C_{7}H_{6}-N=NCl}$$

This diazo compound may be converted by heating with nitrous acid into a tetrazo compound, which latter when combined with 3° phenols, amines, or their sulpho or carbo acids in a well-known manner produces dyestuffs adapted for dyeing and printing. These dye-stuffs possess the valuable property of directly dyeing cotton—that is to say, they 35 have the property of dyeing cotton without the use of a mordant—and possess, besides this, the property of being unaffected by the action of light and air.

The following examples will clearly show 40 the manner of carrying out my invention:

Example 1.—10.9 kilos of toluidine are converted in a well-known manner into a tetrazo compound, the solution of which is combined with a solution of 7.5 kilos of a salt of alpha-45 naphthylamine. The compound is allowed to stand for some time and is afterward acidulated with hydrochloric acid and again diazotized by treatment with nitrite of soda. The tetrazo compound thus obtained is now

(40) kilos of a soda salt of alpha-naphthol disulpho-acid D, such as is described in Letters Patent of the United States No. 333,034. After standing for some hours the mixture is heated to boiling and the resulting dye-stuff 55 is salted out, filtered, and dried. The product is a coloring matter which dyes unmordanted cotton a deep-blue violet.

Example 2.—If the naphthol disulpho-acid D is replaced by alpha-naphthol disulpho- 60 acid E, which is described in Letters Patent of the United States No. 405,938, a dye-stuff is obtained that is substantially like that described in reference to example 1 and has substantially the same properties.

Example 3.—Instead of toluidine, dianisidine may be employed in the process, as described in reference to example 1. In this case 17.1 kilos of sulphate of dianisidine are used; otherwise the mode of proceeding re- 70 mains the same as described in reference to said example 1. The dye-stuff obtained dyes unmordanted cotton a fine blue-green shade.

Example 4.—Instead of toluidine, as described in example 2, dianisidine may also be 75 employed, the results being the same.

Example 5.—If instead of toluidine, as described in example 1, diphenititine is used, a dye-stuff is obtained that differs but little from the dye-stuff obtained according to ex- 80 ample 3.

Example 6.—A like result is obtained if diphenylidine is used-instead of toluidine, as

described in example 2.

The above-described hexa-azo dyes are gen- 85 erally soluble in water and sulphuric acid and insoluble in alcohol. They dissolve blue in sulphuric acid and they are not readily affected by air and light.

By "dianisidine" and "diphenititine" I 90 mean either a methyl or ethyl ether of diamidodi-phenol. Hence by the "dianisidine" referred to is meant the dimethyl ether of diamido diphenol

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m 9.5} \\ {
m NH}_2 & {
m C}_6 {
m H}_3 \left\{ egin{array}{cc} {
m NH}_2 \\ {
m OCH}_3 & {
m OCH}_3 \end{array} 
ight\} \end{array}$$

50 combined with an alkaline solution of forty and by the "diphenititine" referred to is 100

meant the diethyl ether of diamido diphenol

 $egin{array}{c} {
m C_6H_3} \left\{ egin{array}{c} {
m OC_2H_5} \\ {
m NH_2} \\ {
m C_6H_3} \left\{ egin{array}{c} {
m NH_2} \\ {
m OC_2H_5} \end{array} 
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described by Knecht in his work on the dyestuffs derived from coal-tar.

Having now described my invention, what

10 I claim is—

1. The process of obtaining blue direct-dyeing dye-stuffs, which consists in combining one molecule of a tetrazo salt substantially such as described with one molecule of alpha - naphthylamine, again diazotizing the compound, and combining with the resultant

product two molecules of an alpha-napththol

disulpho-acid, as set forth.

2. The herein-described blue hexa-azo dyes derived from one molecule of toluidine or anisidine, one molecule of alpha-naphthylamine, and two molecules of an alpha-naphthol disulpho-acid, said dyes being insoluble in alcohol, readily soluble in water and sulphuric acid, and are not readily affected by air and light.

In testimony whereof I affix my signature in

presence of two witnesses.

GUSTAV SCHULTZ.

Witnesses:

ADOLF DEMELIUS.