## United States Patent Office.

OTTO BORGMANN, OF BERLIN, GERMANY, ASSIGNOR TO THE ACTIEN-GES-ELLSCHAFT FÜR ANILIN FABRIKATION, OF SAME PLACE.

## ORANGE DYE.

SPECIFICATION forming part of Letters Patent No. 454,840, dated June 30, 1891.

Application filed December 11, 1890. Serial No. 374,383. (No specimens.) Patented in Germany November 1, 1889, No. 52,328; in England November 11, 1889, No. 17,957, and in France December 16, 1889, No. 160,722.

To all whom it may concern:

Be it known that I, Otto Borgmann, doctor of philosophy, a subject of the King of Prussia, residing at Berlin, Prussia, German Empire, have invented certain new and useful Improvements in the Production of Orange-Yellow Azo Colors, (for which I have obtained Letters Patent in the following countries: Germany, No. 52,328, dated November 1, 1889; Ito France, No. 160,722, dated December 16, 1889, and Great Britain, No. 17,957, dated November 11, 1889;) 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 appertains to make and use the same.

The invention relates to the manufacture of orange-yellow azo dye-stuffs; and it consists in the mode of obtaining the same and in a novel orange-yellow dye-stuff, as will now

be fully described.

The British patents granted to Peter Jensen under date of the 12th of December, 1885, No. 15,296, and to Carl A. Martius under date 25 of the 15th of February, 1886, No. 2,213, describe certain processes for obtaining orangeyellow dye-stuffs by combining tetrazodiphenyl or tetrazoditolyl with beta-naphthylamine disulpho-acid Rand phenol. These dye-stuffs, 30 however, are readily affected by alkalies, under the action of which the color or depth of color undergoes material changes, and this leads to great inconveniences in the use of said dye-stuffs in dyeing textiles. I have discovered that these dye-stuffs can be rendered proof or substantially proof against the action of the alkalies by the substitution of an alkyl group for one H of the free hydroxyl of the phenol. This I accomplish in the following 40 manner, to wit: I dissolve, say, ten parts of the dye-stuff above referred to in about forty to sixty parts of hot water, and add thereto the theoretical quantity of caustic soda to convert the dye into the neutral salt. Besides this I add a small quantity of soda (about ten to fifteen per cent. of the weight of the dye) and mix the solution with an equal volume of alcohol. To this I add about 2.5 to 3.5 parts of ethyl bromide, or in lieu thereof from 50 3.2 to four parts of methyl iodide. An excess l

of the halogen alkyl is preferred, in order to insure the complete alkalization of the mixture, which is then heated for about five or six hours in a reflux condenser on a water bath, after which the alcohol is distilled off 55 and the coloring-matter precipitated with common salt, filtered off, pressed, and dried. With Glauber's salt in a soap bath it dyes cotton without the use of a mordant a brilliant orange-yellow shade, which is substantially 6c proof against the action of the alkalies. If, instead of the benzidine dye its tolidine homologue is alkalized in the manner described, a similar orange-yellow dye-stuff is obtained, the shade of which is, however, a little more 65 reddish than that of the benzidine dye.

The new dye-stuffs have the following chemical constitution:

$$X$$
  $NH_{2}$   $7$   $C_{6}H_{3}-N=N-C_{10}H_{4}(SO_{3}Na)2$   $C_{6}H_{3}-N=N-C_{6}H_{4}OC_{2}H_{5}$   $7$ 

in which X represents either hydrogen or methyl. These dye-stuffs consist, essentially, of an amorphous orange-red powder that dissolves readily in hot water with an orange-80 red color and but sparingly in hot alcohol. An addition of acetic acid to the aqueous solution causes it to turn brown, while an addition of concentrated mineral acids produces a brown precipitate. Caustic-soda lye added 85 to the aqueous solution will not affect its color; but the dye-stuffs are precipitated by an excess of soda-lye in the form of an orange-red gelatinous precipitate. The dye-stuffs themselves enter into solution with concentrated 90 sulphuric acid with an indigo-blue color, and when this solution is diluted with water the free acid of the dye-stuff separates in the form of brown flakes, while reducing agents destroy the dye-stuffs forming tolidine or benzidine, 95 diamidonaphthalene disulpho-acid, and paraamidophenol ethyl ether.

Having described my invention, what I claim is—

1. The process of obtaining orange-yellow 100

dye-stuffs capable of withstanding the action of alkalies, which consists in alkalizing the orange-yellow dye-stuffs obtained by the combination of a tetrazodiphenyl or tetrazoditolyl with beta-naphthylamine disulpho-acid R and phenol by treating the same with a halogen alkyl, as set forth.

2. The herein-described orange-yellow dyestuffs, readily soluble in hot water and spar-

ingly soluble in alcohol, said dye-stuffs being turned blue by strong sulphuric acid and giving a brown precipitate in dilute mineral acid.

In testimony whereof I affix my signature in presence of two witnesses.

OTTO BORGMANN.

Witnesses:

GEORGE LOUBIER, ADOLF DEMELIUS.