UNITED STATES PATENT OFFICE.

ALBERT HERRMANN, OF HÖCHST-ON-THE-MAIN, GERMANY.

BLUE COLORING-MATTER.

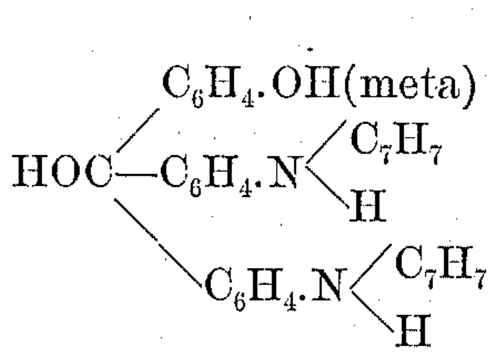
SPECIFICATION forming part of Letters Patent No. 567,567, dated September 8, 1896.

Application filed January 11, 1895. Serial No. 534,586. (Specimens.) Patented in Germany June 6, 1893, No. 74,014; in France July 31, 1893, No. 192,807, and in England July 31, 1893, No. 14,671.

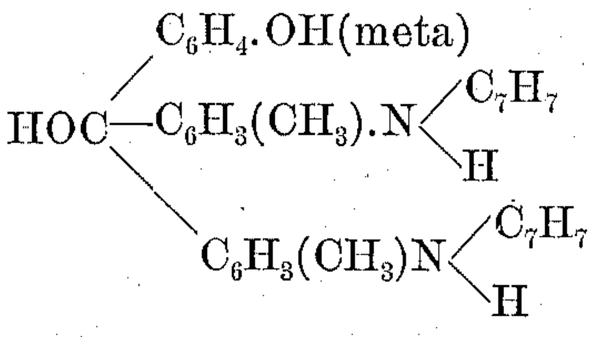
To all whom it may concern:

Be it known that I, ALBERT HERRMANN, a citizen of the Empire of Germany, and a resident of Höchst-on-the-Main, in the Empire of Germany, have invented certain new and useful Improvements in the Production of Green-Blue Coloring-Matter, (for which Letters Patent were granted to me in Germany, No. 74,014, dated June 6, 1893; in France, by Certificate of Addition to Letters Patent No. 192,807, dated July 31, 1893, and in Great Britain, No. 14,671, dated July 31, 1893,) of which the following is a specification.

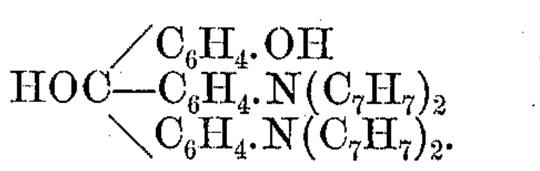
I have discovered a method for manufacturing a fast-blue acid coloring-matter. This coloring-matter consists of the sulfonic acids of metaoxydibenzyldiamidotriphenylcarbinol,



or metaoxydibenzyldiamidoditolylphenylcarbinol,



or metaoxytetrabenzyldiamidotriphenylcarbinol,



The manufacture of this coloring-matter is carried out in a manner analogous to the process described in Reissued Letters Patent granted to me on May 20, 1890, No. 11,077, where in place of the bases mentioned those indicated by the above formulæ are used. Thus in accordance with said process the symmetrical metaamidodibenzyldiamidotriphenylmethane or symmetrical metaamidodibenzyldiamidoditolylphenylmethane or metaamidotetrabenzyldiamidotriphenylmethane are converted into the corresponding metaoxyleuco bases by means of nitrous acid, or the metaoxyleuco bases are obtained by condensation of monobenzylanilin, monobenzylorthotoluidin, or dibenzylanilin with metaoxybenzaldehyde. The resulting metaoxyleuco bases are then sulfonated by concentrated or fuming sulfuric acid and the leuco-sulfonic acid oxidized by peroxid of lead or similar agents.

Instead of the bases the sulfonic acid of monobenzylanilin or monobenzylorthotoluidin or dibenzylanilin may be condensed with metaoxybenzaldehyde and the resulting leuco-sulfonic acids then further sulfonated to the leuco-sulfonic acids of the blue coloring-matters, and these oxidized to the coloring-matters.

Example: 39.4 parts, by weight, of monobenzylorthotoluidin are dissolved in two hundred parts, by weight, of alcohol. To this solution twenty-two parts, by weight, of thirty-one per cent. hydrochloric acid and 12.2 parts, by weight, of metaoxybenzaldehyde are added. This solution is boiled for twenty-four hours in an inverted condenser and then the alcohol is distilled off. The residual leuco base is boiled with much water with addition of dilute sulfuric acid, then treated with boiling water until it reacts neutral, and finally dried on a water-bath. The sulfonation is effected by treating this leuco base with sulfuric acid, monohydrated sulfuric acid, or fuming sulfuric acid. It has proved most advantageous to dissolve the leuco base in five parts, by weight, of monohydrated sulfuric acid. The product of the reaction is poured into water and the precipitated leuco sulfonic acid filtered off and converted into the calcium or sodium salt. This is then treated with the calculated quantity of peroxid of lead and of a mineral acid or acetic acid. The lead is precipitated from the resulting blue solution with Glauber salt. In the filtrate the coloring-matter is precipitated with salt. The solution of the coloring-matter may also be evaporated to dryness.

The new coloring-matter is a copper-red powder with metallic luster. It dissolves in

water with a blue color, is very slightly soluble in alcohol, almost insoluble in benzene and ether. The aqueous solution becomes green on addition of a mineral acid. Ammonia and soda solution scarcely alter the blue color of the aqueous solution. By heating with caustic soda the coloring-matter is destroyed and the solution becomes red.

The coloring-matter dyes wool and silk in an acid bath in even and fast blue tints.

What I claim as new, and desire to secure by Letters Patent, is—

1. The process of producing fast-blue coloring-matter, which consists in condensing monobenzylanilin or its homologues with metaoxybenzaldehyde, sulfonating the metaoxyleuco bases obtained thereby, and then oxidizing the resulting leuco-sulfonic acid, substantially as set forth.

2. The process of producing fast-blue coloring-matter, which consists in condensing the sulfonic acids of monobenzylanilin or its homologues with metaoxybenzaldehyde, then again sulfonating the resulting leuco-sulfonic acids whereby leuco-sulfonic acids of the blue coloring-matter are produced, and oxidizing the latter to the coloring-matter with a suitable reagent such as lead peroxid, substantially as set forth.

3. As a new product, a fast-blue coloring-matter being a copper-red powder with metallic luster, soluble in water, scarcely soluble in alcohol, insoluble in ether and benzene, dyeing wool and silk in an acid bath blue tints, the aqueous solution turning green with mineral acid and red on heating with caustic soda, substantially as set forth.

In testimony that I claim the foregoing as my invention I have signed my name in presence of two subscribing witnesses.

ALBERT HERRMANN.

Witnesses:

HEINRICH HAHN,

BERNHARD LEYDECKER.