

# UNITED STATES PATENT OFFICE.

EDUARD ULLRICH, OF HÖCHST-ON-THE-MAIN, PRUSSIA, GERMANY, ASSIGNOR TO THE FARBWERKE, VORMALS MEISTER, LUCIUS & BRÜNING, OF SAME PLACE.

## PRODUCTION OF BLUE COLORING-MATTER.

SPECIFICATION forming part of Letters Patent No. 384,480, dated June 12, 1888.

Application filed December 14, 1887. Serial No. 257,877. (No specimens.) Patented in Germany December 25, 1885, No. 33,573; in France December 27, 1885, No. 173,137, and in England January 1, 1886, No. 43.

*To all whom it may concern:*

Be it known that I, EDUARD ULLRICH, doctor of philosophy, a citizen of the Empire of Germany, residing at Höchst-on-the-Main, Prussia, in the Empire of Germany, have invented certain new and useful Improvements in Processes for Producing Blue Coloring-Matter; 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.

My invention relates to an improved process for producing the coloring-matter known as "methylene-blue," in which a mixture of paramido-dimethylaniline,  $C_6H_4 \left\{ \begin{array}{l} (1)N(CH_3)_2 \\ (4)NH_2 \end{array} \right.$ , hydrochlorate of dimethylaniline, and hyposulphite of sodium in aqueous solution, is subjected to the action of an oxidizing agent, (that apparently most suitable being potassium dichromate.)

In carrying out my invention I produce first, in a manner well known, the amido-dimethylaniline by reducing hydrochlorate of nitroso-dimethylaniline by the action of zinc and hydrochloric acid. Of the zinc I employ the quantity necessary to use up all of the hydrochloric acid employed, so that there will not remain any free hydrochloric acid. To this solution of amido-dimethylaniline, (one molecule,) containing chlor-zinc, I add hyposulphite of sodium (somewhat more than one molecule) and hydrochlorate of dimethylaniline, (one molecule,) and oxidize by the addition of potassium dichromate, finishing up the operation by heating. For one molecule of amido-dimethylaniline there will be required of the oxidizing agent the quantity corresponding to three atoms of oxygen. The dimethylaniline does not partake in the first stage of the reaction. It enters into action only when about one-third of the quantity of the oxidizing agent necessary has been added. Out of the product resulting from the reaction the methylene-blue is obtained by boiling, filtering, and precipitating from the solution by means of common salt. Analogous coloring-matters are obtained when in this process the paramido-dimethylaniline is replaced by paramido-dimethylaniline and the dimethylaniline by some other aromatic monamine—as, for example, aniline, ortho-toluidine, and their mono and di alkyl derivatives.

What I claim as new, and desire to secure by Letters Patent, is—  
The production of methylene-blue by subjecting paramido-dimethylaniline, hydrochlorate of dimethylaniline, and hyposulphite of sodium to the action of an oxidizing agent, as bichromate, with heat, substantially as set forth.

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

EDUARD ULLRICH.

Witnesses:

JOSEPH REVERDY,  
HEINRICH HAHN.