UNITED STATES PATENT OFFICE.

RICHARD LAUCH, OF ELBERFELD, GERMANY, ASSIGNOR TO THE FARBEN-FABRIKEN, VORMALS FR. BAYER & CO., OF SAME PLACE.

BROWN DYE.

SPECIFICATION forming part of Letters Patent No. 509,623, dated November 28, 1893.

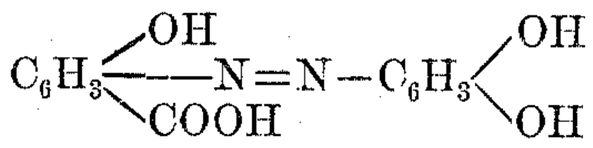
Application filed May 3, 1893. Serial No. 472,857. (Specimens.) Patented in France October 28, 1892, No. 212,713.

To all whom it may concern:

Be it known that I, RICHARD LAUCH, doctor of philosophy, chemist, and assignor to the FARBENFABRIKEN, VORMALS FR. BAYER & Co., of Elberfeld, a subject of the Emperor of Germany, residing at Elberfeld, Prussia, Germany, have invented a new and useful Improvement in the Manufacture of Brown Coloring-Matters; and I do hereby declare the following to be a full, clear, and exact description of the same.

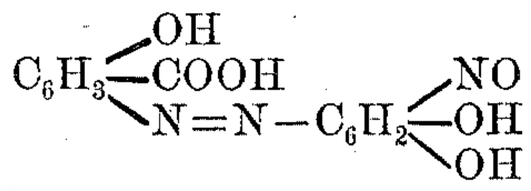
My invention (for which the above Farbenfabriken, vormals Fr. Bayer & Co., of Elberfeld, have already obtained Letters Patent in France, dated October 28, 1892, No.212, 713), relates to the production of brown coloring-matters producing from yellowish-brown to reddish-brown shades on unmordanted wool as well as on wool mordanted with metallic salts (such as iron or chromium mordants) and also dyeing cotton which is mordanted in the usual manner. The new dye-stuffs result from the action of nitrous acid (or other agents producing the same effect) on the intermediate product obtainable by allowing one molecular proportion of diazotized aromatic amidocarbonic acid to act on one molecular proportion of resorcinol or orcinol, referred to in the patent to Schmid of May 12, 1891, No. 452,197.

In carrying out my invention, I proceed as follows: 2.75 parts, by weight, of the intermediate product having the formula:



obtainable by the action of one molecular proportion of diazotized para amidosalicylic acid on one molecular proportion of resorcinol (as described in the United States patent to Jakob Schmid, dated May 12, 1891, No. 452,197) are dissolved in the necessary quantity of water with the addition of a solution in water of 0.4 parts, by weight, of sodium hydroxid. Then 0.7 parts, by weight, of sodium nitrite, dissolved in about 3.5 parts, by weight, of water are added, and the mixture is carefully cooled by means of ice. On continuous stirring an excess of hydrochloric acid is gradually poured into the resulting mixture.

The formation of the dye-stuff, which has probably the following composition:



$$=C_{13}H_9N_3O_6$$

begins almost immediately and is completed by allowing the reaction mixture to stand for from about twelve to fifteen hours. Owing to its only moderate solubility in cold water the dye-stuff separates. It is then purified by filtering off and washing out.

Similar or analogous dye-stuffs are obtained, if instead of the above used para-amidosalicylic acid the isomeric amidohydroxy carbonic acids or other amidocarbonic acids are employed, or if instead of resorcinol the homologous orcinol is employed.

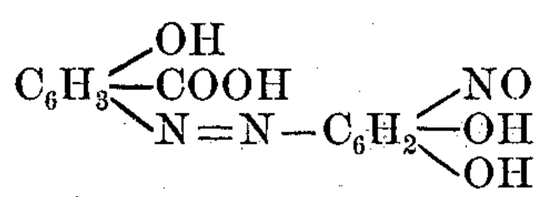
The above coloring-matter obtained from para amidosalicylic acid as hereinbefore described forms a dark-brown paste, soluble in cold water with great difficulty, more easily soluble in hot water, with a yellowish-brown color, easily soluble in alcohol with the same color. It is dissolved by sodium carbonate with a yellowish-brown color, by soda-lye with a dark-brown color. With concentrated sulfuric acid (66° Baumé) it forms a brown solution, the coloring-matter being partly separated on the addition of ice-water to the sulfuric acid solution.

The dye-stuff yields brown shades on unmordanted wool as well as on wool mordanted with metallic mordants (such as iron and chromium salts) and is also suitable for dyeing cotton mordanted in the usual manner. Especially it possesses a great value for printing purposes, as it yields when fixed on the fiber by means of the well known methods, very fast and clear shades. The latter are, if chromium mordants be employed, in general from yellowish-brown to reddish-brown, while in the employment of printing pastes containing iron salts much darker shades, viz, from dark-olive colored to black are obtained.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process for the production of coloring matters, consisting in combining one molecular proportion of a diazotized amido hydroxy carbonic acid of the aromatic series with one molecular proportion of resorcinol or orcinol, and acting on the intermediate body thus obtained with nitrous acid, substantially as described.

2. As a new manufacture, the dye-stuff having probably the formula:



$$=C_{13}H_9N_3O_6.$$

a dark-brown paste, soluble in cold water with great difficulty, more easily soluble in hot water and alcohol with a yellowish-brown color, dissolving in concentrated sulfuric acid (66° Baumé) with a brown color, the coloring-matter being partly separated on the addition of ice-water to the sulfuric acid solution, dyeing unmordanted wool brown, and producing on fibers mordanted with metallic mordants, either in dyeing or printing, brown shades, substantially as described.

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

RICHARD LAUCH.

Witnesses:
EDW. ESSENWEIN,
RUDOLPH FRICKE.