

UNITED STATES PATENT OFFICE.

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DYE AND PROCESS OF DYEING.

1,326,367.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that I, JULIUS A. NIEUWLAND, a citizen of the United States, residing at Notre Dame, in the county of St. Joseph and State of Indiana, have invented certain new and useful Improvements in Dyes and Processes of Dyeing, of which the following is a specification.

It is known that mercuric salts are capable of reacting with certain aromatic derivatives, particularly phenols and amino bodies, with the production of complex derivatives containing mercury (*Beilstein*, 3d edition, Vol. IV, page 1703). According to the present invention a new dye-bath is prepared by the interaction of mercuric salts and p-phenylene diamin. My invention comprises also a process whereby the fabric is dyed simultaneously with the preparation of the dye-bath.

Example I: One molecular proportion of p-phenylene diamin dissolved in water or diluted alcohol is mixed with one molecular proportion of a salt of mercury, preferably mercuric acetate or other readily hydrolyzable salt. The ensuing reaction yields a whitish cloudy precipitate, which is probably a highly complex derivative of mercuric acetate. Upon standing the mixture is found to acquire a dark color as the result of secondary reactions, the precise nature of which is not now known. Heating the mixture to temperatures above normal, and preferably to the boiling-point, causes the formation of a purple-black dye-bath, which is capable of dyeing silk and cotton without an extraneous mordant. This direct dyeing of cotton may perhaps be explained by the mordanting action and oxidizing effect of the mercuric acetate which is a component of the dye-bath.

Example II: Instead of boiling the previously mixed solutions of mercuric acetate and p-phenylene diamin, these solutions containing about equimolecular proportions of the respective compounds, may be heated separately to boiling and then mixed. In dyeing cotton for example, the well washed fabric may be immersed in the hot mercuric acetate solution, or boiled therein, and then transferred to a heated or boiling alcoholic (dilute) p-phenylene diamin solution, preferably after an intermediate washing with

water or dipping in a solution of zinc chlorid or sodium chlorid. The order of dipping is important, the fabric being first immersed in the mercuric salt solution. A material excess of acetate, either in preparation or in dyeing, tends to injure the color.

That the secondary reaction resulting in the formation of the new dye-bath is probably of an oxidizing character is indicated by the fact that some metallic mercury may be deposited, with partial destruction of the dye, in case a considerable excess of mercuric salt is used.

Efforts to isolate the new dye have as yet resulted negatively, since evaporating the solution even at ordinary temperatures results in the deposition of an insoluble black compound which is probably a lake.

The color on cloth is not immediately attacked by dilute acids, nor to any material degree by dilute alkalis. By the action of strong hydrochloric acid upon dyed silk, the fabric is dissolved, leaving some of the dye in solution.

Example III: The dyeing processes of Examples I and II may advantageously be combined in case an especially deep color is desired, in the following manner:—

The well washed fabric is boiled first in mercuric acetate, then in an aqueous or dilute alcoholic solution of p-phenylene diamin and is finally transferred to a heated or boiling equimolecular mixture of the two compounds. By this procedure the color may be deepened without subjecting the dyed fabric to the injurious effect of the mercuric acetate solution.

I claim:—

1. The herein-described method of preparing a dye-bath, which consists in reacting with a mercuric salt upon a solution of p-phenylene diamin at a temperature above normal.

2. The herein-described method of dyeing fabrics, which consists in subjecting the fabric to the successive action of solutions containing respectively a mercuric salt and p-phenylene diamin.

3. The herein-described method of dyeing fabrics, which consists in subjecting the fabric to the successive action of solutions containing respectively a mercuric salt and p-phenylene diamin, and then further sub-

jecting the dyed fabric to a solution containing reaction products of a mercuric salt and p-phenylene diamin.

4. A dye-bath containing the reaction-
5 products of a mercuric salt and p-phenylene diamin.

5. A dye-bath containing the reaction-

products of mercuric acetate and p-phenylene diamin.

6. A dye-bath containing the product of 10
secondary decomposition of mercuric derivatives of p-phenylene diamin.

In testimony whereof, I affix my signature.

JULIUS A. NIEUWLAND.