

UNITED STATES PATENT OFFICE.

DANIEL AUGUSTE ROSENSTIEHL, OF PARIS, FRANCE.

PROCESS OF MAKING AZO COLORS.

SPECIFICATION forming part of Letters Patent No. 483,368, dated September 27, 1892.

Application filed March 2, 1892. Serial No. 423,508. (Specimens.) Patented in France December 17, 1889, No. 202,675; in Germany December 20, 1889, No. 56,456, and in England April 15, 1890, No. 5,736.

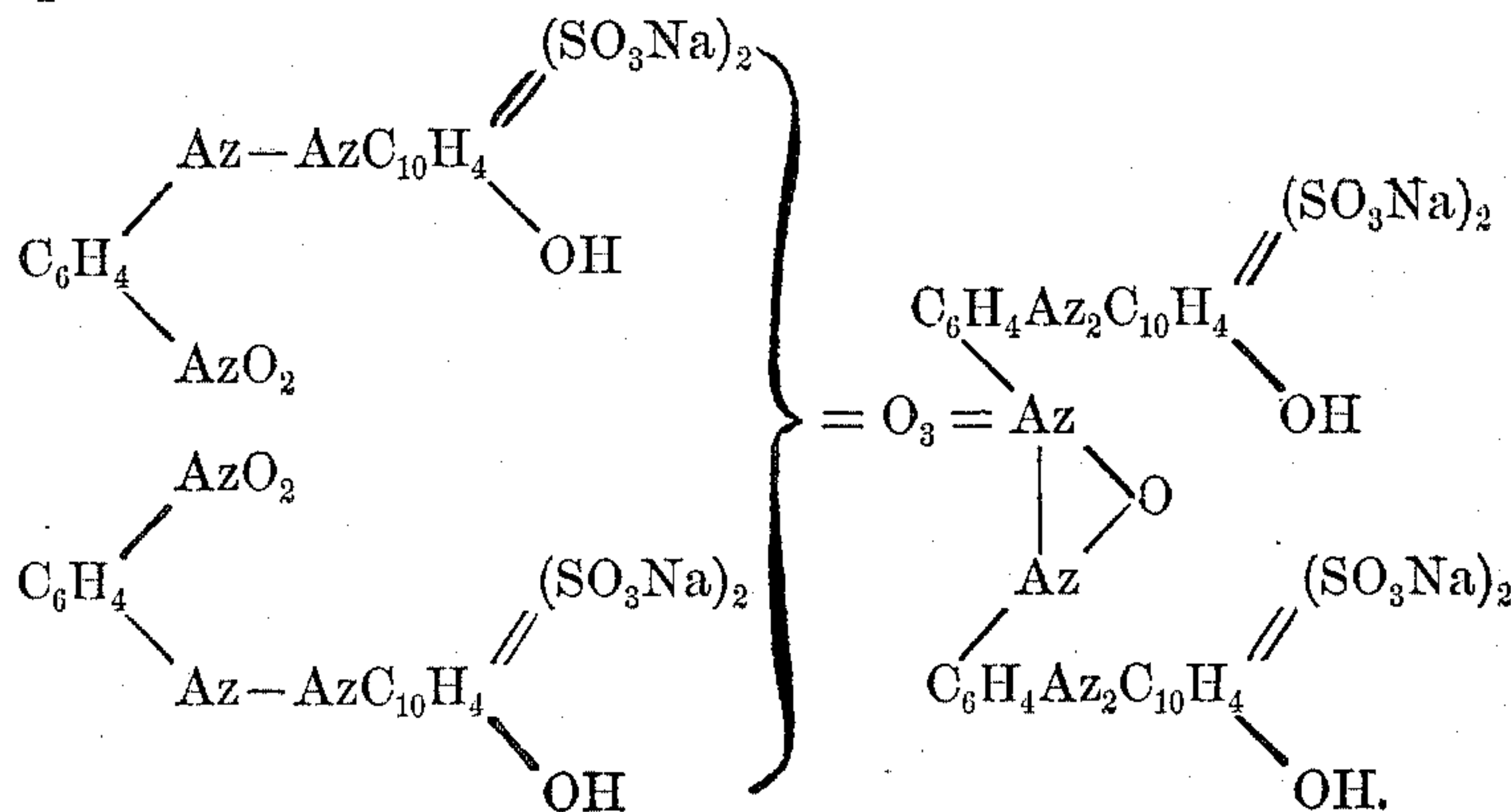
To all whom it may concern:

Be it known that I, DANIEL AUGUSTE ROSENSTIEHL, a resident of Paris, in the Republic of France, have invented a new and useful Improvement in the Manufacture of Azo Coloring-Matter, (for which I have obtained Letters Patent in France, No. 202,675, dated December 17, 1889; in England, No. 5,736, dated April 15, 1890, and in Germany, No. 56,456, dated December 20, 1889,) which improvement is fully set forth in the following specification.

The present invention relates to the production of azoic coloring-matters, derivatives of the azoxyamines; and it consists in subjecting the product obtained by the union of nitramine with phenols and amines or their

sulphonated, carboxylated, or hydroxylated derivatives to reduction in an alkaline medium, (using any suitable reducing agent,) thus transforming said products into derivatives of the azoxyamines, which have the property of dyeing unmordanted vegetable fiber.

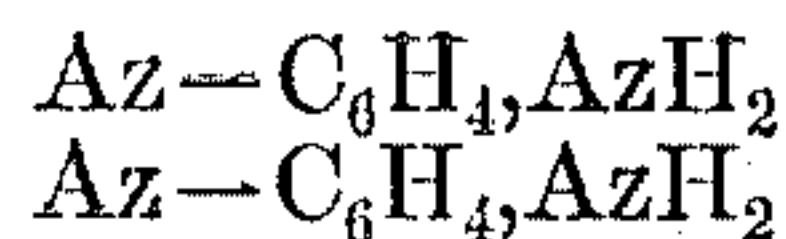
As an example of the process I will describe one reaction with the chemical formula thereof. I treat two molecules of the coloring-matter—such as obtained by the union or paranitro-diazobenzene with acid beta-sulphonaphthol—with a reducing agent, (zinc, arsenious acid, glucose, &c.) the object being to remove three parts of the oxygen contained in the nitro-diazobenzene, according to the following formula:



Two molecules of azo coloring-matter derived from the paranitraniline, less about three atoms of oxygen, } = One molecule of azo coloring-matter derivation of azoxyaniline.

In a former patent, No. 380,927, granted April 10, 1888, to Poirrier and Rosenstiehl is described a process in which a nitramine is first transformed into an azoxyamine and then coupled with a phenol; but the process herein described is very advantageous in point of economy. For example, to prepare para-azoxyaniline with para-nitraniline it is necessary to treat the latter by boiling with powder of zinc or salt-water. The insolubility of the nitramine on one hand and of the zinc on the other renders reaction incomplete, and the action frequently proceeds beyond the desired point. Instead of obtaining the en-

tire theoretical yield of para-azoxyaniline, products of a more advanced reduction are obtained at the same time, such as para-azoxyaniline,



and the paraphenylene-diamine,



products of which the second at least constitutes a notable loss. On the other hand, by coupling at first the nitramine with sulphonaphthol the result is a product soluble in water, upon which the reducing agents act uni-

formly. Thus the difficulties pointed out above are avoided, no insoluble matters have to be dealt with, the reaction does not go beyond the desired point, and the theoretical quantity of the desired coloring-matter is obtained.

Example I.—Paranitraniline is diazotized in acid solution by known processes and is coupled with bisulpho-naphthol. The coloring-matter thus obtained, which dyes wool red in an acid bath, but not cotton, serves as the basis in the process to be described. Fifty-five kilos of this red coloring-matter are dissolved in two thousand seven hundred and fifty liters of water, rendered alkaline by means of seventy-six liters of caustic soda-lye at 40° Baumé. The solution is heated to 80° centigrade, and there is poured into the same under stirring a solution of 17.5 kilos of glucose in three hundred and fifty liters of water. At the end of an hour the new coloring-matter is precipitated by common salt, which coloring-matter possesses the property of dyeing unmordanted cotton blue in an alkaline bath.

Example II.—Nitro-orthotoluidine, fusible at 127° centigrade, is diazotized and coupled with alphasulpho-alphanaphthol, following for this preparation the general method well known to all chemists. 43.5 kilos of this red coloring-matter are dissolved in two thousand two hundred liters of water and forty liters of caustic-soda solution of 40° Baumé. This solution is heated to boiling-point, and there are added fifteen kilos of arsenious acid, taking care to thoroughly agitate the mass. At the end of one hour common salt is added, which precipitates the new coloring-matter in the form of a crystalline powder with green metallic luster. This matter dyes unmordanted cotton violet in an alkaline solution.

Example III.—Nitro-orthotoluidine, fusible at 107° centigrade, is diazotized and coupled with alphasulpho-alphanaphthol. Forty-

five kilos of the red coloring-matter thus obtained are dissolved in two thousand two hundred and fifty liters of water, to which are added fifty-six liters of caustic-soda lye at 40° Baumé. After heating the solution to boiling-point there is introduced into it in small quantities at a time and under agitation 13.75 kilos of zinc powder of eighty per cent. of pure metal. At the end of an hour and a half the reaction is complete, and the new coloring-matter is precipitated by common salt. This coloring-matter dyes cotton red in an alkaline bath.

As reducing agents the above processes have been named zinc powder, glucose, and arsenious acid; but it is to be understood that other reducing agents may be employed, the essential feature of the invention consisting in taking the azo coloring-matter resulting from the coupling of a nitramine with a phenol, an amine, or their sulphonated carboxylated, or hydroxylated derivatives, and by a reduction in an alkaline medium transforming the same into coloring-matters derived from azoxyamines, which have the advantage over the first named that they dye vegetable fiber without mordanting.

Having now particularly described my said invention, what I claim, and desire to secure by Letters Patent, is—

The process of producing coloring-matters, consisting in coupling a nitramine with a phenol, amine, or the specified derivative thereof and subjecting the product thus obtained to the action of a reducing agent in an alkaline medium, substantially as described.

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses.

DANIEL AUGUSTE ROSENSTIEHL.

Witnesses:

JULES ARMENGAUD, Jeune,
ROBT. M. HOOPER.