

UNITED STATES PATENT OFFICE.

WILLIAM H. CLAUS, ALFRED RÉE, AND LEON MARCHLEWSKI, OF MANCHESTER, ENGLAND.

BLACK SULFUR DYE.

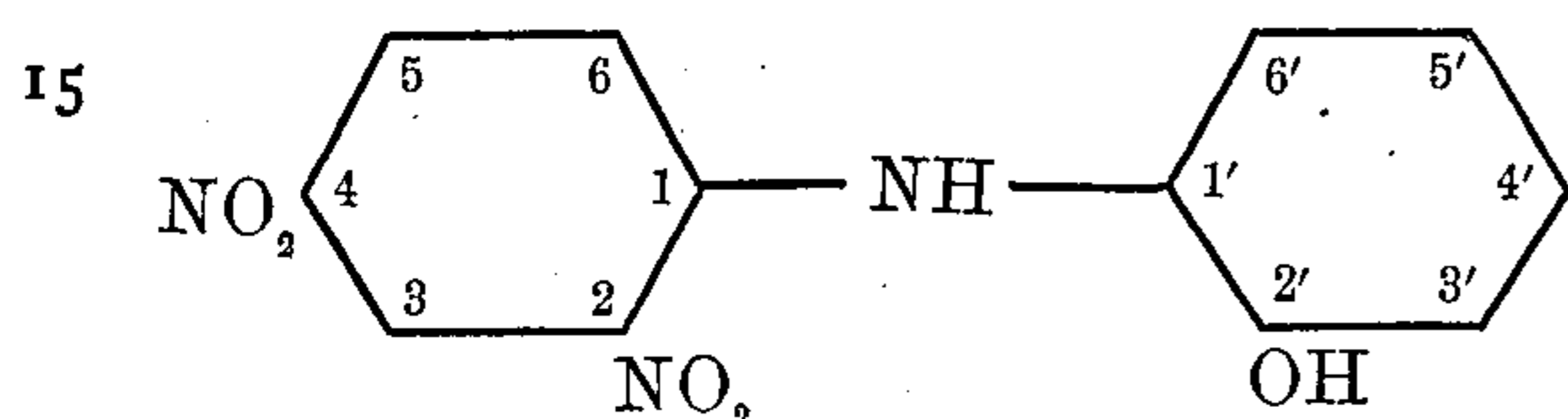
SPECIFICATION forming part of Letters Patent No. 639,806, dated December 26, 1899.

Application filed August 14, 1899. Serial No. 727,172. (No specimens.)

To all whom it may concern:

Be it known that we, WILLIAM HENRY CLAUS, ALFRED RÉE, and LEON MARCHLEWSKI, citizens of Great Britain, residing at Manchester, in the county of Lancaster, England, have invented certain new and useful Improvements in Black Coloring-Matters, of which the following is a full, clear, and exact description.

10 We have found that a black coloring-matter can be obtained by heating with sulfur and an alkaline sulfid dinitro-ortho-hydroxydiphenylamin of the constitution:



25 The following will serve as an example of the invention: Five parts of dinitro-ortho-hydroxydiphenylamin, ten parts of sulfur, thirty parts of crystallized sodium sulfid, and ten parts of water are heated for several hours at 100° to 110° centigrade and the temperature is then gradually raised to 140° centigrade, at which temperature the melt is kept for about three hours, when it is finally

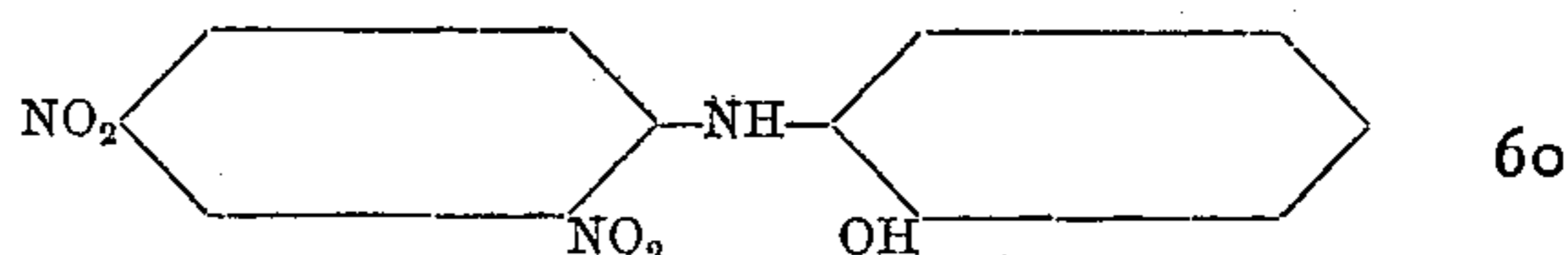
30 raised to 150° to 160° centigrade. There is thus obtained a black mass, which dissolves in water with a greenish-blue color and which can be used directly for dyeing. It gives greenish shades of black on unmordanted

35 cotton, which may be further developed by aging or by an after treatment with bichro-

mates or with copper salts in presence of acetic acid, or with a mixture of these salts. The shades obtained are extremely fast to light, acids, and alkalies. Our discovery that certain compounds in which the OH group is in ortho position to an NH group give on heating with sulfur and alkaline sulfids black coloring-matters of exceptional fastness contradicts the hitherto prevalent belief that only compounds may be used for this purpose which contain an hydroxy group in para position to an NH group. (Compare *Revue Générale des Matières Colorantes*, Vol. III, No. 30, p. 214.)

50 Having now described our invention, what we claim as new is—

The process for the production of black coloring-matter capable of directly dyeing cotton fiber, which consists in heating dinitroortho-hydroxydiphenylamin having the constitution



with sulfur and an alkaline sulfid, substantially as described.

In witness whereof we subscribe our signatures in presence of two witnesses.

WILLIAM H. CLAUS.
ALFRED RÉE.
LEON MARCHLEWSKI.

Witnesses:

WILLIAM GEO. HEYS,
ARTHUR MILLWARD.