

UNITED STATES PATENT OFFICE.

BENNO HOMOLKA, OF FRANKFORT-ON-THE-MAIN, AND FRIEDRICH VON BOLZANO, OF HÖCHST-ON-THE-MAIN, GERMANY, ASSIGNORS TO FARBWERKE, VORM. MEISTER, LUCIUS & BRÜNING, OF HÖCHST-ON-THE-MAIN, GERMANY, A CORPORATION OF GERMANY.

PROCESS OF MAKING INDOXYL.

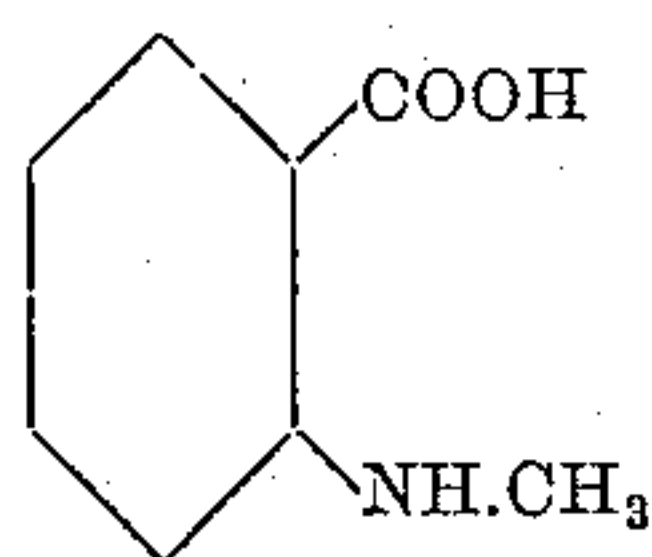
SPECIFICATION forming part of Letters Patent No. 712,798, dated November 4, 1902.

Application filed May 13, 1902. Serial No. 107,181. (No specimens.)

To all whom it may concern:

Be it known that we, BENNO HOMOLKA, Ph. D., a citizen of the Empire of Germany, residing at Frankfort-on-the-Main, and FRIEDRICH VON BOLZANO, Ph. D., a citizen of the Empire of Austria-Hungary, residing at Höchst-on-the-Main, Germany, have invented certain new and useful Improvements in the Manufacture of Indoxyl from Methylantranilic Acid or Certain Derivatives Thereof, of which the following is a specification.

We have found that indoxyl may be obtained if sodium amid is allowed to act at a high temperature on the salts of methylantranilic acid



or on the salts of methylantranilic acids having an acidyl group attached to the nitrogen, especially those of formyl and acetyl methylantranilic acids. If, for instance, a mixture of about equal parts of potassium methylantranilate or potassium formylmethylantranilate and sodium amid be heated, an orange-yellow product is obtained, which dissolved in water yields indigo when exposed to the air. However, the action of sodium amid on the said salts is extremely lively, often accompanied by combustion and partial decomposition of the organic substance. It is therefore advisable to moderate the reaction by adding suitable solvents, such as the hydroxid or the cyanid of an alkali metal, especially potassium hydroxid or sodium hydroxid, potassium cyanid or sodium cyanid, or mixtures of these bodies.

This invention relates to the manufacture of indoxyl from methylantranilic acid or its acidyl derivatives by the foregoing reaction.

The following example illustrates the process: A thorough mixture of two hundred parts,

by weight, of potassium methylantranilate or the equivalent proportion of another alkali or alkaline-earth methylantranilate, one hundred and twenty parts of sodium amid, and two hundred to three hundred parts of caustic potash or caustic soda, or a mixture thereof, is melted in a closed vessel, preferably without access of air, and kept in the fused state as long as ammonia is evolved, which may be recognized by the froth of the melt. When cool, the orange-yellow mass is dissolved in water and the dissolved indoxyl is worked up by oxidation in the known manner to indigo.

If on heating the cyanid of an alkali is used as a diluent, the general process is the same. For instance, a mixture of about equal parts of potassium methylantranilate, sodium amid, and potassium cyanid is heated without access of air. The phenomena in the course of the melt are the same as in the first example; also, the further working up of the product to indigo or indoxyl.

In the above examples the equivalent proportion of formylmethylantranilate or acetylmethylantranilate may be substituted for methylantranilate.

Having now described our invention, what we claim is—

The herein-described process for the manufacture of indoxyl, which consists in causing sodium amid to act at a high temperature in the presence of suitable diluents, on the salts of methylantranilic acid, whereby the amid-hydrogen atom of the methylantranilic acid may be substituted by an acid radical, substantially as set forth.

In testimony that we claim the foregoing as our invention we have signed our names in presence of two subscribing witnesses.

BENNO HOMOLKA.

FRIEDRICH VON BOLZANO.

Witnesses:

ALFRED BRISBOIS,

HEINRICH HAHN.