UNITED STATES PATENT OFFICE.

MARTIN MUGDAN AND WILLY HERRMANN, OF NUREMBERG, GERMANY, ASSIGNORS TO THE FIRM OF CONSORTIUM FÜR ELEKTROCHEMISCHE INDUSTRIE, GESELLSCHAFT MIT BESCHRÄNKTER HAFTUNG, OF NUREMBERG, GERMANY.

PROCESS OF MAKING INDOXYL, &c.

960,671. Specification of Letters Patent. Patented June 7, 1910.

No Drawing. Application filed January 25, 1910. Serial No. 540,011.

To all whom it may concern:

Be it known that we, MARTIN MUGDAN, doctor of philosophy, chemist, and WILLY HERRMANN, doctor of philosophy, chemist, subjects of the King of Prussia, German Emperor, residing at 54 Gugelstrasse, Nuremberg, Kingdom of Bavaria, German Empire, have jointly invented new and useful Improvements in Processes of Manufacturing Indoxyl and Derivatives Thereof, of which the following is a specification.

This invention relates to a process for the production of indoxyl and derivatives thereof from aryl-glycin bodies. It is already known that phenylglycin and its derivatives and homologues, on being heated with alkali hydroxids can be converted into indoxyl, or its derivatives, or homologues, and that indigo may be obtained from the indoxyl thus obtained by oxidation for instance by blowing air into the aqueous solution of the melt. The yield of indigo in this case, however, is very bad. We have found that indoxyl and derivatives thereof can be obtained from aryl-glycin bodies by heating these compounds with alloys of silicon and metals of the alkaline earths in the presence of anhydrous caustic alkalies. The process yields good results and is easily carried out, as the alloys of silicon and metals of the alkaline earths are readily and cheaply obtained.

The following are examples showing how the invention is performed but the invention is not limited to these.

Example I: Into an anhydrous melt of 280 parts by weight of caustic potash and of 200 parts by weight of caustic soda, are slowly introduced at about 250° centigrade, 50 parts by weight of pulverized calcium-silicon (containing about 30 per cent. calcium). The calcium-silicon alloy dissolves while a brisk evolution of hydrogen takes place. While the melt is kept well stirred, 50 parts by weight of finely pulverized phenylglycin potassium are introduced at a temperature of 220° centigrade. The temperature is then raised to about 250° centigrade and the reaction is interrupted when blowing air through a sample dissolved in water the amount of the dye formed no longer increases. The melt is dissolved in 10000 parts by weight of water at 100° centigrade, and the solution thus obtained which contains the indoxyl, is filtered off from any insoluble substances produced. If it be desired to obtain the indoxyl in the solid state, the solution is evaporated to dryness in the presence of an inert gas such as hydrogen. As the indoxyl is generally prepared for the purpose of obtaining indigo, its isolation may be omitted and the indigo prepared directly by introducing air into the solution. The indigo dye thus obtained is of great purity.

In carrying out the process the quantitative proportions and temperatures may be varied. It has been found advantageous to introduce the alloy of silicon and alkaline earth metal intimately mixed with the aryl-glycin body into the melt of caustic alkalies.

Example II: Into an anhydrous melt of 300 parts by weight of caustic soda and 500 parts by weight of caustic potash is slowly introduced while stirring, at a temperature of about 220° centigrade, an intimate mixture of 200 parts by weight of phenylglycin-potassium and 130 parts of calcium-silicon (containing about 30 per cent. of calcium). The temperature is then raised to 250° centigrade and the process carried out as described in Example I.

In the above examples the calcium-silicon can be replaced by alloys of silicon with other metals of the alkaline earths, for instance barium-silicon may be made.

Instead of phenylglycin-potassium other compounds of phenylglycin can be used, for instance phenylglycin-sodium, phenylglycin-amid or phenylglycin-anilid.

If it be desired to produce homologues of indoxyl homologues of phenylglycin are used. From tolyl-glycin-potassium

$$CH_3.C_6H_4.NH.CH_2COOK$$

methyl indoxyl is obtained.

We claim:

1. A process for producing indoxyl and derivatives thereof by heating an aryl-glycin body with an alloy of silicon and a metal of

the alkaline earths in the presence of anhydrous caustic alkalies.

2. A process for producing indoxyl and derivatives thereof by introducing a mixture of an alloy of silicon and a metal of the alkaline earths and of an aryl-glycin body into melted anhydrous caustic alkalies.

In testimony whereof we affix our signatures in presence of two witnesses.

MARTIN MUGDAN.
WILLY HERRMANN.

Witnesses:
ERICK HOEFER,
ADAM KISCHEA.