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UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING INDOXYL AND DERIVATIVES.

SPECIFICATION forming part of Letters Patent No. 756,171, dated March 29, 1904.

Application filed September 9, 1902. Serial No. 122,701. (No specimens.)

To all whom it may concern:

Be it known that we, RUDOLF KNIETSCH, doctor of philosophy, a subject of the King of Prussia, German Emperor, PAUL SEIDEL, doctor of philosophy and chemist, a subject of the King of Saxony, and GEORG WILHELM MEISER, doctor of philosophy and chemist, a subject of the King of Bavaria, all residing at Ludwigshafen-on-the-Rhine, in the Kingdom of Bavaria, Empire of Germany, have invented new and useful Improvements in Processes for the Manufacture of Indoxyl and Derivatives, of which the following is a specification.

It is known that on heating phenyl glycin or a homologue thereof at a high temperature with an alkaline hydroxid or hydroxids, either alone or in admixture, leuco compounds of the indigo series are formed—namely, indoxyl or derivatives thereof. We have discovered that alkali-metal oxids—for instance, potassium or sodium oxid or mixtures of these two oxids—can be advantageously used instead of alkali hydroxid to effect the formation of indoxyl or derivatives thereof. We hereinafter employ the term "alkali oxid" to mean sodium oxid or potassium oxid or a mixture of sodium oxid and potassium oxid. For instance, if a phenyl-glycin salt and sodium oxid be intimately mixed, even at the ordinary temperature, then on being treated with water an abundant formation of indoxyl or indigo results, whereas if an alkali hydroxid is employed a high temperature, as aforesaid, is necessary to bring about this result. Good results are obtained when the aforesaid alkali oxids are employed in the presence of a suitable diluent, such as alkali hydroxid or an alkali-earth oxid. The alkali oxids used in our new process can be obtained in the known manner, (see Dammer, *Handbuch der Anorganischen Chemie*, Vol. II, part 2, pages 8 and 116)—for example, by heating together sodium and sodium superoxid or potassium and potassium hydroxid or in any other convenient manner.

The following examples will serve to exemplify the nature of our invention, which, however, is not confined to these examples. The parts are by weight.

Example 1: Intimately mix together ten (10) parts of phenyl-glycin potassium salt and fourteen (14) parts of sodium oxid and heat the mixture for about one (1) hour at a temperature of about two hundred and fifty degrees centigrade, (250° C.,) while excluding air. When cold, dissolve the melt in water and work it up to indigo.

Example 2: Mix together fifteen (15) parts of caustic potash and twenty-five (25) parts of an alkaline mixture containing sodium oxid. This mixture can be obtained, for example, by introducing ten (10) parts of metallic sodium into a molten mixture of thirty (30) parts of anhydrous caustic potash and twenty (20) parts of sodium superoxid. Into the aforesaid mixture of caustic potash and alkali mixture containing sodium oxid introduce, while stirring, at a temperature of about two hundred and fifty degrees centigrade (250° C.) ten (10) parts of phenyl-glycin potassium salt and heat for about one (1) hour at a temperature of two hundred and forty degrees centigrade, (240° C.) When cold, work up the melt.

Example 3: Thoroughly free caustic potash from moisture and then convert it into oxid by subjecting it to electrolysis as long as can be done without free metallic potassium becoming noticeable or until free metallic potassium is beginning to be formed. Then discontinue the electrolysis. Mix three hundred (300) parts of potassium hydrate which has been so treated with one hundred and fifty (150) parts of anhydrous caustic soda, heat the mixture to two hundred and forty degrees centigrade, (240° C.,) and add thereto one hundred (100) parts of phenyl-glycin potassium salt and work up the melt.

Example 4: Into a molten mixture of two thousand (2,000) parts of potassium hydroxid, fifteen hundred (1,500) parts of sodium hydroxid, and five hundred (500) parts of quicklime introduce, while stirring, at a temperature of two hundred to two hundred and

twenty degrees centigrade (200°-220° C.) sufficient metallic sodium to effect the formation of alkali oxid. Then add an intimate mixture of one thousand (1,000) parts of phenyl-glycin potassium salt and five hundred (500) parts of quicklime and maintain the temperature for about one hour at two hundred and fifty degrees centigrade, (250° C.) When cold, work up the melt in the known manner. If desired, the quicklime can be replaced by strontium oxid or barium oxid.

In the foregoing example the quantity of sodium must be so chosen that no excess is present, the object being merely to produce sodium oxid, and the action of sodium on the phenyl glycin is to be altogether avoided.

Example 5: Melt together four thousand (4,000) parts of potassium hydroxid, fifteen hundred (1,500) parts of sodium hydroxid, and five hundred (500) parts of sodium oxid. Introduce into the melt at a temperature of two hundred to two hundred and twenty degrees centigrade, (200°-220° C.,) while stirring, one thousand (1,000) parts of phenyl-glycin potassium salt and one hundred and thirty (130) parts of metallic sodium. Heat for about one (1) hour at a temperature of two hundred and fifty degrees centigrade (250° C.) and when cold work up the melt.

We claim—

1. The process of making indoxyl and derivatives thereof by treating a phenyl-glycin body with an alkali oxid.

2. The process of making indoxyl and derivatives thereof by heating a phenyl-glycin body with an alkali oxid in the presence of a suitable diluent.

3. The process of making indoxyl and derivatives thereof by heating a phenyl-glycin body with sodium oxid.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

RUDOLF KNIETSCH.
PAUL SEIDEL.
GEORG WILHELM MEISER.

Witnesses:
JOHN L. HEINKE,
JACOB ADRIAN.