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The Preparation of m-Hydroxybenzotrile

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panying some of these transformations are ring fission and ring closure, and rearrangements involving unsaturated linkages. Typical illustrations of these changes were presented.

IOWA STATE COLLEGE,
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THE TOXICITIES OF SOME ORGANO-LEAD COM-
POUNDS FOR CANCER AND RELATED STUDIES

HENRY GILMAN, O. M. GRUHZIT, J. D. ROBINSON
AND E. B. TOWNE

In connection with the application of organo-lead compounds in cancer, some plant diseases and as anti-knock reagents, a pharmacological study has been made of the relative toxicities of a miscellany of products derived from alkyl and aryl lead compounds. Some of the salts reported at this time have varying appreciable solubilities in water.

IOWA STATE COLLEGE,
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THE PREPARATION OF *m*-HYDROXYBENZONITRILE

JAMES B. CULBERTSON, ERWIN L. CARPENTER
AND ERNEST K. NIELSEN

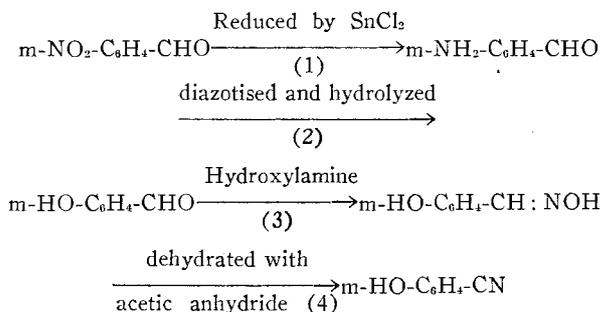
It has been a general project in this laboratory to prepare the three monohydroxy diphenyl ketimines upon which certain studies have been planned. Last year the preparation of the 2-hydroxy and 4-hydroxy diphenyl ketimines were reported in these Proceedings. The present work has been directed toward the 3-hydroxy diphenyl ketimine. The former pair of ketimines were obtained by the action of the Grignard reagent, magnesium phenyl bromide, upon the corresponding hydroxybenzotriles. To employ this same synthesis for the 3- or *m*-hydroxy diphenyl ketimine it has been necessary to prepare the appropriate hydroxybenzotrile. Much greater difficulty has arisen in the preparation of this last nitrile than with the other two.

Ahrens¹ reported the preparation of this nitrile through the application of the Sandmeyer reaction to *m*-aminophenol, replacing the amine group by the nitrile. We have employed his directions

¹ Ahrens, Ber. 20, 2953-54 (1887).

with failure. A number of variations have been tried upon this general procedure with no better success. It seems that Ahrens has specified too small a quantity of acid in the diazotisation so that coupling easily takes place through the influence of the phenolic group in the molecule being diazotised. In fact we attribute all of the failures in the varied procedures to this coupling which we have found unavoidable.

We have succeeded in making this nitrile by the following series of reactions, portions of which have been described by various investigators.² Steps (1) and (2) were carried out without isola-



tion of the amino-aldehyde. The greatest difficulty encountered was in the isolation of the hydroxyaldehyde from the tin salt-containing mixture. Ether extraction took up much of the tin salts. After evaporation of the ether the tin was removed from a hot aqueous solution of the mixture by addition of ammonium hydroxide. Aldehyde carried down by the tin hydroxide precipitate was recovered by extraction with alcohol. We found the aldehyde best purified by crystallization from hot carbon tetrachloride after treatment with Norit. (We also found the oxime and the nitrile to be best purified from carbon tetrachloride). Most satisfactory results were obtained in step (4) by omitting the isolation of the acetyl-phenolic compound produced. After the reaction with acetic anhydride was complete, the acetylated product was precipitated as an oil on the addition of water, the oil was saponified with warm 10% potassium hydroxide solution, the acidified product extracted with ether and the nitrile remaining after evaporation of the ether was purified by crystallization from carbon tetrachloride.

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² Tiemann and Ludwig, Ber. 15, 2044 (1882). Rieche, Ber. 22, 2347 (1889). Clem, Ber. 24, 826 (1891).