

1940

Behavior of Some P-Hydroxybenzaldehyde Derivatives toward Oxidizing Agents

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Recommended Citation

Perry, R. P. and Raiford, L. Charles (1940) "Behavior of Some P-Hydroxybenzaldehyde Derivatives toward Oxidizing Agents," *Proceedings of the Iowa Academy of Science*, 47(1), 264-265.

Available at: <https://scholarworks.uni.edu/pias/vol47/iss1/54>

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As an example, *n*-octadecyl amine and acetic acid gives a 24 per cent yield of salt melting at 84.5-85°. Upon heating this salt at 225° for fifteen minutes there is a quantitative conversion to *n*-octadecylacetamide (m.p., 78-78.5°).

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PYRROLIDINES FROM PRIMARY AMINES

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HAROLD A. HOPPENS

Pyrrolidine has been prepared from *n*-butylamine by heating *N*-chloro-*N*-acyl derivatives of the amine with concentrated sulfuric acid.

N-chloro-*N*-butylacetamide underwent ring closure with the formation of pyrrolidine in 50 per cent yields when heated with 95 per cent sulfuric acid for one hour at 130-140°. With 99.5 per cent acid the percentage yield was much lower.

N-chloro-*N*-*n*-butyl-*p*-toluenesulfonamide when heated with 95 per cent sulfuric acid for thirty minutes at 140° formed pyrrolidine in 50 per cent yields.

In both cases considerable amounts of *n*-butylamine were also formed.

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BEHAVIOR OF SOME P-HYDROXYBENZALDEHYDE DERIVATIVES TOWARD OXIDIZING AGENTS

R. P. PERRY AND L. CHARLES RAIFORD

Benzaldehyde¹ and its alkyl, alkoxy and halogen substitution products are readily oxidized to the corresponding acids. Contact with air often brings about the change.² Bücking,³ and Fittig and

¹ Wöhler and Liebig, *Ann.*, 3,250 (1832).

² Bornemann, *Ber.*, 17, 1466 (1884).

³ Bücking, *Ber.*, 9,529 (1876).

Remsen ⁴ found that p-hydroxybenzaldehyde and protocatechuic aldehyde, respectively, are not readily oxidized by potassium permanganate solution. Tiemann ⁵ could not oxidize vanillin, the 3-methyl ether of protocatechuic aldehyde, into vanillic acid by the usual methods, and Brady and Dunn ⁶ failed to oxidize 5-bromovanillin with acetic acid solution of chromic acid and also by alkaline solution of potassium permanganate.

The behavior of many vanillin substitution products ⁷ toward the last-named reagent is now being studied. If the hydroxyl group in position 4 is exposed almost all the starting material is recovered. When the alkyl derivatives are used the corresponding acids are obtained. Results thus far indicate that the weight of the alkyl group may determine the yield of product. When a heavy alkyl is present the yield is low.

⁴ Fittig and Remsen, *Ann.*, 159, 150 (1871).

⁵ Tiemann, *Ber.*, 9, 415 (1876).

⁶ Brady and Dunn, *J. Chem. Soc.*, 107, 1859 (1915).

⁷ Raiford and Wells, *J. Am. Chem. Soc.*, 57, 2500 (1935).

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IODINATION OF ANISOLE

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As a rule direct iodinations do not proceed readily, often explained as due to the unfavorable equilibrium attained with the reverse reaction of hydrogen iodide upon the iodo-compound. Iodine is more commonly introduced into an aromatic nucleus through the Sandmeyer reaction by the addition of potassium iodide to a diazotized primary aromatic amine. Thus p-iodoanisole has been prepared from p-anisidine.¹

A procedure of direct iodination for the preparation of iodoaromatic compounds would appear to offer important advantages. In 1901, Brenans² reported such a process in the preparation of p-iodoanisole through the interaction of an absolute ethyl alcohol solution of anisole with mercuric oxide and iodine. Later in 1912, Kauffmann³ used the same procedure for the iodination of resorcinol dimethyl ether to produce 1-iodo-2, 4-dimethoxybenzene, al-

¹ Reverdin, *Ber.* 29, 1000 (1896).

² Brenans, *Bull. Soc. Chim.* [3] 25, 819 (1901).

³ Kauffmann, *Ber.* 45, 2334-35 (1912).