

SHORT
COMMUNICATIONS

Oxidative Heterocyclization of 1,4-Butanediol to 4-Butanolide

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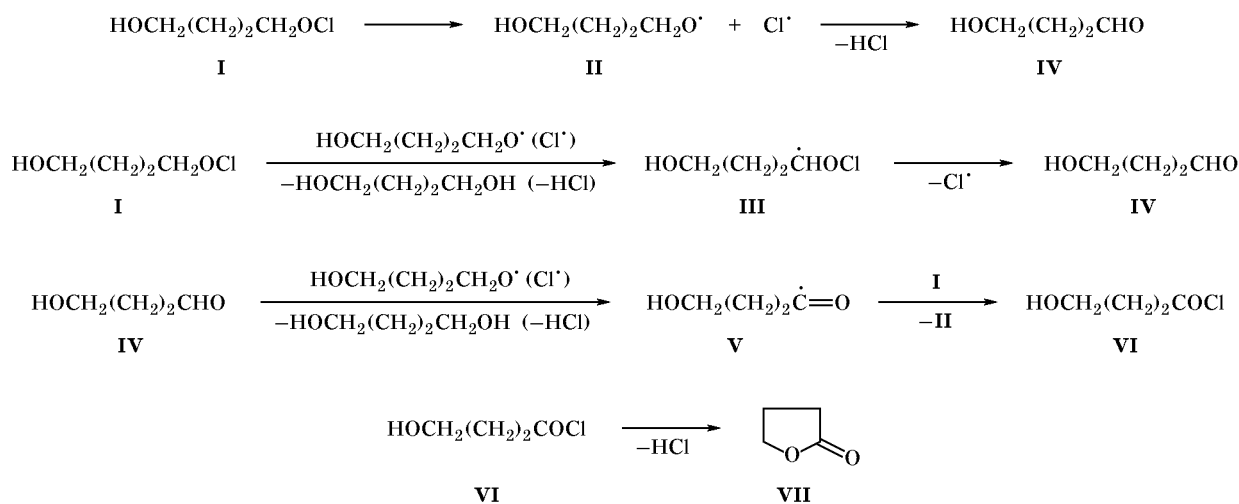
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According to Jenner [1], alkyl hypochlorites can be converted into esters on exposure to daylight in benzene or carbon tetrachloride in an inert atmosphere. The reaction was shown to involve intermediate formation of the corresponding aldehydes and then acid chlorides; the latter undergo esterification by the action of alcohol formed during the process [2]. In continuation of these studies, we examined homolytic decomposition of monohypochlorite **I** derived from 1,4-butanediol. We found that hypochlorite **I** is converted into 4-butanolide and 1,4-butanediol on exposure to daylight at 20–25°C under argon. The reaction is accompanied by liberation of hydrogen chloride, and the yield of 4-butanolide is 46–54%. Presumably, the final product is formed through steps indicated above. Comparison of our results with those reported in [3] led us to presume the reaction sequence shown in Scheme 1. Homolytic cleavage of the O–Cl

bond in **I** gives alkoxy radical **II** and atomic chlorine which abstract hydrogen atom from the most reactive α -position in hypochlorite **I**. The resulting radical **III** decomposes into aldehyde **IV** and chlorine atom. Aldehyde **IV** can also be formed via disproportionation of the primary radical pair **II**+Cl \cdot generated by cleavage of the O–Cl bond in initial hypochlorite **I**. Aldehyde **IV** reacts with radical species present in the reaction mixture to give acyl radical **V** which abstracts chlorine atom from hypochlorite **I**, yielding chloride **VI**. Heterocyclization of the latter results in formation of the final product, 4-butanolide (**VII**).

Initial hypochlorite **I** was synthesized by reaction of 1,4-butanediol with an aqueous solution of NaOCl and a benzene solution of acetic acid at 0°C [3]. When a benzene extract of hypochlorite **I** warmed up to room temperature, its vigorous decomposition started to give the corresponding products.

Scheme 1.



The products were isolated by vacuum distillation of the reaction mixture. Their physical constants were in agreement with those given in [4].

REFERENCES

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