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Microwave-Assisted Henry Reaction: Solventless Synthesis of Conjugated Nitroalkenes§

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Key Words: Arylaldehydes; Nitroalkanes; Ammonium acetate; Conjugated nitroalkenes; Henry reaction; Microwave irradiation

Abstract: In a solventless system and under microwave irradiation, nitroalkanes react with arylaldehydes in the presence of a catalytic amount of ammonium acetate to afford, in one step, conjugated nitroalkenes without the isolation of intermediary β-nitro alcohols.
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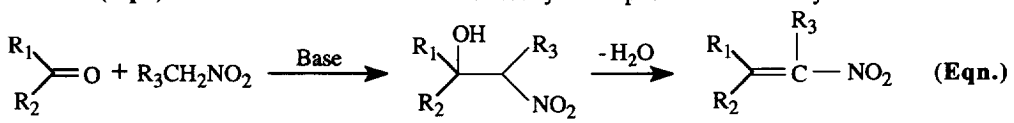
Nitroalkenes are abundantly utilized for the preparation of a variety of organic compounds[1] and possess significant biological activities such as insecticidal[2,3] fungicidal,[3-6] bactericidal,[7,8] rodent-repellent[9] and antitumor agents.[10] The utility of nitroalkenes in organic synthesis is largely due to their ease of conversion into a variety of functionalities. They are strong dienophiles in Diels-Alder reactions and alternatively, these electrophilic alkenes readily undergo addition reactions with various nucleophiles thus providing an array of valuable products.

The most versatile synthesis of nitroalkenes involves the Henry condensation reaction of a carbonyl compound **1** with nitroalkane **2** to give β-nitro alcohol **3** which undergoes dehydration affording conjugated nitroalkene **4** (**Eqn.**).[11] This condensation reaction is usually accomplished under mildly basic conditions.



1 **2** **3** **4**

Several reagents such as phthalic anhydride,[12,13] methanesulfonyl chloride,[14] dicyclohexylcarbodiimide (DCC),[15] pivaloyl chloride,[16,17] ammonium acetate-acetic acid[18,19] and amines[7,19] have been used for the dehydration of the ensuing β-nitro alcohols. However, some of these reactions require elevated temperature and may not be amenable to the dehydration of functionalized nitroalcohol intermediates.