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A one-pot three-component reactions for the synthesis of fully substituted spiro indeno[1,2-*b*]quinoxaline derivatives

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ABSTRACT

An efficient and simple approach of the synthesis of some spiro indeno[1,2-*b*]quinoxalines via a one-pot three-component reaction of 11*H*-indeno[1,2-*b*]quinoxalin-11-one, pyrazolone, and malononitrile in the presence of Na_2CO_3 at 70 °C is reported. This reaction has shown to have high atom economy.

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1. Introduction

Multicomponent reactions (MCRs) provide unmatched opportunities for the expeditious increase of complexity and diversity in synthetic outcomes. The strategy offers significant advantages over classical stepwise approaches, allowing the formation of several bonds and the construction of complex molecular architectures from simple precursors in a single synthetic operation without the need for the isolation of intermediates [1]. MCRs, particularly those performed in aqueous media, have become increasingly useful tools for the synthesis of chemically and biologically important compounds because of their environmentally friendly, atom economy and green characteristics [2,3].

Important pharmaceuticals often possess heterocyclic moieties as their building blocks. Since pyrazoles and its derivatives possess various biological activities, such as anti-inflammatory, antipyretic, gastric secretion stimulatory, antidepressant, antibacterial, and antifilarial agents [4–7], the development of new methods for the synthesis of pyrazole derivatives, which will yield subsets of heterocycles having the potentiality to serve as templates for new biologically active molecules, is of great importance.

2. Results and discussion

In continuation of our interest in the synthesis of heterocyclic compounds based on in MCRs [8], we developed herein the synthesis of spiro indeno[1,2-*b*]quinoxalines **8** via a three-component condensation reaction of 11*H*-indeno[1,2-*b*]quinoxalin-11-one **6**, pyrazolone **3** and malononitrile **7** in ethanol by using Na_2CO_3 at 70 °C in high yield (Scheme 1). It should be noted that pyrazolone **3** was synthesized by the condensation between β-keto esters **1** and hydrazines **2** in ethanol after 5 min [9]. Also, 11*H*-indeno[1,2-*b*]quinoxalin-11-ones **6** was synthesized according to a previous work [10] by

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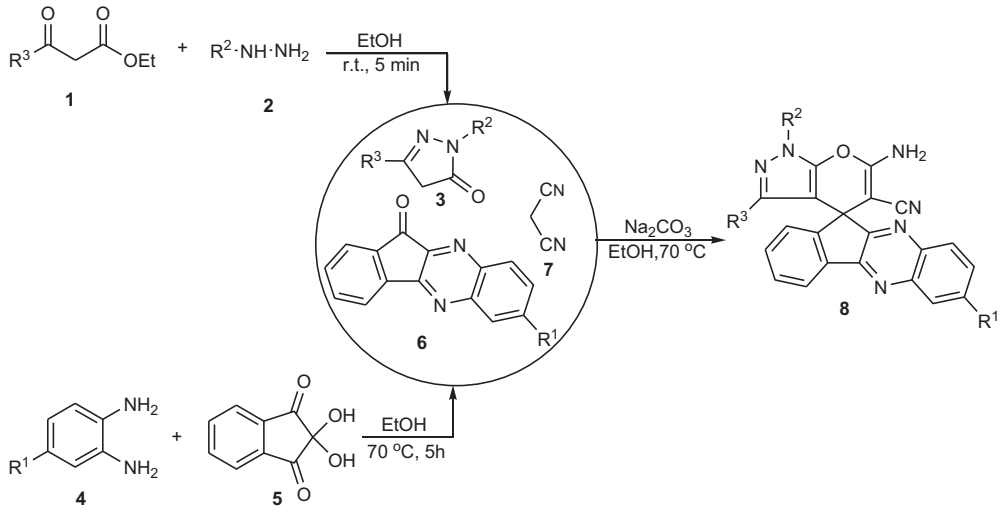
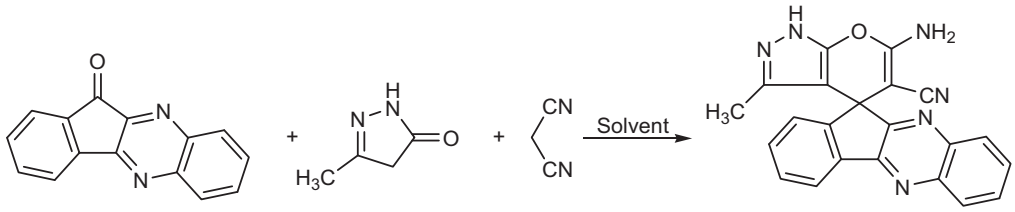


Scheme 1. Synthesis of spiro indeno[1,2-*b*]quinoxalines.

Table 1
Optimization of the reaction.



Entry	Solvent	Temperature (°C)	Yield (%)
1	Water	70	85
2	Ethanol	70	93
3	Methanol	25	90
4	Ethyl acetate	70	75
5	Acetonitrile	70	92
6	Toluene	70	Trace
7	Dichloromethane	25	Trace
8	Water/ethanol (25%)	70	83
9	Water/ethanol (50%)	70	80
10	Water/ethanol (75%)	70	75
11	Ethanol	25	80
12	Ethanol	50	85
13	Ethanol	100	93

means of a reaction between 1,2-phenylenediamines **4** and ninhydrine **5** (Scheme 1).

As a model reaction, we first investigated the condensation of 11*H*-indeno[1,2-*b*]quinoxalin-11-one **6**, 3-methyl-1*H*-pyrazol-5-ol **3** and malononitrile **7** under various conditions (Table 1). We first investigated the model reaction rate in different solvents by measuring the isolated yield using identical amounts of reactants in the presence of 10% mol of Na_2CO_3 for a fixed reaction time of 12 h at 70 °C (Table 1, entries 1–7). The desired product was obtained in polar solvents, such as water, ethanol, and methanol, ethyl acetate, and acetonitrile, but ethanol can afford the product in good yield even better than other solvents (Table 1, entry 2). The desired product was not obtained in non-polar solvents, such as dichloromethane, toluene (Table 1, entries 6–7). This effect can be explained by a simple acid-catalysis mechanism facilitated by the strong hydrogen bond interaction at the organic–ethanol interface, which stabilizes the reaction intermediate. Also, it was found that the addition of water to the ethanol solution cannot improve the reaction outcome (entry 2 and entries 8–10) and that, interestingly, the corresponding product was obtained in high yield when the reaction was performed in pure ethanol (entry 2).

Next, we studied the model reaction in ethanol at different temperatures (entry 2 and entries 11–13). The reaction rate increased as the temperature was raised. At 70 °C, the maximum yield (93%) was obtained in a reaction time of 12 h (entry 2).

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