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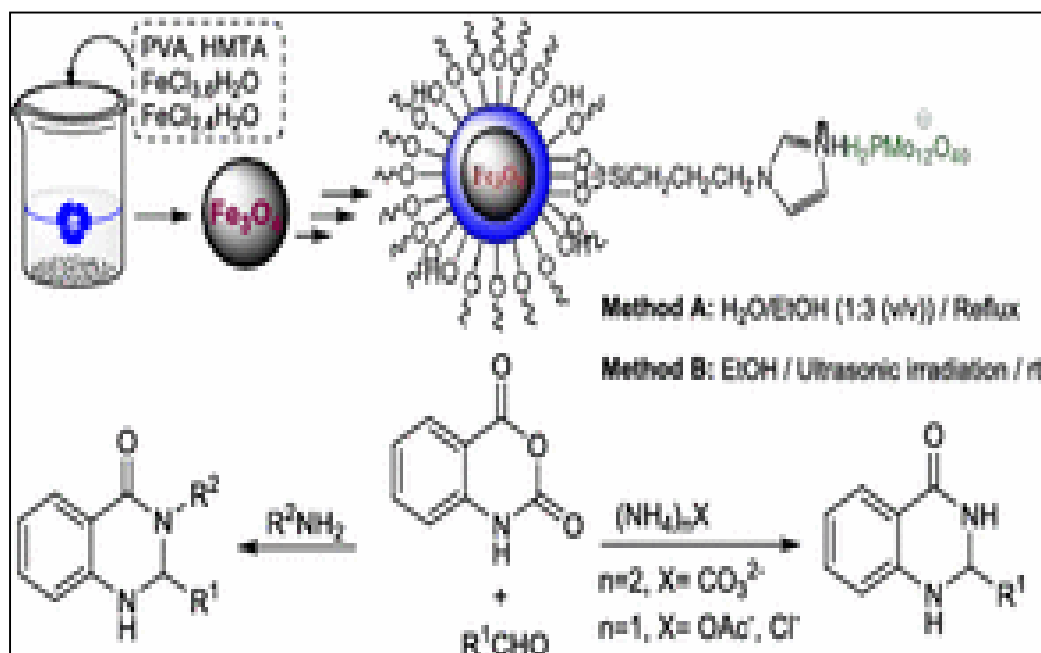
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A SIMPLE AND CONVENIENT SYNTHESIS OF 2, 3-DIHYDROQUINAZOLIN-4(1H)-ONES USING $\text{SiO}_2\text{-HNO}_3$



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ABSTRACT

An efficient synthesis of 2, 3-dihydroquinazolin-4(1H)-ones using $\text{SiO}_2\text{-HNO}_3$ as a catalyst. It was prepared via condensation of 2-aminobenzamide with various types of aldehydes. The reaction proceeded in short period of time with excellent yields.

KEYWORDS: 2, 3-dihydroquinazolin-4(1H)-ones, Aldehydes, $\text{SiO}_2\text{-HNO}_3$, 2-aminobenzamide.

INTRODUCTION

Multi-component Reactions (MCRs) are powerful tools for build the products in organic chemistry. In which more than two starting materials react to form a product for their high degree of atom economy, save time, minimize cost, environmental friendliness and application in the diversity-oriented of convergent synthesis of complex organic molecules from simple and readily

available substrates in a single synthetic operation [1-2]. 2,3-Dihydroquinazolin-4(1H)-ones are an important class of fused heterocyclic compounds that have drawn much attention because of their variety of biological and pharmaceutical activities [3-6]. Recently, number of organic reactions is reported in the literature by employing various catalysts like clay [7-9], phosphates [10-12] etc.

However, many of these methods suffer from one or more of the drawbacks such as requirement of strong acidic conditions, long reaction times, low yields, tedious work-up procedures, requirement of excess amounts of reagent and use of toxic reagents, catalysts or solvents. Therefore, there is a strong demand for a highly efficient and environmentally benign method.

Nitric acid supported on a silica gel (SiO₂-HNO₃) catalyst is an inexpensive and easily available oxidizing reagent that possesses high efficiency in organic transformations [13, 14]. Today, there is a great demand for green and inexpensive acids instead of conventional mineral acids such as HF, H₂SO₄ and HCl in chemical processes.

EXPERIMENTAL

All commercially available reagents used without further purification and the reactions were monitored by thin layer chromatography (TLC) Merck 60 F₂₅₀ silica-gel plates. All yields refer to isolated products after purification. Melting points were recorded by open tube capillary method and are uncorrected.

PREPARATION OF CATALYST

In a conical flask, 10 ml conc. HNO₃ was added to 5 g silica gel (Acme's; 100–200 mesh size) and stirred for 10 min. filtering produced a white solid which was kept in an oven for 10 min at 120 °C to activate the catalyst. Acidity of reagents the neutralization titration gave us the acidity of 0.080352 g (NaOH)/1 g SiO₂-HNO₃.

General procedure for the synthesis of 2, 3- dihydroquinazolin-4(1H)-ones:

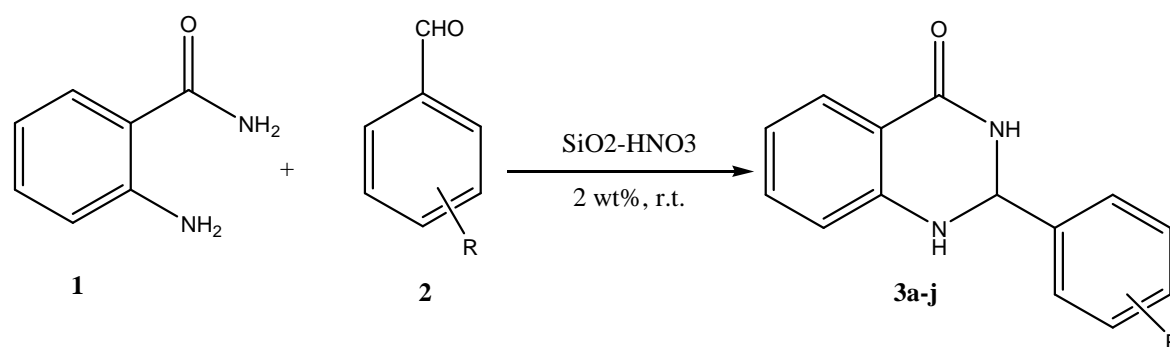
To a mixture of 2- aminobenzamide **1** (1 mmol) and aldehyde **2** (1 mmol) taken in round bottom flask was added SiO₂-HNO₃ (2 wt %) and the reaction mixture was stirred at room temperature. Instantly, an exothermic reaction occurred with completion of the reaction (TLC) and the reaction mixture became a yellowish orange solid. After completion of reaction, the solid residue was washed with ethanol. The obtained solid was collected by filtration and purified by recrystallization from ethanol.

The spectral data of compounds are given below:

2-Phenyl-2,3-dihydroquinazolin-4(1H)-one(3a):

Yellow solid m.p. 223–224 °C; IR (KBr, m, cm⁻¹): 3304, 3188, 1668, 1652, 1507, 1487, 1455, 1393, 1300, 1149, 748, 643; ¹H-NMR (400 MHz, DMSO-d₆) (d, ppm): 5.76 (1H, s), 6.68 (1H, t), 6.75 (1H, d), 7.14 (1H, s, NH), 7.25 (1H, t), 7.35–7.42 (3H, m, ArH), 7.50 (2H, d), 7.62 (1H, dd), 8.32 (1H, s, NH);

All products are known compounds; their physical and spectroscopic data (IR and ¹HNMR) were compared with those reported in the literature and found to be identical.



RESULT AND DISCUSSION

To explore the use of SiO₂-HNO₃ as catalysts, a reaction of 2-aminobenzamide **1** and aldehyde **2** was taken as a standard model reaction for the preparation of 2, 3-dihydroquinazolin-4(1H)-ones (**3a-3j**) (Scheme 1). In general, all the reactions were very clean and the 2,3-dihydroquinazolin-4(1H)-ones derivatives were obtained in high yields under room temperature.

Inspiring by this result, we examined the scope and limitations of this approach by applying the optimal reaction conditions to a number of aromatic aldehydes bearing electron-withdrawing and electron-donating substituents. We found that the property of substituent groups of the aromatic aldehydes does not affect these reactions. The results of the reaction are listed in **Table 1**.

Table 1 Multicomponent reaction of aromatic aldehyde **2** and 2-aminobenzamide **1** for the synthesis of **3a-j**^a

Entry	Aldehyde	Product	Time (min)	Yield ^b (%)
1	C ₆ H ₅	3a	5	82
2	4-CH ₃ OC ₆ H ₄	3b	6	81
3	4-BrC ₆ H ₄	3c	5	80
4	4-OHC ₆ H ₄	3d	6	79
5	4-NO ₂ C ₆ H ₄	3e	5	82
6	4-CH ₃ C ₆ H ₄	3f	4	84
7	2-ClC ₆ H ₄	3g	5	81
8	2-OHC ₆ H ₄	3h	6	79
9	3-NO ₂ C ₆ H ₄	3i	5	82
10	4-Cl-C ₆ H ₄	3j	4	84

^aReaction conditions: Aromatic aldehyde (1 mmol), 2-aminobenzamide (1 mmol), SiO₂-HNO₃ (2 wt %) at room temperature. ^bIsolated yield.

CONCLUSION

We have developed a new protocol for the synthesis of 2, 3-dihydroquinazolin-4(1H)-one derivatives via the one-pot multi-components reaction using SiO₂-HNO₃ under room temperature. High efficiency, easy availability, low cost, operational simplicity, and improved yields within short reaction times are the advantages of this new method.

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