

Efficient One-Pot Three-Component Mannich Reaction for the Synthesis of Barbituric Acid

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Abstract

An environmentally friendly organic synthesis of novel Barbituric acid using $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ under microwave irradiation herein described. The reaction time is very short with reasonable yield enhancement. Further the role of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ is studied in the reaction under microwave irradiation and concluded that microwave assisted $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ catalyzed reaction is the best in terms of catalysis as well as reaction yield improvement.

Keywords: Barbituric acid; Microwave heating; Mannich base

Introduction

Microwave-assisted Organic Reaction Enhancement (MORE) reactions are extremely fast, cleaner than conventional reactions and lead to higher atom economy (less chemical waste) [1-8]. Because of the short time requirement, ease of workability and environmentally friendliness, microwaves provide an alternative green approach to conventional methods. It can be termed as 'e-chemistry' because it is easy, effective, economical and eco-friendly and is believed to be the next step towards Green Chemistry for the attainment of sustainable development [4-22].

Over the past few decades, Mannich bases of heterocyclic molecules have been grabbing the attention of the synthetic organic chemists for their wide applications of biological activities. The Mannich reaction is widely used for the construction of a nitrogen-containing molecule. In this three component transformation, compounds possessing β -hydrogen atom, an aldehyde, and an amine react to form β -aminoketone derivative. Recently, the use of inorganic solid supports as catalysts has been developed for solvent-free reactions resulting in milder conditions and simple experimental procedures [4]. $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ catalyzed organic reactions are gaining importance due to their inexpensive nature and special catalytic attributes in heterogeneous reactions for the preparations of heterocyclic molecules. Several Mannich bases have diverse biological activities. In continuation of our research work [5-9], we herein developed a new synthetic procedure for the synthesis of Barbituric acid catalyzed by $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ [22]. The synthesized compounds have been characterized by TLC, Elemental analysis, IR and ¹H-NMR Spectroscopy.

Results and Discussion

Condensation of furfural, 2-aminopyridine with uracil in the presence of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ under microwave affording the desired product in reasonable yield (**FIG. 1**).

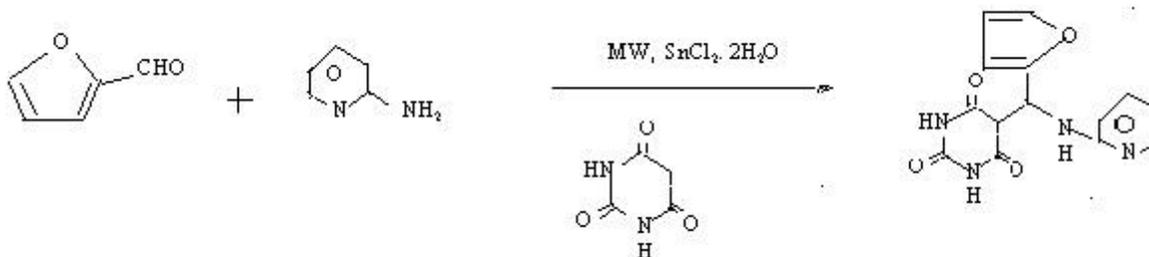


FIG. 1. Reaction for the synthesis of barbituric acid.

Experimental

Synthesis of barbituric acid

In the preparation of Barbituric acid, an acidic $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ was added to the equimolar (0.01 mole) mixture of furfural, 2-aminopyridine with uracil and the reaction mixture bath placed inside the microwave oven (Samsung model at 1050 W, 70% of total power) and irradiated for 5 minutes. Now, the progress of the reaction was carefully monitored through TLC. On completion of the reaction, the reaction mixture was allowed to cooled at room temperature and rendered basic (pH 8) with 10% NaHCO_3 and then extracted with ethyl acetate. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and evaporated to leave behind the crude product, which was further purified by column chromatography over silica gel (hexane: ethyl acetate 4:1). Satisfactory Elemental analysis, IR, NMR spectral data are obtained. Yield: 68%

Mp 290-292°C.

^1H NMR ($\text{CDCl}_3 + \text{DMSO}$) δ 8.1-8.5 (m, 5H, C-H-pyridine), 4.5-4.8 (d, 1H), 5.0 (brs, 1H, NH), 6.2-6.4 (m, 3H, C-H furan ring), 3.0 (d, 1H, C-H).

IR (KBr): 1030 (C=O), 1600 (C=C), 1650 (C=O), 2960 (C-H), 3050 (Py C-H), 3390 (N-H) cm^{-1} Anal. Calculated for $\text{C}_{14}\text{H}_{11}\text{N}_4\text{O}_4$: C 53.33, H 3.49, N 17.77. Found: C 53.35, H 3.48, N 17.79.

Conclusion

We have developed a facile, convenient and efficient synthetic procedure for the synthesis of Barbituric acid. The reported procedure clearly highlights the versatility of solid supports when coupled with microwaves. In conclusion, this condensation method offers an alternative route for the novel synthesis of Mannich reaction in reasonable yield making the process more simple and economic than any other conventional methods.

Conflicts of Interest

The authors declare no conflicts of interest.

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