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A RAPID AND FACILE SYNTHESIS OF 4,6-DIMETHYLPYRIMIDINE-2(1H)-ONE

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ABSTRACT

A rapid and efficient synthesis of 4,6-Dimethylpyrimidine-2(1H)-one has been carried out from acetylacetone and urea in dry media in the presence of an environmentally friendly non-corrosive, non-toxic and recyclable catalyst in 85% yield under microwave irradiation conditions.

KEYWORDS: Pyrimidine-2-one, Dry media, MW, Urea, Acetyl acetone

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INTRODUCTION

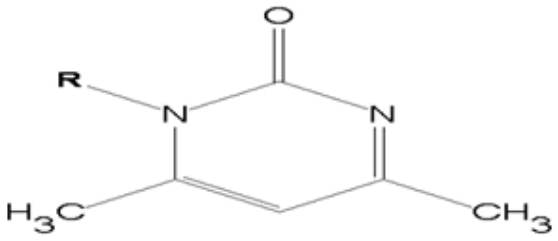
4,6-Dimethylpyrimidine-2(1H)-one is a medicinally important heterocyclic compound which is used as an efficient precursor for the synthesis of a number of heterocyclic compounds having attractive pharmacological profiles.[1] Also, the title compound is a main component of menadione pyrimidinol bisulfate (MPB) which is one of the most effective vitamin K active products for use in animal food stuffs. Furthermore,it also forms a component of Nicarbazine which is a coccodiostatic agent.[2] The title compound is generally prepared by the condensation of acetylacetone with urea under refluxing conditions in presence of conc. sulfuric acid. However, the method suffers from many limitations such as low yields, higher reaction times, use of toxic solvents as well as formation of side products. Besides the reaction mixture has to be neutralized before the product can be isolated. Hence, there was a need to develop a new method for the efficient and green synthesis of the title compound.

MATERIALS AND METHODS

The substrates were procured from Aldrich. Acetylacetone was distilled before use. The Proton NMR spectra were recorded at 400 MHz Bruker NMR spectrometer. The chemical shifts are reported in ppm and were measured in deuterated chloroform and TMS as an internal standard. TLC was used for monitoring the reaction.

RESULTS AND DISCUSSION

There is a great emphasis these days upon carrying out organic synthesis under environmentally benign reaction conditions such as under solvent-free conditions using heterogenous catalysts like Montmorillonote K-10 catalyst etc. Similarly, microwave irradiation has become very popular in organic synthesis because it lowers down the formation of the side products, thereby enhancing the product yield and consequently, the product purity and all that without the use of any organic solvent. Because of our interest in doing rapid synthesis under heterogenous catalysts, we have combined the two techniques to carry out the synthesis of the title compound.[3-6] The reaction was initiated by condensing urea with acetylacetone in 1:1 ratio in the presence of the environmentally friendly, non-corrosive, non-toxic, inexpensive and re-cyclable catalyst Mont K-10. The reaction mixture was microwaved at varying levels of microwave irradiation but 500 W was found to be optimum.



R= H

The reactions were monitored by thin layer chromatography. The product was obtained in an isolated yield of 85%. The PMR of the product exhibited the presence of three signals at 2.12(s), 5.52(s) and 9.67(br) in the relative ratio of 6:1:1 owing respectively to two methyl protons, the olefinic and N-H proton as anticipated. The infra red spectrum confirmed the structure of the compound. The melting

point was determined to be 191C that agreed with that reported in the literature. The beauty of the Mont K-10 catalyst used is that it could be recycled to give almost identical yield of the product.

CONCLUSION

We have developed a new and a green method for the synthesis of 4,6-Dimethylpyrimidine-2(1H)-one from acetylacetone and urea under microwave irradiation conditions using an environmentally friendly non-corrosive, non-toxic, and recyclable catalyst Mont-K-10 in dry media in 85% yield.

CONFLICT OF INTERST

Conflict of interest declared none.

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