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Green Synthesis of 1,3-dimethyl-6-phenylpyrimidine-2,4-dione

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ABSTRACT

1,3-Dimethyl-6-phenylpyrimidine-2,4-dione has been synthesized by condensing ethyl benzoyl acetate and 1,3-dimethyl urea under green chemistry conditions in 80% yield.

Keywords: Pyrimidinediones, uracils, MW, Dry media, Condensation.

INTRODUCTION

Pyrimidinediones have attracted immense interest in pharmaceutical sciences as well as organic synthesis as they exhibit attractive pharmacological profiles such as analgesic, antipyretic, anti-inflammatory and anticancer [1-2]. Additionally, they act as important intermediates for the synthesis of pyrimidine bases that also possess similar therapeutic profiles.

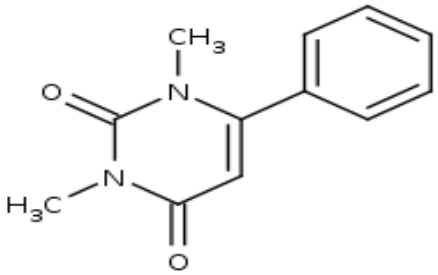
MATERIALS AND METHODS

Ethylbenzoylacetate and dimethylurea were from Aldrich. The proton nmr spectra were recorded on a 400MHz NMR Spectrometer at SAIF, Chandigarh. All chemical shifts are expressed in parts per million with respect to trimethylsilane(TMS) and in $CDCl_3$. The IR spectra were obtained on a FT Nicolet Instrument at SAIF, Chandigarh.

In a typical procedure, EBA(1mmol) and DMU(1mmol) were mixed together in a 25 mL Pyrex beaker placed in a Teflon bath and microwaved at 630 W for 10 min. The reaction was monitored by TLC using CCl_4: ethylacetate 3:1. The crude product was purified by column chromatography (CCl_4/ethylacetate, 94/6) as eluent over silica gel to afford the desired product in 80% yield, mp 92 $^{\circ}$C (ether).

RESULTS AND DISCUSSION

There is a growing interest in doing organic synthesis under green chemistry conditions which involves solvent-free conditions and the use of microwave irradiation [3-6].



Ethylbenzoylacetate(EBA) and 1,3-dimethylurea (DMU)were mixed together in 1:1 ratio and reacted under microwave irradiation at varying power levels under solvent- free conditions. The substrates were microwaved at 90 W and the progress of the reaction monitored by TLC which showed that the reaction occurred very slowly and did not occur to an appreciable extent even after 40 min of irradiation. Consequently, the power level was adjusted to 360 W, 720 W and 900 W but without any substantial change in the reaction progress. TLC monitoring led us to conclude that EBA underwent evaporation to a considerable extent under the open vessel conditions. However, even when the substrate ratio was altered to 2:1 from 1:1, no major change could be noticed in the reaction progress at 90 W, 360 W, 720 W and 900 W for the same reasons.

In view of the above results, we decided to attempt the reaction under closed vessel conditions. Subsequently, the reaction was carried out in a Teflon bath which was fitted with a security disk that could resist pressures up to 10 bars. When the substrates were mixed in 1:1 ratio and the mixture microwaved at 90 W, the reaction occurred slowly and did not progress well even after 40 min. Adjusting the power levels up to 540 W did not result in a sufficient progress. However, the reaction underwent completion within 10 min at 630 W giving the desired product in 76% isolated yield after column chromatography. The proton NMR of the product exhibited two 3H singlets at 3.22 and 3.40ppm owing to two N-methyl protons. The olefinic proton at C-5 appeared at 5.70ppm. The 5H aromatic protons absorbed at 7.27-7.50ppm, thus confirming the structure of the desired product. The ir spectrum showed the presence as expected of three peaks at 1702.8,1655.8 and 1619.5 cm due respectively to CO, conjugated CO and C=C str. The elemental analysis was also in agreement. Attempts were now made to enhance the yield of the product by adjusting the EBA:DMU ratio from 1:1 to 1.5:1,2:1 ,1:1.5 and 1:2 with power level remaining the same at 630 W but the yield almost remained constant.

APPLICATIONS

The pyrimidinediones show various therapeutic profiles like antipyretic, analgesic, anti-inflammatory,anti-cancer and antiviral etc. Some have been reported to exhibit pharmacological profiles such as hyperthyroidism, cardiovascular and antihypertensive [1-2].

CONCLUSIONS

In conclusion, we have developed a new, facile and one-pot synthetic route for the synthesis of 1,3-dimethylphenylpyrimidine-2,4-dione from ethylbenzoylacetate and 1,3-dimethylurea under green chemistry conditions.

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