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Rapid Synthesis of 1, 3-Dioxolanes of Aldehydes And Ketones Using Zinc Oxide

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ABSTRACT

An efficient synthesis of 1,3-dioxolanes of aldehydes and ketones has been carried out in high yields in the presence of zinc oxide in dry media under microwave irradiation.

Keywords: 1,3-dioxolanes, aldehydes, ketones, zinc oxide, MW.

INTRODUCTION

The acetalization of aldehydes and ketones with ethylene glycol as 1,3-dioxolanes is a protocol of great significance in organic synthesis.[1-5]This process serves as one of the most useful methods for the protection of aldehydes and ketones against addition by nucleophiles.

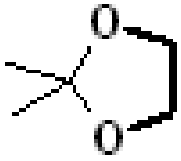
MATERIALS AND METHODS

In a typical procedure, benzaldehyde (5 mmol) was taken in an Erlenmeyer flask (25mL) and ethylene glycol (50 mmol) was added followed by the addition of zinc oxide (100 mg) and the reaction mixture microwaved at different power levels but 200 W was found to be the optimum level. The reaction mixture was transferred to water (50mL). The organic product was extracted with benzene (2x25mL) and dried over sodium sulfate anhydrous. Next, the solvent was distilled off, giving the desired 1,3-dioxolane. The products were identified on the basis of their spectral analysis as well as by comparison of their R_f values with those of authentic samples prepared by standard routes[4].

RESULTS AND DISCUSSION

1,3-dioxolanes are synthesized by using catalysts such as toluene-p-sulfonic acid (TsOH), Me_3SiCl,TBAF etc.[1-2] However, these generally require longer reaction times (1-16 hours), azeotropic removal of the water formed in the reaction and tedious work-up. Furthermore, while aldehydes, in general, afford the dioxolanes in good yields, ketones in particular, cyclic ketones either do not form at all or give very poor yields of the corresponding dioxolanes. Hence, there is a need for developing methods for this important reaction of great synthetic value.

We have recently reported the dioxolane formation in the presence of silica gel Mont KSF,Mg SO_4(anhyd.) and Na_2SO_4 (anhyd.). [4-5] Because of our interest in developing new rapid methodologies for organic reactions [6-9], we now report herein the synthesis of the title compounds using zinc oxide .



A number of aldehydes and ketones were converted into their dioxolanes. The yields of the products obtained are shown in table.

Table1. Synthesis of 1,3-dioxolanes of carbonyl compounds at 200 W using ZnO

Sr No	Reactants	Yields
1	Benzaldehyde	92%
2	2,5-Dimethoxy benzaldehyde	93%
3	Cinnamaldehyde	90%
4	Benzophenone	60%
5	Acetophenone	86%
6	4-Hydroxy benzaldehyde	85%
7	4-Methoxy benzaldehyde	83%
8	Furan-2-aldehyde	90%

As can be seen from the table, the products from arylaldehydes were obtained in 83-93% yield. The ketones gave moderate yields.

APPLICATIONS

1,3-dioxolanes of a number of heterocyclic compounds have exhibited fascinative pharmacotherapeutic profiles such as anti-fungal, anti-bacterial, anti-neoplastic, anti-viral, anesthetic, anticonvulsants etc [1-2].

CONCLUSIONS

We have shown that the 1,3-dioxolanes of aryl aldehydes can be rapidly obtained under solvent-free conditions in presence of zinc oxide under microwave irradiation.

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