

**NOTE**

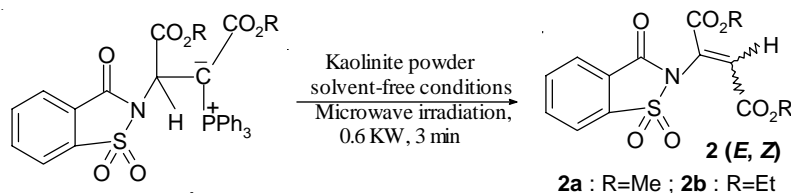
**Microwave Induced Stereoselective Conversion of  
Dialkyl-2-(1,1,3-trioxo-1,3-dihydro-2H-1,2-benzisothiazol-2-yl)-  
3-(triphenylphosphoranylidene)succinates to Dialkyl-2-(1,1,3-  
trioxo-1,3-dihydro-2H-1,2-benzisothiazol-2-yl)-2-butendioates**

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Kaolinite powder was found to catalyze conversion of stabilized phosphorus ylides to corresponding electron-poor *N*-vinylated isothiazoles in solvent-free conditions under microwave irradiation at microwave power 0.6 KW in 3 min in fairly good yields.

**Key Words:** Microwave irradiation, Catalyst, Kaolinite, Saccharin, Phosphorus ylide, Solvent-free conditions.

Silica gel as an additive promotes the Wittig reactions<sup>1,2</sup> of phosphorus ylides with aldehydes, including sterically hindered aldehydes to increase the rate and yields of alkenes<sup>3,4</sup>. Earlier, a one-pot method for preparing stabilized phosphorus ylides utilizing *in situ* generation of the phosphonium salts is reported<sup>5</sup>. In this paper, the catalytic role of kaolinite powder in the stereoselective conversion of dialkyl-2-(1,1,3-trioxo-1,3-dihydro-2H-1,2-benzisothiazol-2-yl)-3-(triphenylphosphoranylidene)succinates (**1**) to dialkyl-2-(1,1,3-trioxo-1,3-dihydro-2H-1,2-benzisothiazol-2-yl)-2-butendioates (**2**) in solvent-free conditions under microwave irradiation at microwave power 0.6 KW in 3 min in fairly good yields is reported (**Scheme-1**).



**Scheme-1**

Commercial oven Butane M245 was used for microwave irradiation. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu IR-460

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spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured with a Bruker DRX-500 Avance spectrometer at 500 and 125 MHz, respectively.

**General procedure for the preparation of compounds 6a-b:** Mixture of Kaolinite powder (2 g) and the ylide **1** (1 mmol) were heated under microwave irradiation at microwave power 0.6 KW in 3 min and then placed over a column of silica gel (10 g). The column chromatography was washed using ethyl acetate-light petroleum ether (1:9) as eluent. The solvent was removed under reduced pressure and products (**2a-b**) were obtained (**2a**: White crystals, m.p. 130-137°C; Yield: 46%. *Z*-isomer: 83%, *E*-isomer: 17 %; **2b**: Viscous colourless oil; Yield: 42%. *Z*-isomer: 66%, *E*-isomer: 34%). The characterization data of the compounds (**6a-b**) are given in our previous reports<sup>7</sup>.

Kaolinite powder was found to catalyze stereoselective conversion of dialkyl-2-(1,1,3-trioxo-1,3-dihydro-2*H*-1,2-benzisothiazol-2-yl)-3-(triphenylphosphoranylidene)succinates (**1**) to dialkyl-2-(1,1,3-trioxo-1,3-dihydro-2*H*-1,2-benzisothiazol-2-yl)-2-butendioates (**2**) in solvent-free conditions under microwave irradiation at microwave power 0.6 KW in 3 min in fairly good yields (**Scheme-1**). Several catalysts have also been used *e.g.*,  $\text{K}_2\text{HPO}_4$ ,  $\text{KH}_2\text{PO}_4$ ,  $\text{MgO}$ ,  $\text{MnSO}_4$ ,  $\text{KHSO}_4$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{NaHCO}_3$  and  $\text{MgSO}_4$ , but yields of corresponding products **2** in cases  $\text{MgO}$ ,  $\text{K}_2\text{HPO}_4$  and  $\text{KH}_2\text{PO}_4$  were low and in the others cases no product were observed. The structures **2a-b** were elucidated from their IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra.

In short, the kaolinite powder is found to catalyze stereoselective conversion of ylides **1** to compounds **2** in solvent-free conditions under microwave irradiation at microwave power 0.6 KW in 3 min in fairly good yields. Other aspects of this process are under investigation.

#### ACKNOWLEDGEMENT

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