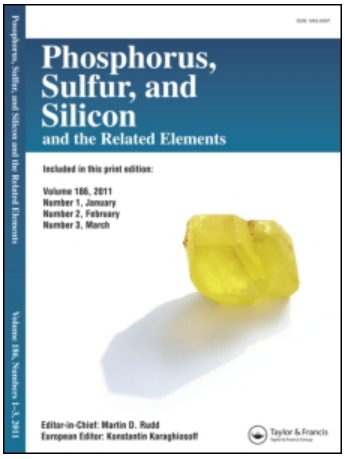

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*Magnesium hydrogen sulfate powder was found to catalyze stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylphosphoranylidene)butanedioates to electron-poor (Z)-N-vinylimides in solvent-free conditions at 95°C 1 h in high conversions. Microwave also was found to catalyze the same reactions in the presence of magnesium hydrogen sulfate powder in solvent-free conditions in 3 min.*

Keywords: Magnesium hydrogen sulfate; microwave irradiation; phosphorus ylide; solvent-free conditions; (*Z*)-*N*-vinylimide

Organophosphorus compounds have been extensively used in organic synthesis as useful reagents.[1] β-Additions of nucleophiles to the vinyl group of vinylic phosphonium salts leading to the formation of new alkylidenephosphoranes has attracted much attention as a very convenient and synthetically useful method in organic synthesis.[1–11] In the past we have established a convenient, one-pot method for preparing stabilized phosphorus ylides utilizing in situ generation of the phosphonium salts.[1–11] In this article, we report on the catalytic rule of magnesium hydrogen sulfate powder in the stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylphosphoranylidene)butanedioates (**1**)[11] to electron-poor (*Z*)-*N*-vinylimides (**2**)[10] in solvent-free conditions[13] at 95°C (also under microwave heating) with fairly high conversions (Scheme 1).

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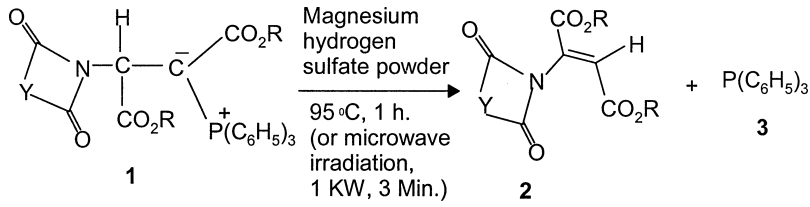

2	a	b	c	d
R	Me	Et	Me	Et
Y	$(CH_2)_2$	$(CH_2)_2$	*o*-C_6H_4	*o*-C_6H_4

SCHEME 1

RESULTS AND DISCUSSION

Magnesium hydrogen sulfate was found to catalyze stereoselective conversion of ylides **1**[11] to electron-poor (*Z*)-*N*-vinylimides (**2**)[10] in solvent-free conditions[13] at 95°C with fairly good conversions (Scheme 1).[10–11] TLC indicated that the reaction was completed after 1 h. The reaction proceeds smoothly and cleanly under solvent-free conditions[15] at 95°C and no side reactions were observed. In the absence of magnesium hydrogen sulfate powder, this reaction did not afford the corresponding compounds (**2a**) even at reflux temperature (toluene as solvent) after 24 h. TLC indicated that the solution contained unreacted ylide **1a**.[11] Microwave irradiation (1 KW, 3 min) also was found to catalyze the same reactions in the presence of magnesium hydrogen sulfate powder in solvent-free conditions with high conversions (Scheme 1). The structures **2a–d** were deduced from their ^{1}H NMR and ^{13}C NMR spectra and also via x-ray single crystal (for **2c**) structure determination.[12]

In summary, we have found that magnesium hydrogen sulfate is able to catalyze stereoselective conversion of ylides **1**[11] to compounds **2**[10] in solvent-free conditions under normal and microwave heating. Other aspects of this process are under investigation.

EXPERIMENTAL

Commerical oven Butane M245 was used for microwave irradiation. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. ^{1}H and ^{13}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 2a–d

The powdered mixture of dry magnesium hydrogen sulfate (2 g) and ylide **1**[11] (1 mmol) were heated for 1 h at 95°C (or were irradiated in the microwave oven at a microwave power of 1 KW (100%) for 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–d**) were obtained. The characterization data of the compounds (**2a–d**) are given in our previous report.[10]

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