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NOTE

Microwave Induced Stereoselective Conversion of Dialkyl 2-(Imido-*N*-yl)-3-(Triphenylphosphoranylidene)butanedioates to Electron-poor (Z)-*N*-Vinylimides in the Presence of Silica Gel Powder in Solvent-free Conditions

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Silica gel 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 under microwave irradiation.

Key Words: Microwave, Silica gel, (Z)-*N*-Vinylimide, Phosphorus ylide, Solvent-free conditions.

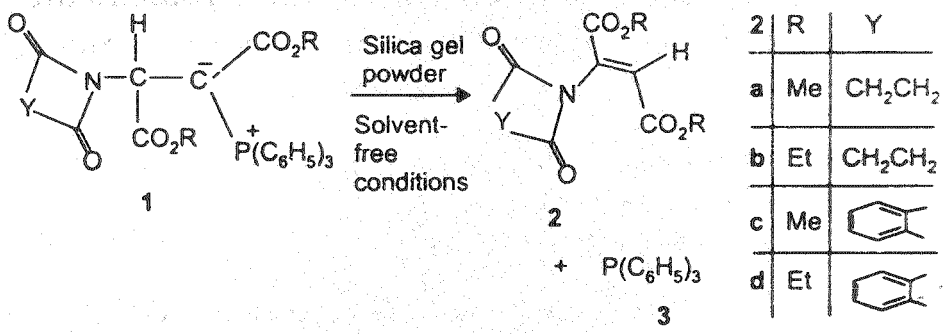
β-Additions of nucleophiles to the vinyl group of vinylic phosphonium salts leading to the formation of new alkylidenephosphoranes has attracted much attention as a very convenient and synthetically useful method in organic synthesis[1–8]. Organophosphorus compounds have been extensively used in organic synthesis as useful reagents as well as ligands of a number of transition metal catalysts[2]. Silica gel as an additive promotes the Wittig reactions of phosphorus ylides with aldehydes, including sterically hindered aldehydes to increase the rate and yields of alkenes[9, 10]. In this article, the catalytic rule of silica gel powder in the stereoselective conversion of dialkyl 2-(imido-N-yl)-3-(triphenylphosphoranylidene)-butanedioates (**1**)[10] to electron-poor (Z)-N-vinylimides (**2**)[8] in solvent-free conditions[14] under microwave irradiation with high coversions **(Scheme-1)** has been reported.

Commercial oven Butane M245 was used for microwave irradiation. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. ^1H and ^{13}C NMR spectra were measured with a Bruker DRX-500 AVANCE spectrometer at 500 and 125 MHz respectively.

Preparation of Compounds 2a–d: The homogeneous mixture of powdered dry silica gel (2 g) and ylide **1**[10] (1 mmol) were irradiated in a microwave oven at microwave power 0.1–0.5 kW for 3 min (**Scheme-1**) and then placed over a column of silica gel (12 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 already reported earlier[8, 9].

Silica gel powder was found to catalyze stereoselective conversion of ylides (**1**)[10] to electron-poor (Z)-*N*-vinylimides (**2**)[8] in solvent-free conditions[14] under microwave irradiation at microwave power 0.1–0.5 kW with high coversions (**Scheme-1**)[8–12]. TLC indicated that the reaction was completed after 3 min. The

reaction proceeds smoothly and cleanly under solvent-free conditions[14] at microwave power 0.1–0.5 kW (in all cases the reaction works efficiently with high conversions) and no side reactions were observed.



2	R	Y
a	Me	CH_2CH_2
b	Et	CH_2CH_2
c	Me	
d	Et	

2	a	b	c	d
Thermal conditions T(°C), t(Min), %Yield	95, 45, 47	95, 60, 45	95, 60, 70	110, 120, 37
Microwave conditions microwave power (KW), t(Min), %Yield	0.3, 3, 52	0.4, 3, 49	0.1, 3, 68,	0.5, 3, 40

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[13].

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