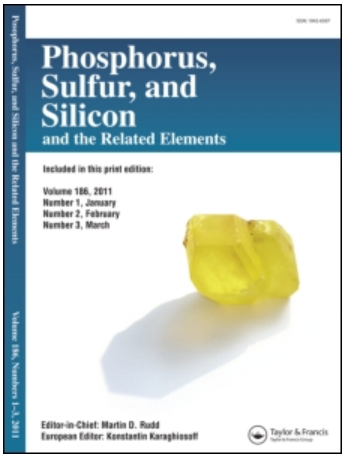

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

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Online publication date: 16 August 2010

To cite this Article Ramazani, Ali and Ahmadi, Ebrahim(2004) 'MICROWAVE INDUCED STEREOSELECTIVE CONVERSION OF DIALKYL 2-(IMIDO-*N*-YL)-3- (TRIPHENYLPHOSPHORANYLIDENE)BUTANEDIOATES TO ELECTRON-POOR (*Z*)-*N*-VINYLIMIDES IN THE PRESENCE OF IRON(II) SULFATE POWDER IN SOLVENT-FREE CONDITIONS', Phosphorus, Sulfur, and Silicon and the Related Elements, 179: 7, 1455 — 1457
To link to this Article: DOI: 10.1080/10426500490463673
URL: http://dx.doi.org/10.1080/10426500490463673

Phosphorus, Sulfur, and Silicon, 179:1455–1457, 2004

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500490463673

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(Received November 6, 2003; in final form November 25, 2003)

*Microwave was found to catalyze stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylphosphoranylidene)butanedioates to electron-poor* (Z)-N-*vinylimides in the presence of iron(II) sulfate powder in solvent-free conditions in 4 min.*

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

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.[1–10] Organophosphorus compounds have been extensively used in organic synthesis as useful reagents.[1] In the past we have established a convenient, one-pot method for preparing stabilized phosphorus ylides utilizing in situ generation of the phosphonium salts.[1–10] In this article, we report on the catalytic rule of iron(II) sulfate powder in the stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylphosphoranylidene)butanedioates (**1**)[10] to electron-poor (*Z*)-*N*-vinylimides (**2**)[9] in solvent-free conditions[12] under microwave heating with fairly high conversions (Scheme 1).

This work was supported by the Zanjan University.

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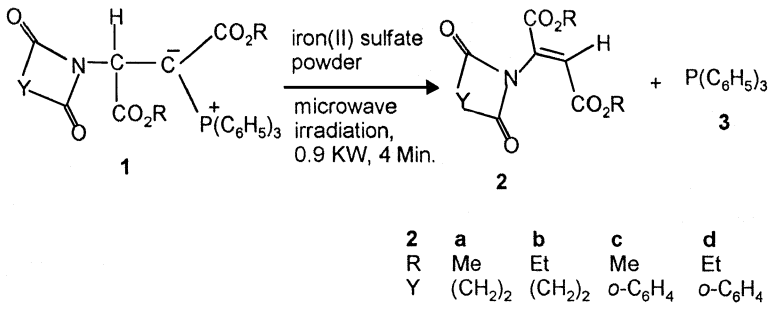

SCHEME 1

RESULTS AND DISCUSSION

Iron(II) sulfate powder was found to catalyze stereoselective conversion of ylides **1**[10] to electron-poor (*Z*)-*N*-vinylimides (**2**)[9] in solvent-free conditions[12] under microwave irradiation with fairly good conversions (Scheme 1).[9–10] TLC indicated that the reaction was completed after 4 min at microwave power 0.9 KW. The reaction proceeds smoothly and cleanly under solvent-free conditions[12] and no side reactions were observed. In the absence of iron(II) 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**.[10] This reactions were not completed under thermal conditions in the presence of iron(II) sulfate powder in solvent-free conditions (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.[11]

In summary, we have found that iron(II) sulfate powder is able to catalyze stereoselective conversion of ylides **1**[10] to compounds **2**[9] in solvent-free conditions under 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 iron(II) sulfate powder (2 g) and ylide **1**[10] (1 mmol) were irradiated in the microwave oven at microwave power 0.9 KW (90%) for 4 min. Purification conditions and the characterization data of the compounds (**2a–d**) are given in our previous report.[9]

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