Asian Journal of Chemistry Vol. 18, No. 1 (2006), 125–128

Microwave Induced Stereoselective Conversion of Dialkyl 2-(imido-*N*-yl)-3-(triphenylstibanylidene)succinates to Electron-poor (Z)-*N*-vinylimides

ALI RAMAZANI*, EBRAHIM AHMADI† and NADER NOSHIRANZADEH
Chemistry Department, Zanjan University, P.O. Box 45195-313, Zanjan, Iran
E-mail: a-ramazani@mail.znu.ac.ir, aliramazani@yahoo.com

Protonation of the highly reactive 1 : 1 intermediates, produced in the reaction between triphenylstibine and dialkyl acetylenedicarboxylates, by imides (phthalimide and succinimide) leads to vinyltriphenylstibinium salts, which undergo Michael addition reaction with conjugate base to produce dialkyl 2-(imido-*N*-yl)-3-(triphenylstibanylidene)succinates. Microwave irradiation was found to catalyze stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylstibanylidene)succinates to electron-poor (Z)-*N*-vinylimides in the presence of silica gel in solvent-free conditions at microwave power 0.3 kW in 3 min in high conversions.

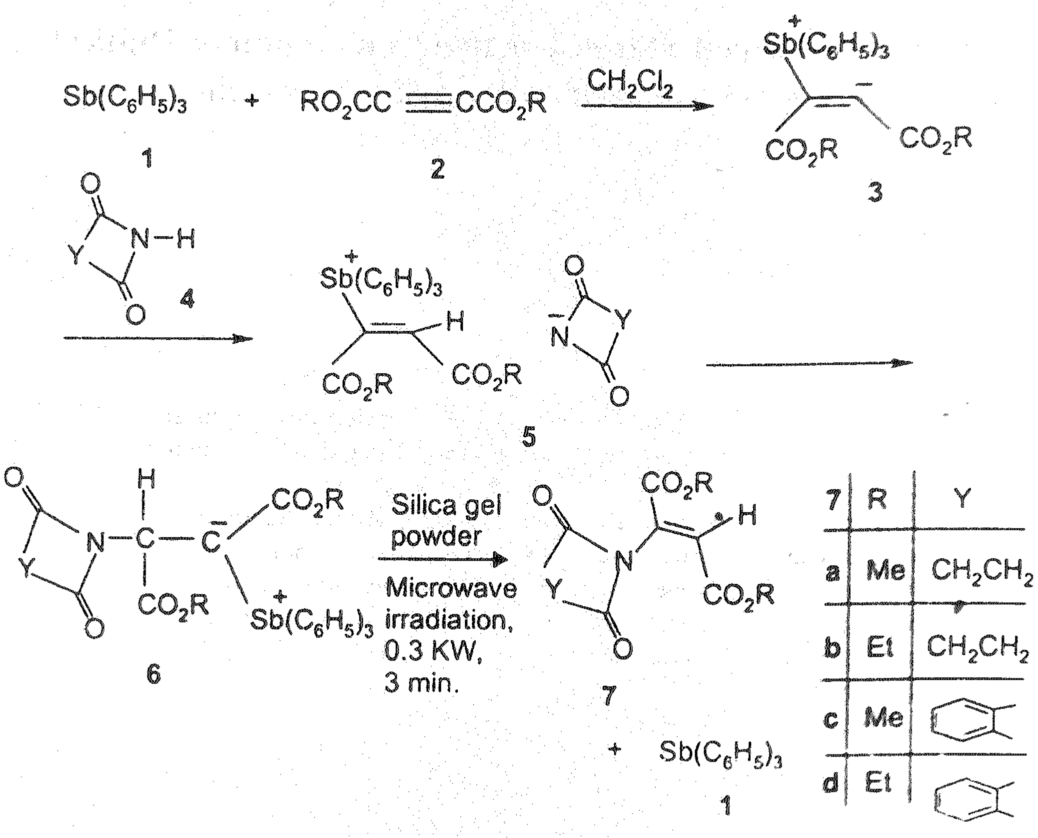
Key Words: Microwave irradiation, Stibine ylide, Silica gel, (Z)-*N*-Vinylimide.

INTRODUCTION

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]. 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[3, 4]. In the past, a convenient one-pot method for preparing stabilized phosphorus ylides utilizing *in situ* generation of the phosphonium salts[1] was established. In this article, the catalytic role of microwave irradiation in the stereoselective conversion of dialkyl 2-(imido-*N*-yl)-3-(triphenylstibanylidene)succinates (6)[5] to electron-poor (Z)-*N*-vinylimides (7)[6] in the presence of silica gel in solvent-free conditions[7] at microwave power 0.3 kW in 3 min with high conversions is reported **(Scheme-1)**.

---

†Chemistry Department, Zanjan University, P.O. Box 45195-313, Zanjan, Iran



Scheme-1

EXPERIMENTAL

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.

General Procedure for the Preparation of Compounds 7a–d

To a magnetically stirred solution of triphenylstibine **1** (1 mmol) and imide **4** (1 mmol) in CH_2Cl_2 (5 mL) was added dropwise a mixture of **2** (1 mmol) in CH_2Cl_2 (3 mL) at –10°C over 15 min. The mixture was allowed to warm up to room temperature. Silica gel powder (1 g) was added and the solvent was evaporated. Dry silica gel and the residue were irradiated under microwave (at microwave power 0.3 kW in 3 min.) and then placed over a column of silica gel (5 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 (**7a–d**) were obtained. The characterization data of the compounds (**7a–d**) are already reported[6].

RESULTS AND DISCUSSION

Protonation of the highly reactive 1 : 1 intermediates (**3**), produced in the reaction between triphenylstibine (**1**) and dialkyl acetylenedicarboxylates (**2**), by imides (**4**) (phthalimide and succinimide) leads to vinyltriphenylstibinium salts (**5**), which undergo Michael addition reaction with conjugate base to produce dialkyl 2-(imido-N-yl)-3-(triphenylstibanylidene)succinates (**6**). Microwave irradiation was found to catalyze stereoselective conversion of ylides (**6**)[5] to electron-poor (Z)-*N*-vinylimides (**7**)[6] in the presence of silica gel powder in solvent-free conditions[7] at microwave power 0.3 kW in 3 min with high conversions (**Scheme-1**)[3–6]. TLC indicated that the reaction was completed after 3 min. The reaction proceeds smoothly and cleanly under solvent-free conditions[7] at microwave power 0.3 kW in 3 min (in all cases the reaction works efficiently with high conversions) and no side reactions were observed. The structures **7a–d** were deduced from their ^{1}H NMR and ^{13}C NMR spectra and also *via* X-ray single crystal (for **7c**) structure determination (Fig. 1 and Fig. 2)[8].

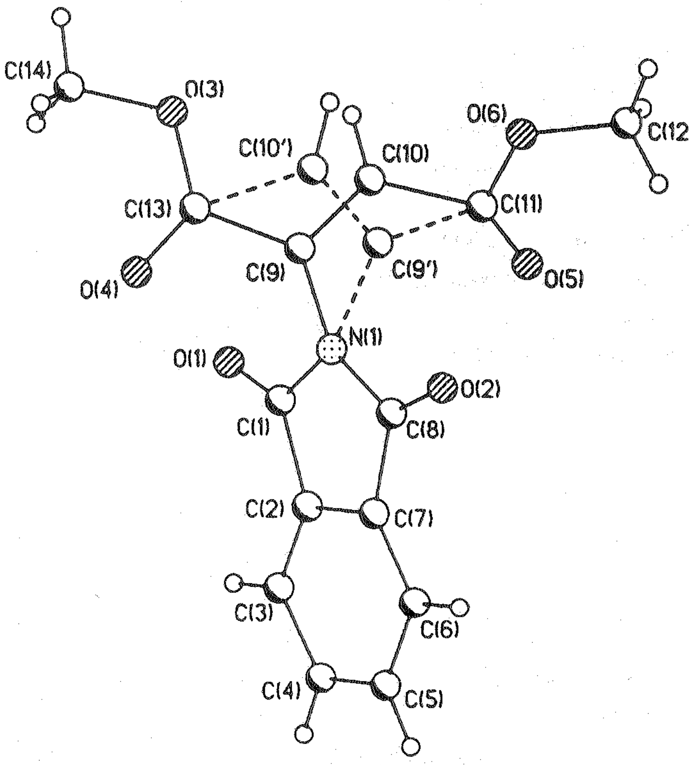


Fig. 1. Molecular structure of 7c

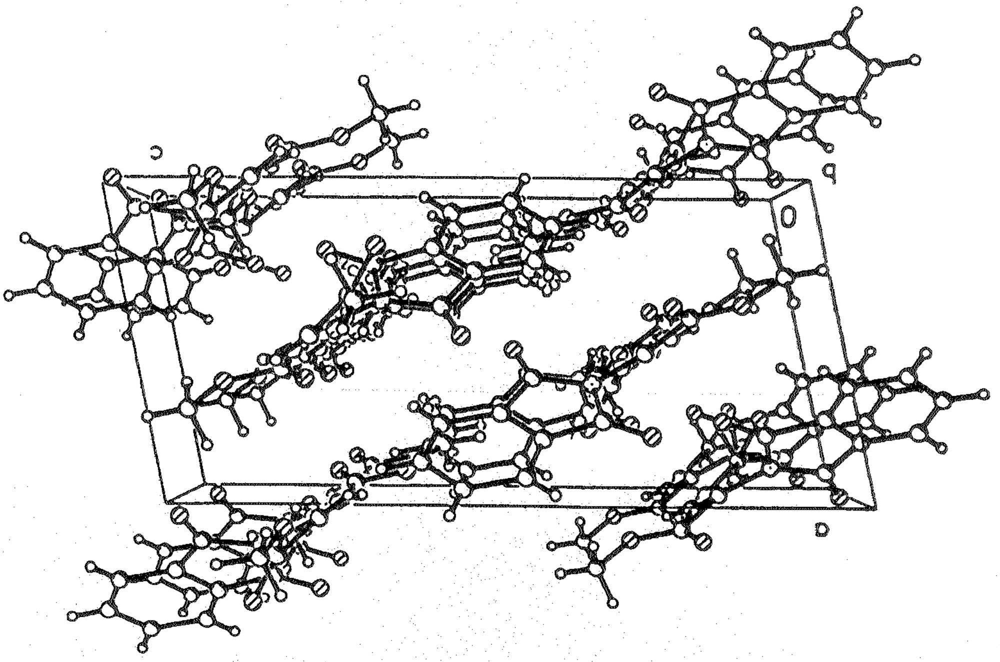


Fig. 2. Unit cell crystal structure of 7c

ACKNOWLEDGEMENT

This work was supported by the Zanjan University.

REFERENCES

1. A. Ramazani, L. Yousefi, E. Ahmadi and A. Souldozi, *Phosphorus, Sulfur and Silicon*, **179**, 1459 (2004) and references cited therein.
2. I. Yavari and A. Ramazani, *Synth. Commun.*, **27**, 1449 (1997) and references cited therein.
3. V.J. Patil and U. Mavers, *Tetrahedron Lett.*, **37**, 1281 (1996).
4. C. Xu, G. Chen, C. Fu and X. Huang, *Synth. Commun.*, **25**, 2229 (1995).
5. A. Ramazani and H. Ahani, *Phosphorus, Sulfur and Silicon*, **170**, 181 (2001).
6. A. Ramazani, N. Noshiranzadeh and B. Mohammadi, *Phosphorus, Sulfur and Silicon*, **178**, 539 (2003).
7. K. Tanaka and F. Toda, *Chem. Rev.*, **100**, 1025 (2000).
8. A. Ramazani, F. Marandi, E. Ahmadi and A. Morsali, *Z. Kristallogr., NCS*, **219**, 179 (2004).

(*Received*: 23 October 2004; *Accepted:* 5 September 2005) AJC-4373