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NOTE

Sodium Hydrogen Carbonate Powder Catalyzed Intermolecular Wittig Reaction of Fluorin-Containing Stabilized Phosphorus Ylides with Ninhydrin

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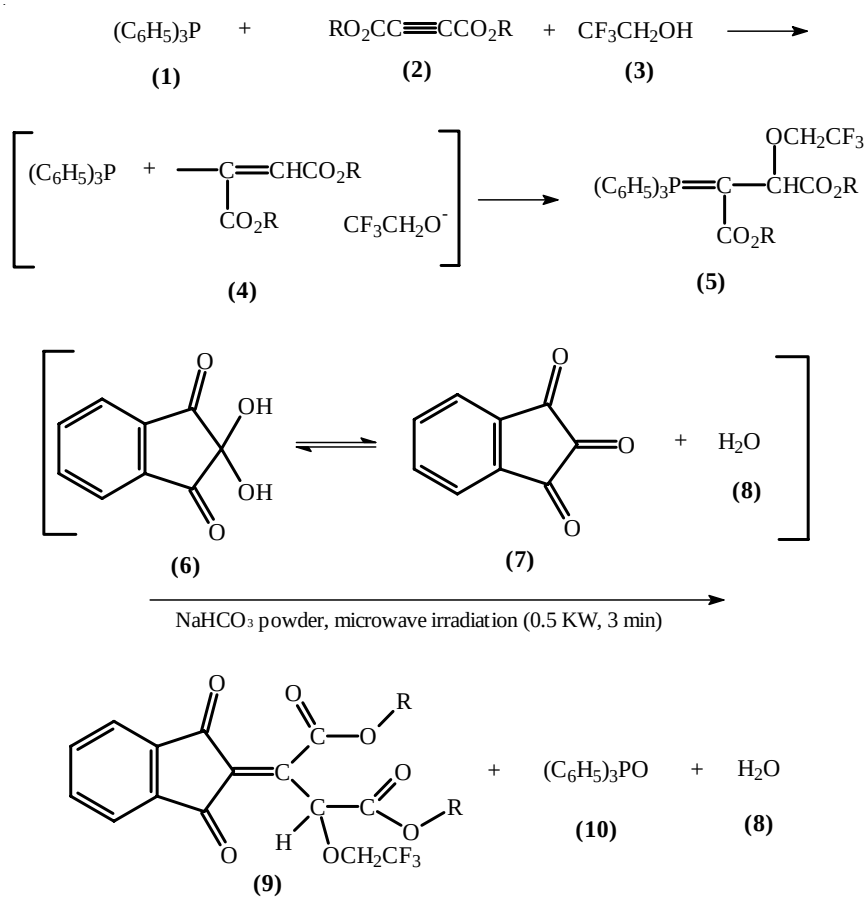
Protonation of the highly reactive 1:1 intermediates, produced in the reaction between triphenylphosphine and dialkyl acetylenedicarboxylates, by 2,2,2-trifluoroethanol leads to vinyltriphenylphosphonium salts, which undergo Michael addition reaction with conjugate base to produce the corresponding fluorine-containing stabilized phosphorus ylides. Sodium hydrogen carbonate powder was found to catalyze intermolecular Wittig reaction of the fluorine-containing stabilized phosphorus ylides with ninhydrin under microwave irradiation (0.5 KW, 3 min) in solvent-free conditions in high conversions.

Key Words: Microwave irradiation, Sodium hydrogen carbonate, Fluorine-containing stabilized phosphorus ylides, Intermolecular Wittig reaction, Ninhydrin, Solvent-free conditions, Catalyst.

Organophosphorus compounds have been extensively used in organic synthesis[1,2]. 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]. Earlier a convenient, one-pot method for preparing of stabilized phosphorus ylides utilizing *in situ* generation of the phosphonium salts has been reported[1]. In this peper, the catalytic role of sodium hydrogen carbonate powder in the intermolecular Wittig reaction of the fluorine-containing stabilized phosphorus ylides (**5**) with ninhydrin (**6**) under microwave irradiation (0.5 KW, 3 min) in solvent-free conditions is reported (**Scheme-1**).

Commerical oven Butane M245 was used for microwave irradiation. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. ^{1}H, ^{13}C and ^{19}F NMR spectra were measured with a Bruker DRX-500 Avance spectrometer at 500, 125 and 470.6 MHz, respectively.



9a: R = Me; **9b:** R = Et

Scheme-1

General procedure for the preparation of dialkyl-2-(1,3-dioxo-1,3-dihydro-2*H*-indan-2-yliden)-3-(2,2,2-trifluoroethoxy)succinates (9a-b): To a magnetically stirred solution of triphenylphosphine (**1**) (0.524 g, 2 mmol) and 2,2,2-trifluoroethanol (**3**) (0.15 mL, 2 mmol) in CH_2Cl_2 (7 mL) was added dropwise a solution of **2** (0.26 mL, 2 mmol) in CH_2Cl_2 (7 mL) at -10ºC over 15 min. The solution was allowed to warm up to room temperature, powdered ninhydrin (**6**) (0.36 g, 2 mmol) and sodium hydrogen carbonate powder (1.5 g) were added and the solvent was removed under reduced pressure. Sodium hydrogen carbonate powder and the residue were irradiated in microwave oven for 3 min at microwave power 0.5 KW and then placed over a column of silica gel powder (12 g). The column chromatography was washed using ethyl acetate-light petroleum ether as eluent. The solvent was removed under reduced pressure and the products were obtained as white solids (**9a**, m.p. 113.4-115.9ºC; **9b**, m.p.

79.5-80.9ºC). The characterization data of the compounds (**2a-d**) are given in our previous report[6].

Sodium hydrogen carbonate powder was found to catalyze the intermolecular Wittig reaction of the fluorine-containing stabilized phosphorus ylides (**5**) with ninhydrin (**6)** under microwave irradiation (0.5 KW, 3 min) in solvent-free conditions[5] (**Scheme-1**) with high conversions. TLC indicated that the reaction was completed after 3 min. The reaction proceeds smoothly and cleanly under solvent-free conditions[5] at microwave power 0.5 KW (in all cases the reaction works efficiently with high conversions) and no side reactions were observed.

Conclusion

In summary, it is found that sodium hydrogen carbonate powder is able to catalyze the intermolecular Wittig reaction of the fluorine-containing stabilized phosphorus ylides (**5**) with ninhydrin (**6**) under microwave irradiation (0.5 KW, 3 min) in solvent-free conditions[5]. Other aspects of this process are under investigation.

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