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SHORT COMMUNICATION

SYNTHESIS OF STABILIZED PHOSPHORUS YLIDES FROM ELECTRON-POOR ALCOHOLS AND THEIR APPLICATIONS IN THE PREPARATION OF 2,5-DIHYDROFURAN DERIVATIVES

Sadegh Salmanpour[1], Ali Ramazani[2*] and Yavar Ahmadi[3]

[1]Department of Chemistry, Sari Branch, Islamic Azad University, Sari, Iran
[2]Department of Chemistry, Zanjan Branch, Islamic Azad University, P.O. Box 49195-467, Zanjan, Iran
[3]Chemistry Department, Zanjan University, P.O. Box 45195-313, Zanjan, Iran

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ABSTRACT. Protonation of the highly reactive 1:1 intermediates, produced in the reaction between triphenylphosphine and dialkyl acetylenedicarboxylates, by alcohols (2-methanol thiophen, 3-methanol thiophen, 1,1,1,3,3,3-hexafluoro-2-propanol and [4-(trifluoromethyl)-phenyl]methanol) leads to vinyltriphenylphosphonium salts, which undergo Michael addition reaction with conjugate base to produce the corresponding stabilized phosphorus ylides. Wittig reaction of the stabilized phosphorus ylides with ninhydrin leads to the corresponding densely functionalized 2*H*-indeno[2,1-*b*]furans in fairly good yields.

KEY WORDS: Electron-poor alcohol, Acetylenic esters, Ninhydrin, Intramolecular Wittig reaction, Vinyltriphenylphosphonium salt

INTRODUCTION

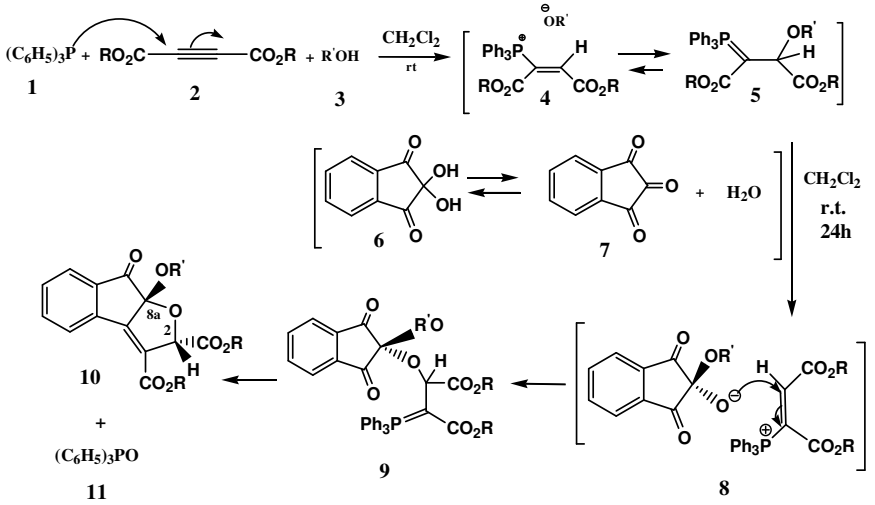
Phosphorus ylides are important reagents in synthetic organic chemistry [1-6], especially in the synthesis of naturally occurring products, compounds with biological and pharmacological activity [6]. The development of the modern chemistry of natural and physiologically active compounds would have been impossible without the phosphorus ylides [1-6]. They have found use in a wide variety of reactions of interest to synthetic chemists [1-6]. These compounds have attained great significance as widely used reagents for linking synthetic building blocks with the formation of carbon-carbon double bonds, and this has aroused much interest in the study of the synthesis, structure and properties of P-ylides and their derivatives [1-6]. Several methods have been developed for preparation of phosphorus ylides [1]. These ylides are most often prepared by the treatment of a phosphonium salt with a base. Most of the phosphonium salts are usually made from phosphine and an alkyl halide [1], and they are also obtained by the Michael addition of phosphorus nucleophiles to activated olefins [2]. β-Additions of nucleophiles to the vinyl group of vinylic phosphonium salts leading to the formation of new alkylidenephosphoranes have attracted much attention as a very convenient and synthetically useful method in organic synthesis [7-16]. Phosphorus ylides are a class of special type of zwitterions, which bear strongly nucleophilic electron rich carbanions. The electron distribution around the P^+–C^- bond and its consequent chemical implications had been probed and assessed through theoretical, spectroscopic and crystallographic investigations [2]. The proton affinity of these ylides can be used as a molecular guide to assess their utility as synthetic reagents and their function as ligands in coordination and organometallic chemistry [3, 4]. In the past we have established a convenient, one-pot method for preparing stabilized phosphorus ylides utilizing an *in situ* generation of the phosphonium salts [7-16]. Stabilized phosphorus ylides, versatile intermediates in synthetic organic chemistry can be prepared by the novel reaction of

*Corresponding author. E-mail: aliramazani@gmail.com

dialkyl acetylenedicarboxylates (DAAD), triphenylphosphine (TPP) and acids such as phenols, imides, amides, enols, oximes and alcohols [7-16]. The reaction [2] involves an intermediate formed by the 1:1 conjugate addition reaction of the TPP to DAAD, and concomitant protonation of the intermediate by an acid leads to vinyltriphenylphosphonium salts [7-16]. The salts are unstable intermediates and converted to stabilized phosphorus ylides *via* a Michael addition reaction [7-16]. The stabilized phosphorus ylides are able to take part in the intramolecular Wittig reactions [2] but they are not generally able to participate in the intermolecular Wittig reactions [2]. The ylides are converted to electron-poor alkenes *via* elimination of TPP in solvent-free conditions [2]. Almost all of the final products are valuable families of compounds [2].

Compounds with [2,1-*b*]furan skeleton have attracted interest in bio-organic, natural products and medicinal chemistry. They show pharmaceutical and biological activities including antimicrobial and *anti*-fungal effects [17-20]. Since years acetylenic esters have attracted the attention of organic chemists, mostly as Michael acceptors [2]. In recent years, there has been increasing interest on the applications of acetylenic esters in multi-component syntheses [2], specially for preparing stabilized phosphorus ylides [2]. Due to atom economy, convergent character and simplicity of one-pot procedures, multi-component condensation reactions (MCRs) have great potentials in synthesis. The development of novel MCRs is also receiving growing interest from industrial chemistry research groups and represents a challenge for organic chemists [21, 22]. Few methods have been reported in the literature for the synthesis of indeno[2,1-*b*]furans [18, 19]. These protocols are multi-step in nature.

In the past we have established a convenient, one-pot method for preparing stabilized phosphorus ylides utilizing *in situ* generation of the phosphonium salts [9-16]. As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds [23-43], we sought to develop a convenient preparation of densely functionalized 2*H*-indeno[2,1-*b*]furans **10**. In this paper, the one-pot diastereoselective synthesis of densely functionalized **10** from triphenylphosphine (**1**), dialkyl acetylenedicarboxylates (**2**), electron-poor alcohols (**3**), and ninhydrin (**6**) in fairly good yields is being described (Scheme 1 and Table 1).



Scheme 1. Synthesis and mechanism of the formation of dihydrofuran derivatives **10a-h** (see Table 1).

Table 1. Synthesis of dihydrofuran derivatives **10a-h** (see Scheme 1).

10	R	R'	%Yield[a]
a	Me	Thiophene-2-CH_2O-	80
b	Et	Thiophene-2-CH_2O-	70
c	Me	Thiophene-3-CH_2O-	80
d	Et	Thiophene-3-CH_2O-	75
e	Me	$(CF_3)_2HCO$-	80
f	Et	$(CF_3)_2HCO$-	78
g	Me	4-$CF_3C_6H_4CH_2O$-	88
h	Et	4-$CF_3C_6H_4CH_2O$-	71

[a]Isolated yields.

RESULTS AND DISCUSSION

Reactions are known in which an α,β-unsaturated carbonyl compound is produced from a phosphorane and a carbonyl compound such as an aldehyde or ketone [7, 16]. Thus, compounds **10a-h** may be regarded as the product of an intramolecular Wittig reaction. Such addition-olefination products may result from an initial addition of triphenylphosphine **1** to the acetylenic ester **2** and concomitant protonation of the 1:1 adduct, followed by attack of the anion **4** of electron-poor alcohol **3** on the vinylphosphonium ion to form the stabilized phosphorane **5**. It seems that the compounds **4** and **5** are in equilibrium in CH_2Cl_2 at room temperature. Addition of **4** to the highly electron-deficient carbonyl group of indane-1,2,3-trione (**7**) leads to the formation of the vinyltriphenylphosphonium salt **8** that converts to phosphorus ylide **9**. Intramolecular Wittig reaction of the phosphorus ylide **9** will then lead to the formation of corresponding 2*H*-indeno[2,1-*b*]furan derivatives **10a-h** and triphenylphosphine oxide (Scheme 1 and Table 1). TLC results indicated that the reactions are completed after 24 h in CH_2Cl_2 at room temperature. The reactions proceed smoothly and cleanly under mild conditions and no side reactions were observed. The mechanism of the reaction has not been established experimentally. However, the reaction sequences proposed in Scheme 1 are plausible.

CONCLUSIONS

In conclusion, the reported method offers a mild, simple, and efficient route for the diastereoselective synthesis of dihydrofuran derivatives **10a-h**. The ease of work-up, good yields and fairly mild reaction conditions make it a useful addition to modern synthetic methodologies. Other aspects of this process are under investigation.

EXPERIMENTAL

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu IR-460 spectrometer. ^{1}H and ^{13}C NMR spectra were measured with a Bruker Spectrospin spectrometer at 250 and 62.5 MHz, respectively.

*Synthesis of dihydrofuran derivatives **10a-h***. To a magnetically stirred solution of Ph_3P (**1**; 0.262 g, 1 mmol) and alcohol **3** (1 mmol) in CH_2Cl_2 (4 mL), diester **2** (1 mmol) in CH_2Cl_2 (4 mL) was added drop wise at -10 ºC over 15 min. The mixture was allowed to warm up to room temperature. Then powdered ninhydrin (**6**; 0.18 g, 1 mmol) was added, and the mixture was stirred for 24 h. The solvent was evaporated and the viscous residue purified by flash column chromatography (silica gel, petroleum ether/AcOEt (5/2)).

*Dimethyl 8-oxo-8a-(thiophen-2-ylmethoxy)-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10a**)*. Colorless solidified oil; yield (80%); IR (neat) (ν_{max}, cm^{-1}): 2956, 1754, 1720,

1438, 1033 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 3.77 and 3.84 (6H, 2s, $2OCH_3$); 4.88 and 5.11 (2H, 2d, $^2J_{HH}$ = 12 Hz, OCH_AH_BC4H3S); 6 (1H, 1s, OCH); 6.89 and 7.25 (3H, arom); 7.64 (1 H, t, $^3J_{HH}$ = 7.6 Hz, arom.); 7.77 (1H, t, $^3J_{HH}$ = 7.6 Hz, arom.); 7.87 (1H, d, $^3J_{HH}$ = 7.6 Hz, arom.); 8.24 (1H, d, $^3J_{HH}$ = 7.6 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 52.33 and 52.91 ($2OCH_3$); 61.92 (OCH_2); 89.66 (OCH); 125.42, 126.52, 126.77, 127.26, 128.34, 132.40 and 136.06 (7CH, arom.); 122.65, 138.20, 139.45, 140.14 and 149.20 (5C); 162.03 and 168.10 (2C=O of 2 esters); 189.75 (C=O of ketone).

*Diethyl 8-oxo-8a-(thiophen-2-ylmethoxy)-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10b**)*. Colorless solidified oil; yield (70%); IR (neat) (ν_{max}, cm^{-1}): 2988, 1739, 1723, 1605, 1468, 1243 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 1.28 and 1.36 (6H, 2t, $^3J_{HH}$ = 7.1 Hz, $2CH_3$ of 2Et); 4.10-4.40 (4H, m, $2OCH_2$ of 2Et); 4.32 and 4.54 (2H, 2d, $^2J_{HH}$ = 12 Hz, OCH_AH_BC4H3S); 6.05 (1H, s, OCH); 6.89 and 7.25 (3H, arom); 7.65 (1H, t, $^3J_{HH}$ = 7.7 Hz, arom.); 7.81 (1H, t, $^3J_{HH}$ = 7.7 Hz, arom.); 7.87 (1H, d, $^3J_{HH}$ = 7.7 Hz, arom.); 8.34 (1H, d, $^3J_{HH}$ = 7.7 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 13.99 and 14.16 ($2CH_3$); 61.70 and 62.14 (2 OCH_2 of 2Et); 76.31 (OCH_2CCl_3); 90.35 (OCH); 125.52, 126.58, 126.80, 127.30, 128.62, 132.50 and 136.34 (4CH, arom.); 122.80, 138.25, 139.55, 140.24 and 149.70 (4C); 161.51 and 167.37 (2 C=O of 2 esters); 189.14 (C=O of ketone).

*Dimethyl 8-oxo-8a-(3-thiophenylmethoxy)-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10c**)*. Yellow oil; yield (80%); IR (neat) (ν_{max}, cm^{-1}): 2950, 1750, 1715, 1434, 1034, 767 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 3.77 and 3.85 (6H, 2s, $2OCH_3$); 4.74 and 4.95 (2H, 2d, $^2J_{HH}$ = 11.5 Hz, $OCH_AH_BC_4H_3S$); 5.96 (1H, 1s, OCH); 6.96 and 7.20 (3H, arom); 7.63 (1 H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.77 (1H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.87 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.); 8.24 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 52.33 and 52.89 ($2OCH_3$); 62.54 (OCH_2); 89.64 (OCH); 123.62, 125.36, 126.13, 127.33, 128.30, 132.38 and 135.99 (7CH, arom.); 113.59, 122.53, 137.91, 138.21, 140.20 and 149.29 (6C); 162.04 and 168.13 (2 C=O of 2 esters); 189.82 (C=O of ketone).

*Diethyl 8-oxo-8a-(3-thiophenylmethoxy)-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10d**)*. Colorless oil; yield (75%); IR (neat) (ν_{max}, cm^{-1}): 2976, 1741, 1710, 1597, 1466, 1029, 706 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 1.27 and 1.36 (6H, 2t, $^3J_{HH}$ = 7 Hz, 2 CH_3 of 2 Et); 4.20 and 4.35 (4H, m, $2OCH_2$ of 2 Et); 4.74 and 4.94 (2H, 2d, $^2J_{HH}$ = 11.5 Hz, $OCH_AH_BC_4H_3S$); 5.94 (1H, s, OCH); 6.96 and 7.23 (3H, arom); 7.61 (1H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.76 (1H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.86 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.); 8.27 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 13.97 and 14.15 ($2CH_3$); 61.70 and 62.14 (2 OCH_2 of 2Et); 62.48 (OCH_2); 89.90 (OCH); 123.61, 125.31, 126.10, 127.35, 128.39, 132.25 and 135.90 (7CH, arom.); 113.63, 123.07, 137.96, 138.33, 140.16 and 149.02 (6C); 161.63 and 167.74(2 C=O of 2 esters); 189.96 (C=O of ketone).

*Dimethyl 8-oxo-8a-(2,2,2-trifluoro-1-(trifluoro methyl)ethoxy)-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10e**)*. Yellow solids; yield: 80%; m. p.: 116 -118 °C. IR (KBr) (ν_{max}, cm^{-1}): 2963, 2931, 1758, 1602, 1441, 1364, 1290, 1193 and 1103 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 3.78 and 3.87 (6H, 2s, $2OCH_3$); 4.99 (1H, septet, $^3J_{HF}$ = 5.75 Hz, OCH (CF_3)$_2$); 6.00 (1H, 1s, OCH); 7.67 (1 H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.81 (1H, t, $^3J_{HH}$ = 7.5 Hz, arom.); 7.87 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.); 8.30 (1H, d, $^3J_{HH}$ = 7.5 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 52.58 and 53.08 ($2OCH_3$); 70.08 ($2CF_3$, septet, ^1JCF= 132.5 Hz); 90.57 (OCH); 125.60, 128.46, 132.96 and 136.7 (4CH, arom.); 113.44, 123.80, 137.92, 139.58 and 147.22 (5C); 161.59 and 167.29 (2 C=O of 2 esters); 188.65 (C=O of ketone).

*Diethyl 8-oxo-8a-(2,2,2-trifluoro-1-(trifluoromethyl)ethoxy)-8,8a-dihydro-2H-indeno[2,1-b]-furan-2,3-dicarboxylate (**10f**)*. Yellow solids; yield: 78%; m. p.: 68-70 °C. IR (KBr) (ν_{max}, cm^{-1}): 2988, 2963, 1725, 1614, 1371, 1290, 1240 and 1192 cm^{-1}. ^1H NMR ($CDCl_3$) δ_H: 1.28 and 1.35 (6H, 2t, $^3J_{HH}$ = 7.25 Hz, $2CH_3$ of 2 Et); 4.15 and 4.46 (4H, m, $2OCH_2$ of 2 Et); 4.99 (1H, septet, $^3J_{HF}$ = 5.75 Hz, $OCH(CF_3)_2$); 6.00 (1H, 1s, OCH); 6.00 (1H, s, OCH); 7.68 (1H, t, $^3J_{HH}$ = 7.75 Hz, arom.); 7.81 (1H, t, $^3J_{HH}$ = 7.75 Hz, arom.); 7.87 (1H, d, $^3J_{HH}$ = 7.75 Hz, arom.); 8.32 (1H, d, $^3J_{HH}$ = 7.75 Hz, arom.). ^{13}C NMR ($CDCl_3$) δ_C: 13.93 and 14.16 ($2CH_3$); 62.13 and 62.48 (2 OCH_2 of 2Et); 70.09 ($2CF_3$, septet, ^1JCF = 132.5 Hz); 90.84 (OCH); 122.48, 124.56, 129.53 and 134.57 (4CH, arom.); 112.95, 125.55,128.54, 132.83 and 138.05 (5C); 161.51 and 166.91(2 C=O of 2 esters); 189.14 (C=O of ketone).

*Dimethyl 8-oxo-8a-[4-(trifluoromethyl)benzyloxy]-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10g**)*. White crystals, yield: 88%, m.p.: 126.0-127.5. IR (KBr) (ν_{max}, cm^{-1}): 2965 (C–H, aliphatic); 1758 (C=O, ketone); 1727 (C=O, ester); 1400-1600 (C=C, arom), 1241 (C–O, ether). ^1H NMR ($CDCl_3$) δ_H: 3.77 and 3.86 (6H, 2s, $2OCH_3$); 4.78 and 4.99 (2H, 2d, $^3J_{HH}$ = 12 Hz, OCH_2); 5.96 (1H, s, OCH); 7.35 and 7.54 (4H, 2d, $^3J_{HH}$ = 8 Hz, arom); 7.67(1H,t, $^3J_{HH}$ = 7.5Hz, CH, arom); 7.82 (1H, t, $^3J_{HH}$ = 7.5 Hz, CH, arom); 7.88 (1H, d, $^3J_{HH}$ = 7.5 Hz, CH, arom); 8.26 (1H, d, $^3J_{HH}$ = 7.5 Hz, CH, arom). ^{13}C NMR ($CDCl_3$) δ_C: 52.41 and 52.96 ($2OCH_3$); 66.39 (OCH_2); 89.74 (OCH); 113.57 (1C); 126.18 (CF_3, q, $^1J_{CF}$ = 270 Hz); 122.7 and 149.06 (2C, C=C); 125.33 (CH, q, $^3J_{CF}$ = 3.7 Hz, arom); 125.45, 127.88, 128.34, 132.51, 136.17 (5CH, arom); 130.03 (C, q, $^2J_{CF}$ = 31 Hz, arom); 138.18, 140.08, 141.03 (3C, arom); 161.97, 168.01 (2C = O, ester); 189.88 (C = O, ketone).

*Diethyl 8-oxo-8a-[4-(trifluoromethyl)benzyloxy]-8,8a-dihydro-2H-indeno[2,1-b]furan-2,3-dicarboxylate (**10h**)*. White crystals, yield: 71%, m.p.: 111.0-112.0. IR (KBr) (ν_{max}, cm^{-1}): 2985 and 2931 (C–H, aliphatic); 1727 (C=O, ketone); 1400-1600 (C=C, arom), 1249 (C–O, ether). ^1H NMR ($CDCl_3$) δ_H: 1.28 and 1.35 (6H, 2t, $^3J_{HH}$ = 7.5 Hz, $2CH_3$); 4.1- 4.4 (4H, m, $2OCH_2$); 4.78 and 4.99 (2H, 2d, $^3J_{HH}$ = 12 Hz, OCH_2); 5.941 (1H, s, OCH); 7.35 and 7.545 (4H, 2d, $^3J_{HH}$ = 8 Hz, arom); 7.65 (1H, t, $^3J_{HH}$ = 7.7 Hz, CH, arom); 7.81 (1H, t, $^3J_{HH}$ = 7.7 Hz, CH, arom); 7.87 (1H, d, $^3J_{HH}$ = 7.7 Hz, CH, arom); 8.28 (1H, d, $^3J_{HH}$ = 7.7 Hz, CH, arom). ^{13}C NMR ($CDCl_3$) δ_C: 14.3 and 14.28 ($2CH_3$); 61.6 and 62.19 ($2CH_2$); 66.4 (OCH_2); 90.1 (OCH); 114.3 (1C); 126.18 (CF_3, q, $^1J_{CF}$ = 270 Hz); 122.7 and 148.88 (2C, C = C); 125.33 (CH, q, $^3J_{CF}$ = 3.7 Hz, arom); 125.45, 127.88, 128.34, 132.51, 136.17 (5CH, arom); 130.03 (C, q, $^2J_{CF}$ = 31 Hz, arom); 138.18, 140.08, 141.03 (3C, arom); 161.71, 167.37 (2C = O, ester); 189.8 (C = O, ketone).

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