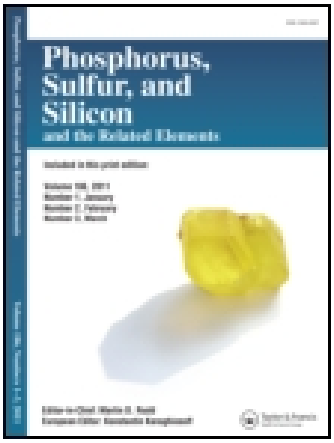

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Triphenylphosphine-Catalyzed Preparation of Sterically Congested, Electron-Poor N-Vinylimidazole Derivatives from Acetylenic Esters and Imidazole-Containing NH-Acids

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TRIPHENYLPHOSPHINE-CATALYZED PREPARATION OF STERICALLY CONGESTED, ELECTRON-POOR *N*-VINYLIMIDAZOLE DERIVATIVES FROM ACETYLENIC ESTERS AND IMIDAZOLE-CONTAINING NH-ACIDS

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Protonation of the highly reactive 1:1 intermediates, produced in the reaction between triphenylphosphine and alkyl acetylenecarboxylates (or ethyl 3-phenyl-2-propynoate), by azathioprine or imidazole leads to vinyltriphenylphosphonium salts, which undergo a Michael addition reaction with a conjugate base to produce phosphorus ylides. Dipotassium hydrogen phosphate, potassium dihydrogen phosphate, and potassium iodide were found to catalyze the conversion of the phosphorus ylides to electron-poor N-vinyl imidazoles in solvent-free conditions under thermal (90°C, 1 h) conditions. The structural analysis of the products indicated that the reaction is regio- and stereoselective.

Keywords Acetylenic esters; dipotassium hydrogen phosphate; electron-poor *N*-vinyl imidazoles; potassium dihydrogen phosphate; potassium iodide; regioselective; stereoselective; vinyltriphenylphosphonium salt

INTRODUCTION

Imidazole chemistry currently attracts considerable attention, where the imidazole derivatives are widely applied as *N*-ligand coordinating transition metals.[1,2] The application of imidazoles in medicinal chemistry[3] or chemistry of natural products/alkaloids[4,5] or of 1,3-disubstituted imidazole salts as ionic liquids[6,7] are also well known.

1-Vinylimidazole is employed as a copolymer in the production of cationic polymers for various uses. Alkylimidazoles are used as hardeners for epoxy resins and for polyurethanes.[8] Less important uses for alkyl- and arylimidazoles include photography and dyes.[8]

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

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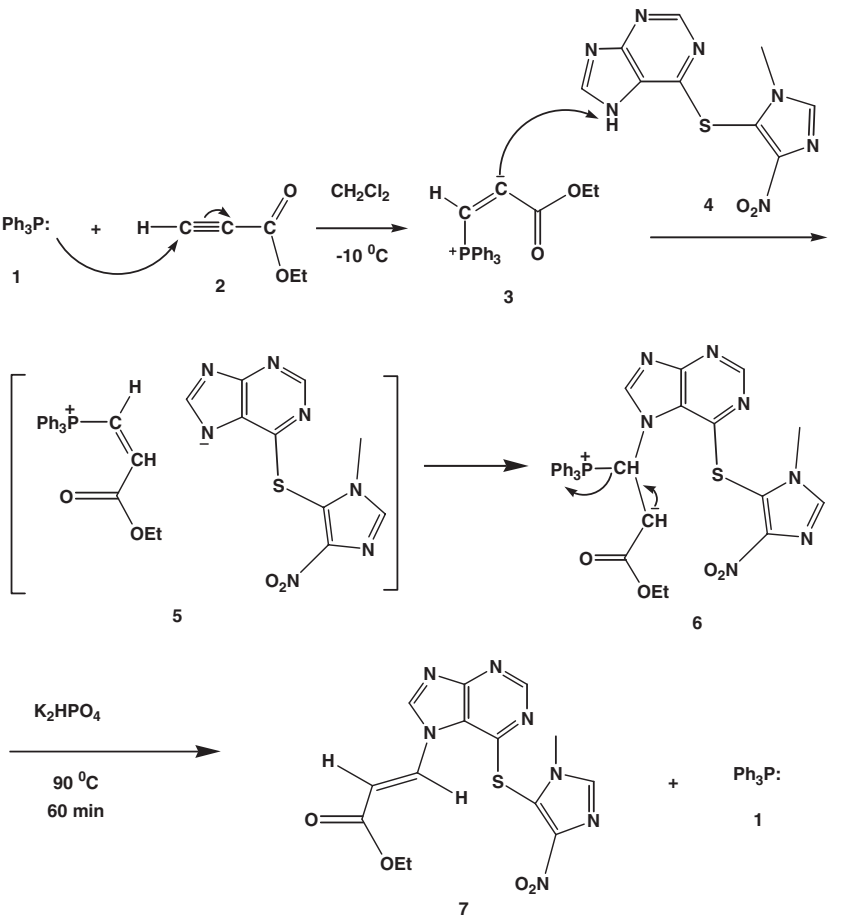
very convenient and synthetically useful method in organic synthesis.[9] Organophosphorus compounds have been extensively used in organic synthesis as useful reagents as well as ligands of a number of transition metal catalysts.[10] Phosphorus ylides are a class of a special type of zwitterions, which 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.[9] The nucleophilicity at the ylidic carbon is a factor of essential mechanistic importance in the use of these ylides as Wittig reagents. Phosphorus ylides are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products, and compounds with biological and pharmacological activity.[11] These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually obtained from a phosphine and an alkyl halide. Phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins and in other ways.[12] The phosphonium salts are most often converted to the ylide by treatment with a strong base, though weaker bases can be used if the salt is acidic enough. In recent years, we have established a one-pot method for the synthesis of stabilized phosphorus ylides.[13–35] In this article, we describe the preparation of sterically congested, electron-poor *N*-vinyl imidazoles from alkyl acetylenecarboxylates (or ethyl 3-phenyl-2-propynoate) and azathioprine or imidazole in the presence of triphenylphosphine in fairly good yields.

RESULTS AND DISCUSSION

The zwitterionic intermediate **6** may result from the initial addition of triphenylphosphine **1** to the ethyl acetylenecarboxylate **2** and concomitant protonation of the 1:1 adduct **3**, followed by attack of the NH-acid anion on the vinyltriphenylphosphonium cation (Scheme 1). TLC indicated the formation of zwitterionic intermediate **6** in CH_2Cl_2. Dipotassium hydrogen phosphate, potassium dihydrogen phosphate, and potassium iodide powders were found to catalyze conversion of the zwitterionic intermediates **6** to electron-poor imidazole **7** in solvent-free conditions[36] under thermal (90°C, 1 h) conditions. In the absence of the K_2HPO_4, KH_2PO_4, and KI powders, the conversion of the zwitterionic intermediates **6** to electron-poor *N*-vinyl imidazole **7** were not observed, and decomposition of the starting materials were observed (Scheme 1). The reaction proceeds smoothly and cleanly under mild conditions, and no side reactions were observed. The mechanism of the reaction has not been established experimentally. However, a possible explanation[37,38] is proposed in Scheme 1. We have also used MgO, K_2CO_3, Al_2O_3, SiO_2, and Na_2CO_3 in this reaction, but no products were observed (or in the some cases the conversion percentages were very low), and in all cases decomposition was observed.

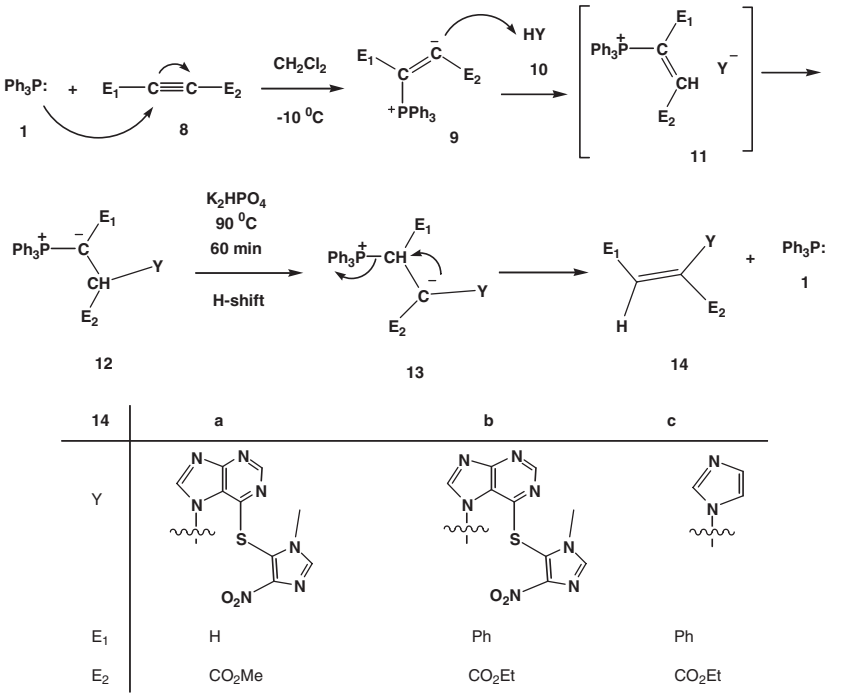
We have also used methyl acetylenecarboxylate (or ethyl 3-phenyl-2-propynoate) **8** (Scheme 2) instead of ethyl acetylenecarboxylate **2** in this reaction, but the regiochemistry of the products was completely different from the ethyl ester derivative. Differences in the steric effects of Me and Et groups may be plausible factors in the formation of different regioisomers. The zwitterionic intermediate **13** may result from the initial addition of triphenylphosphine **1** to the methyl acetylenecarboxylate (or ethyl 3-phenyl-2-propynoate) **8** and concomitant protonation of the 1:1 adduct **9**, followed by attack of the NH-acid anion on the vinyltriphenylphosphonium cation to form the phosphorane **12** (Scheme 1). That fact that phosphorane **12** undergoes intramolecular proton transfer[37,38] leads to formation of zwitterionic intermediate **13** in CH_2Cl_2. Dipotassium hydrogen phosphate, potassium



Scheme 1 Synthesis of *N*-vinyl azathioprine **7** from ethyl acetylenecarboxylate **2** and azathioprine **4** in the presence of triphenylphosphine **1**.

dihydrogen phosphate, and potassium iodide powders were found to catalyze conversion of the zwitterionic intermediate **13** to electron-poor imidazoles (**14**) in solvent-free conditions[36] under thermal (90°C, 1 h) conditions. In the absence of the K_2HPO_4, KH_2PO_4, and KI powders, the conversion of the zwitterionic intermediate **13** toelectron-poor *N*-vinyl imidazoles (**14**) was not observed, and decomposition of the starting materials were observed (Scheme 2). The reaction proceeds smoothly and cleanly under mild conditions and no side reactions were observed. The mechanism of the reaction has not been established experimentally. However, a possible explanation[37,38] is proposed in Scheme 2. We have also used MgO, K_2CO_3, Al_2O_3, SiO_2, and Na_2CO_3 in this reaction, but no products were observed (or in the some cases the conversion percentages were very low), and in all cases decomposition were observed.

The structures **7** and **14** (**a**, **b**, and **c**) were deduced from their IR, ^{1}H and ^{13}C NMR spectra. The ^{1}H NMR spectra of compound **7** exhibited two doublet peaks at $\delta = 7.09$ and 8.23 for the olefinic protons of the electron-poor double bond, which are very consistent with their ***E*** ($^3J_{HH} = 14.3$ Hz) stereochemistry. The ethoxy group and the heterocyclic



Scheme 2 Synthesis of *N*-vinyl imidazole derivatives **14** from acetylenic esters **8** and imidazoles **10** in the presence of triphenylphosphine **1**.

moiety give characteristic signals at the appropriate chemical shifts (see the Experimental section). Further evidence was obtained from the ^{13}C NMR spectra, which displayed a signal for the carbonyl carbon at $\delta = 166.00$ (see the Experimental section). The ^{1}H NMR spectra of compound **14a** exhibited two doublet peaks at $\delta = 6.42$ and $\delta = 6.75$ ($^{2}J_{HH} = 1.0$ Hz) for the two diastereotopic protons of the olefinic methylene group.[36] The methoxy group and the heterocyclic moieties give characteristic signals at the appropriate chemical shifts (see the Experimental section). Further evidence was obtained from the ^{13}C NMR spectra, which displayed a signal for the methylene carbon at $\delta = 125.19$ and a signal for the carbonyl carbon at $\delta = 162.24$ (see the Experimental section). The ^{1}H NMR spectra of compounds **14b** exhibited a singlet peak at $\delta = 7.76$ for the olefinic proton of the electron-poor double bond, which is very consistent with its ***Z*** stereochemistry.[23] The ethoxy group and the heterocyclic moiety give characteristic signals at the appropriate chemical shifts (see the Experimental section). Further evidence for **14b** was obtained from the ^{13}C NMR spectra, which displayed a signal for the carbonyl carbon at $\delta = 163.00$ (see the Experimental section), which is very consistent with its structure. The ^{1}H and ^{13}C NMR spectra of compound **14c** were similar to those of **14b**, except for the heterocyclic moiety, which exhibited characteristic signals with appropriate chemical shifts.

CONCLUSION

In conclusion, we have developed a convenient, one-pot, regio-, and stereoselective method for preparing electron-poor imidazoles **7** and **14** (**a**, **b**, and **c**) utilizing in situ

generation of the phosphonium salts (**5** and **11**) (Schemes 1 and 2). We believe that the reported method offers a mild and simple route for the preparation of these derivatives. Its ease of workup and reaction conditions make it a useful addition to modern synthetic methodologies.[39–49] 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. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer.

General Procedure for the Preparation of 7 and 14a

To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and azathioprine (0.28 g, 1 mmol) in dichloromethane (5 mL), a mixture of ethyl acetylenecarboxylate **2** (or methyl acetylenecarboxylate **8a**) (1 mmol) in dichloromethane (2 mL) at –10°C was added dropwise over 15 min. The mixture was allowed to warm up to room temperature. Dipotassium hydrogen phosphate powder (1.5 g) was added, and the solvent was evaporated. Dry dipotassium hydrogen phosphate and the residue were heated (yield for **7**, 66%; yield for **14a**, 60%) for 60 min at 90°C and then placed over a column of silica gel powder (12 g). The flash column chromatography was washed using ethyl acetate/light petroleum ether (5:10) as eluent. The solvent was removed under reduced pressure, and products were obtained as viscous yellow oils (**7, 14a**) (Scheme 1 and Scheme 2). The characterization data of the compounds (**7**, **14a**) are given below.

Selected data for ethyl (E)-2-{6-[(1-methyl-4-nitro-1H-imidazol-5-yl) sulfanil]-7H-purin-7-yl}acrylate (7). Viscous yellow oil; yield: 66%, IR (Neat) (υ_{max}, cm^{-1}): 2923, 2853, 1723, 1661 and 1192. ^{1}H NMR (DMSO-d_6) δ_H: 1.24 (t, 3 H, $^{3}J_{HH}$ = 7.0 Hz, CH_3 of OEt), 3.34 (s, 3 H, CH_3), 4.19 (q, 2 H, $^{3}J_{HH}$ = 7.0 Hz, OCH_2 of OEt), 7.09 and 8.23 (2 d, 2 H, $^{3}J_{HH}$ = 14.3 Hz, HC=CH), 8.22 and 8.73 (2 s, 2 H, CH=N of imidazole), 8.91 (s, 1 H, CH=N of pyrimidine). ^{13}CNMR (DMSO-d_6) δ_C: 14.52 (CH_3 of OEt) 33.43 (CH_3), 61.02 (CH_2 of OEt), 109.95 and 134.85 (2 CH of vinyl), 140.01 and 145.72 (2 CH of imidazole), 153.18 (CH of pyrimidine), 116.96, 131.81, 149.65, 150.02 and 156.95 (5 C), 166.00 (C=O of ester). Anal. Calcd for $C_{14}H_{13}N_7O_4S$ (375.36): C, 44.80; H, 3.49; N, 26.12%. found: C, 45.02; H, 3.44; N, 26.01%.

Selected data for methyl-2-{6-[(1-methyl-4-nitro-1H-imidazol-5-yl) sulfanil]-7H-purin-7-yl} acrylate (14a). Viscous yellow oil; yield: 60%. IR(neat)(υ_{max}, cm^{-1}): 3123, 2923, 1738, 1646 and 1192 . ^{1}H NMR ($CDCl_3$) δ_H: 3.76 (s, 3 H, CH_3), 3.89 (s, 3 H, OCH_3), 6.42 and 6.75 (2 d, 2H, $^{2}J_{HH}$ = 1.0 Hz, $=CH_2$), 7.76 and 8.24 (2 s, 2 H, CH=N of imidazole), 8.59 (s, 1 H, CH=N of pyrimidine). ^{13}C NMR ($CDCl_3$) δ_C: 33.21 (CH_3), 53.36 (OCH_3), 125.19 ($=CH_2$), 138.03 and 143.94 (2 CH of imidazole), 152.54 (CH of pyrimidine), 116.51, 130.57, 138.03, 150.35 and 157.04 (6 C), 162.24 (C=O of ester). Anal. Calcd for $C_{13}H_{11}N_7O_4S$ (361.34): C, 43.21; H, 3.07; N, 27.13%. found: C, 43.32; H, 3.03; N, 27.01%.

General Procedure for the Preparation of 14b and 14c

To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and azathioprine **10b** (or imidazole **10c**) (1 mmol) in dichloromethane (5 mL), a mixture

of ethyl 3-phenyl-2-propynoate **8b** (1 mmol) in dichloromethane (2 mL) at –10°C was added dropwise over 15 min. The mixture was allowed to warm up to room temperature. Dipotassium hydrogen phosphate powder (1.5 g) was added, and the solvent was evaporated. Dry dipotassium hydrogen phosphate and the residue were heated (yield for **14b**, 71%; yield for **14c**, 97%) for 60 min at 90°C and then placed over a column of silica gel powder (12 g). The flash column chromatography was washed using ethyl acetate/light petroleum ether (5:10) as eluent. The solvent was removed under reduced pressure, and products were obtained as viscous yellow oil (**14b**) and white crystals (**14c**) (Scheme 2). The characterization data of the compounds (**14b** and **14c**) are given below.

Selected data for ethyl (Z)-2-{6-[(1-methyl-4-nitro-1H-imidazol-5-yl) sulfanil]-7H-purin-7-yl}-3-phenyl-2-propenoate (14b). Viscous yellow oil; yield: 71%. IR (Neat) (υ_{max}, cm^{-1}): 3115, 2930, 1723, 1646, 1269 and 1207. ^{1}H NMR ($CDCl_3$) δ_H: 1.31 (t, 3 H, $^3J_{HH}$ = 7.0 Hz, CH_3 of OEt), 3.75 (s, 3 H, CH_3), 4.33 (q, 2 H, $^3J_{HH}$ = 7.0 Hz, OCH_2 of OEt), 6.90 (d, 2 H, $^3J_{HH}$ = 7.3 Hz, arom), 7.22–7.37 (m, 5 H, arom), 7.76 (s, 1 H, =CH), 7.90 and 8.16 (2 s, 2 H, CH=N of imidazole), 8.53 (s, 1 H, CH=N of pyrimidine). ^{13}C NMR ($CDCl_3$) δ_C: 14.17 and 33.29 (2 CH_3), 62.58 (OCH_2 of OEt), 121.29 (CH of vinyl), 129.29, 129.80 and 130.83 (5 CH of Ph), 141.13 and 144.07 (2 CH of imidazole), 152.76 (CH of pyrimidine), 116.87, 129.29, 131.49, 138.00, 150.35 and 157.01 (7 C), 163.00 (C=O of ester). Anal. Calcd for $C_{20}H_{17}N_7O_4S$ (451.46): C, 53.21; H, 3.80; N, 21.72%. found: C, 53.42; H, 3.84; N, 21.59%.

Selected data for ethyl (Z)-2-(1H-imidazol-1-yl)-3-phenyl-2-propenoate (14c). White crystals; yield: 97%, mp: 90–92°C. IR (KBr) (υ_{max}, cm^{-1}): 2969, 1712, 1643, 1257 and 1211. ^{1}H NMR ($CDCl_3$) δ_H: 1.35 (t, 3 H, $^3J_{HH}$ = 7.0 Hz, CH_3 of OEt), 4.33 (q, 2 H, $^3J_{HH}$ = 7.0 Hz, OCH_2 of OEt), 6.89 and 7.29 (2 d, 2 H, CH=CH of imidazole), 7.25–7.35 (m, 5 H, arom), 7.44 (s, 1 H, =CH), 7.85 (s, 1 H, =CH of imidazole). ^{13}C NMR ($CDCl_3$) δ_C: 14.17 (CH_3), 62.14 (OCH_2 of OEt), 119.37, 130.06 and 138.15 (3 CH of imidazole), 125.12 (CH of vinyl), 128.99, 130.06 and 130.92, (5 CH of Ph), 131.27 and 137 (2 C), 163.78 (C=O of ester). Anal. Calcd for for $C_{14}H_{14}N_2O_2$ (242.27): C, 69.41; H, 2.82; N, 11.56%. found: C, 69.38; H, 2.85; N,11.58%.

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