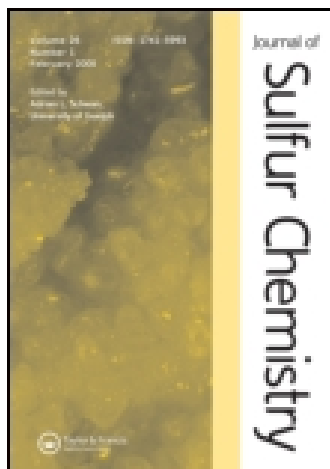


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Three-component reaction between triphenylphosphine, dialkyl acetylenedicarboxylate and 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thiones: an efficient one-pot synthesis of stable phosphorus ylides

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Three-component reaction between triphenylphosphine, dialkyl acetylenedicarboxylate and 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thiones: an efficient one-pot synthesis of stable phosphorus ylides

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Protonation of the reactive 1:1 intermediate produced in the reaction between dialkyl acetylenedicarboxylates and triphenylphosphine by 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thione leads to vinylphosphonium salts which undergo Michael addition with the conjugate base of the NH-acid to produce highly functionalized, salt-free phosphorus ylides in excellent yields.

Keywords: addition reaction; dialkyl acetylenedicarboxylates; phosphorus ylides; triphenylphosphine; NH-acid

1. Introduction

Organophosphorus compounds, those bearing a carbon atom bound directly to a phosphorus atom, are synthetic targets of interest, at least because of their value for a variety of industrial, biological, and chemical synthetic uses (1–3). Several methods have been developed for the preparation of phosphorus ylides. These ylides are usually prepared by treatment of an appropriate phosphonium salt with a base; the corresponding phosphonium salts are usually obtained from the phosphine and an alkyl halide (1, 2). Phosphonium salts are also prepared by the Michael addition of phosphorus nucleophiles to activated olefins (1, 2). Reaction of acetylenic esters with triphenylphosphine in the presence of an organic compound possessing an acidic hydrogen has been recently reported to produce phosphorus ylides (4–12). In continuation of our works on the reaction between triphenylphosphine and acetylene diesters in the presence of organic N–H, O–H or C–H acids (13a–f), we herein report an efficient synthetic route to stable phosphorus ylides using three-component reaction between dialkyl acetylenedicarboxylates (DAADs), triphenylphosphine and 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thione (2).

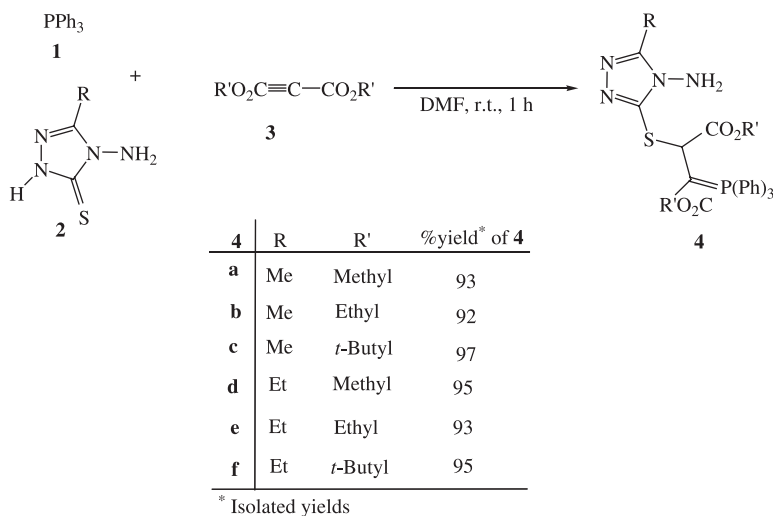
*Corresponding author. Email: mhmoslemin@yahoo.com

2. Results and discussion

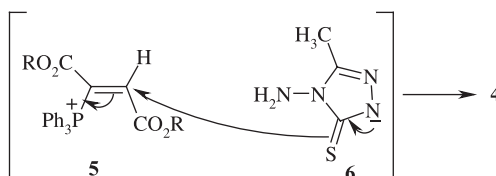
The reaction of the 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thione (**2**) with DAAD (**3**) in the presence of triphenylphosphine **1** leads to the corresponding ylide **4** in good yields (Scheme 1). The ^1H NMR spectrum of compound **4a** displays two sharp singlets (δ 3.13, 3.71 ppm) for the protons of two methoxy groups, a doubled signal for the methine proton at 5.43 ppm ($^3J_{\text{HP}} = 16$ Hz) and multiplets between 7.26 and 7.72 ppm for aromatic protons. A broad singlet was observed at 1.69 ppm that disappeared after the addition of a few drops of D_2O to CDCl_3 solution of compound **4a**. This signal is related to NH_2 protons. The ^{13}C NMR spectrum of compound **4a** showed 13 distinct signals, which is consistent with the proposed structure. The ^{31}P NMR spectrum of compound **4a** consists of one signal at 23.2 ppm. This shift is similar to those observed for other stable phosphorus ylides (*14*, *15*). The structural assignments made on the basis of the NMR spectra of compounds **4a–f** are supported by their IR spectra. The carbonyl region of the spectrum exhibits absorption bands at 1750 cm^{-1} for the ester groups and at 3300 and 3460 cm^{-1} for the NH_2 group.

It is reasonable to assume that ylide **4** results from the initial addition of triphenylphosphine to DAAD and subsequent protonation of the 1:1 adduct by the NH-acidic triazole. The positively charged ion **5** is then attacked by the triazole anion **6** to form **4** (Scheme 2).

In summary, we report herein that the three-component reaction between DAADs and triphenylphosphine by 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thiones produces functionalized phosphorus ylides in good yields. The advantages of the reported method are simple available



Scheme 1. Three-component reaction between triphenylphosphine, DAADs and 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thiones.



Scheme 2. Suggested mechanism for formation of ylides **4**.

starting materials, short reaction time, simple work-up, neutral reaction conditions and high yields.

3. Experimental

All melting points are uncorrected. Elemental analyses were performed at the Analytical Laboratory of Islamic Azad University, Yazd Branch, using a Costech ECS 4010 CHNS-O analyzer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H , ^{13}C and ^{31}P NMR spectra were recorded on a BRUKER DRX-500 AVANCE spectrometer at 500, 128.5 and 202.5 MHz, respectively. ^1H , ^{13}C and ^{31}P NMR spectra were obtained on a solution in CDCl_3 using TMS as the internal standard or 85% H_3PO_4 as the external standard. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

3.1. General procedure

A magnetically stirred solution of triphenylphosphine (2 mmol) and 4-amino-5-alkyl-2,4-dihydro-1,2,4-triazole-3-thione (2 mmol) in DMF (10 mL) was added dropwise to a mixture of DAAD (2 mmol) in DMF (3 mL) at room temperature for 2 min. The reaction mixture was then stirred for 1 h. Water (50 mL) was added and the solid was filtered off and washed with diethyl ether to give the pure product.

3.2. Dimethyl 2-(4-amino-5-methyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4a)

White powder; yield: 93%; m.p. 160–162°C. IR (KBr) (ν_{max} , cm^{-1}): 1750 (C=O), 3300, 3460 (NH_2). Analyses: Calcd. for $\text{C}_{27}\text{H}_{27}\text{N}_4\text{O}_4\text{PS}$: C, 60.66; H, 5.09; N, 10.48. Found: C, 60.4; H, 5.2; N, 10.6. MS (m/z , %): 534 (M^+ , 7).

^1H NMR (500.1 MHz, CDCl_3): δ 1.69 (2H, s, NH_2), 2.40 (3H, s, CH_3), 3.13 (3H, s, OCH_3), 3.71 (3H, s, OCH_3), 5.43 (1H, d, $^3J_{\text{PH}} = 16$ Hz), 7.26–7.72 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 9.8 (CH_3), 48.3 (OCH_3), 50.5 (d, $^1J_{\text{PC}} = 126$ Hz, C=P), 51.7 (OCH_3), 61.5 (d, $^2J_{\text{PC}} = 16$ Hz, CH), 125.3 (d, $^1J_{\text{PC}} = 94$ Hz), 127.8 ($^2J_{\text{PC}} = 12$ Hz), 132.1 (d, $^4J_{\text{PC}} = 2$ Hz), 132.8 (d, $^3J_{\text{PC}} = 10$ Hz), 146.9 (SC=N), 164.6 (NC=N), 169.5 (d, $^2J_{\text{PC}} = 12$ Hz C=O), 171.3 (d, $^3J_{\text{PC}} = 17$ Hz C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.82.

3.3. Diethyl 2-(4-amino-5-methyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4b)

White powder; yield: 92%; m.p. 152–154°C. IR (KBr) (ν_{max} , cm^{-1}): 1748 (C=O), 3295, 3485 (NH_2). Analyses: Calcd. for $\text{C}_{29}\text{H}_{31}\text{N}_4\text{O}_4\text{PS}$: C, 61.91; H, 5.55; N, 9.96. Found: C, 61.7; H, 5.4; N, 10.2. MS (m/z , %): 562 (M^+ , 6).

^1H NMR (500.1 MHz, CDCl_3): δ 0.43 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.15 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.70 (2H, s, NH_2), 1.94 (3H, s, CH_3), 3.65 (2H, d, $^3J_{\text{HH}} = 7$ Hz, OCH_2), 3.77 (2H, d, $^3J_{\text{HH}} = 7$ Hz, OCH_2), 5.44 (1H, d, $^3J_{\text{PH}} = 16$ Hz), 7.26–7.73 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 11.3 (CH_3), 13.9 and 14.2 (2 CH_3), 41.3 (d, $^1J_{\text{PC}} = 122$ Hz, C=P), 57.9 (d, $^2J_{\text{PC}} = 15$ Hz, CH), 61.7 and 62.6 (2 OCH_2), 126.1 (d, $^1J_{\text{PC}} = 91$ Hz), 128.8 ($^2J_{\text{PC}} = 12$ Hz), 132.1 (d, $^4J_{\text{PC}} = 2$ Hz), 133.7 (d, $^3J_{\text{PC}} = 10$ Hz), 147.3 (SC=N), 165.4 (NC=N), 169.8 (d, $^2J_{\text{PC}} = 12$ Hz C=O), 170.9 (d, $^3J_{\text{PC}} = 18$ Hz C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.97.

3.4. Di-*t*-butyl 2-(4-amino-5-methyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4c)

White powder; yield: 97%; m.p. 165–167°C. IR (KBr) (ν_{\max} , cm^{-1}): 1744 (C=O), 3395, 3525 (NH_2). Analyses: Calcd. for $\text{C}_{33}\text{H}_{39}\text{N}_4\text{O}_4\text{PS}$: C, 64.06; H, 6.35; N, 9.06. Found: C, 63.9; H, 6.5; N, 9.2. MS (m/z , %): 618 (M^+ , 9).

^1H NMR (500.1 MHz, CDCl_3): δ 0.83 (9H, s, *t*-Bu), 1.42 (9H, s, *t*-Bu), 1.45 (2H, s, NH_2), 2.32 (3H, s, CH_3), 5.43 (1H, d, $^3J_{\text{PH}} = 16$ Hz), 7.28–7.97 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 11.3 (CH_3), 28.7 and 28.9 (6 CH_3 of 2 *t*-Bu), 55.0 (d, $^1J_{\text{PC}} = 126$ Hz, C=P), 63.4 (d, $^2J_{\text{PC}} = 16$ Hz, CH), 76.7 and 80.5 (2 O-C(CH_3) $_3$), 127.9 (d, $^1J_{\text{PC}} = 94$ Hz), 129.5 ($^2J_{\text{PC}} = 12$ Hz), 132.9 (d, $^4J_{\text{PC}} = 2$ Hz), 134.1 (d, $^3J_{\text{PC}} = 10$ Hz), 147.2 (SC=N), 163.2 (NC=N), 169.5 (d, $^2J_{\text{PC}} = 12$ Hz C=O), 171.3 (d, $^3J_{\text{PC}} = 17$ Hz C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.78.

3.5. Dimethyl 2-(4-amino-5-ethyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4d)

White powder; yield: 95%; m.p. 124–126°C. IR (KBr) (ν_{\max} , cm^{-1}): 1751 (C=O), 3265, 3540 (NH_2). Analyses: Calcd. for $\text{C}_{28}\text{H}_{29}\text{N}_4\text{O}_4\text{PS}$: C, 61.30; H, 5.33; N, 10.21. Found: C, 61.5; H, 5.1; N, 10.4. MS (m/z , %): 548 (M^+ , 4).

^1H NMR (500.1 MHz, CDCl_3): δ 1.31 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.62 (2H, s, NH_2), 2.78 (2H, q, $^3J_{\text{HH}} = 7$ Hz, CH_2), 3.13 (3H, s, OCH_3), 3.72 (3H, s, OCH_3), 5.56 (1H, d, $^3J_{\text{PH}} = 16$ Hz), 7.34–7.72 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 10.6 (CH_3), 18.5 (CH_2), 49.8 (d, $^1J_{\text{PC}} = 126$ Hz, C=P), 52.4 (OCH_3), 52.6 (OCH_3), 62.7 (d, $^2J_{\text{PC}} = 16$ Hz, CH), 125.9 (d, $^1J_{\text{PC}} = 94$ Hz), 128.6 ($^2J_{\text{PC}} = 12$ Hz), 132.0 (d, $^4J_{\text{PC}} = 2$ Hz), 133.7 (d, $^3J_{\text{PC}} = 10$ Hz), 151.3 (SC=N), 165.9 (NC=N), 169.8 (d, $^2J_{\text{PC}} = 12$ Hz C=O), 170.1 (d, $^3J_{\text{PC}} = 17$ Hz, C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.75.

3.6. Diethyl 2-(4-amino-5-ethyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4e)

White powder; yield: 93%; m.p. 113–115°C. IR (KBr) (ν_{\max} , cm^{-1}): 1749 (C=O), 3280, 3535 (NH_2). Analyses: Calcd. for $\text{C}_{30}\text{H}_{33}\text{N}_4\text{O}_4\text{PS}$: C, 62.49; H, 5.77; N, 9.72. Found: C, 62.6; H, 5.6; N, 9.9. MS (m/z , %): 576 (M^+ , 7).

^1H NMR (500.1 MHz, CDCl_3): δ 0.45 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.16 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.32 (3H, s, CH_3), 1.73 (2H, s, NH_2), 2.77 (2H, d, $^3J_{\text{HH}} = 7$ Hz, CH_2), 3.78 (2H, d, $^3J_{\text{HH}} = 7$ Hz, OCH_2), 4.08 (2H, d, $^3J_{\text{HH}} = 7$ Hz, OCH_2), 5.62 (1H, d, $^3J_{\text{PH}} = 16$ Hz, CH), 7.31–7.62 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 10.5 (CH_3), 13.9 and 14.2 (2 CH_3), 18.5 (CH_2), 39.3 (d, $^1J_{\text{PC}} = 122$ Hz, C=P), 57.8 (d, $^2J_{\text{PC}} = 15$ Hz, CH), 61.4 and 62.9 (2 OCH_2), 126.5 (d, $^1J_{\text{PC}} = 91$ Hz), 128.7 (d, $^2J_{\text{PC}} = 12$ Hz), 132.0 (d, $^4J_{\text{PC}} = 2$ Hz), 133.8 (d, $^3J_{\text{PC}} = 10$ Hz), 151.3 (SC=N), 165.9 (NC=N), 169.7 (d, $^2J_{\text{PC}} = 12$ Hz, C=O), 171.0 (d, $^3J_{\text{PC}} = 18$ Hz, C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.81.

3.7. Di-*t*-butyl 2-(4-amino-5-ethyl-4H-1,2,4-triazol-3-yl-sulfanyl)-3-(triphenyl- λ^5 -phosphanylidene)-succinate (4f)

White powder; yield: 95%; m.p. 165–167°C. IR (KBr) (ν_{\max} , cm^{-1}): 1746 (C=O), 3390, 3517 (NH_2). Analyses: Calcd. for $\text{C}_{34}\text{H}_{41}\text{N}_4\text{O}_4\text{PS}$: C, 64.54; H, 6.53; N, 8.85. Found: C, 64.7; H, 6.7; N, 8.9. MS (m/z , %): 632 (M^+ , 6).

^1H NMR (500.1 MHz, CDCl_3): δ 0.81 (9H, s, *t*-Bu), 1.29 (3H, t, $^3J_{\text{HH}} = 7$ Hz, CH_3), 1.40 (9H, s, *t*-Bu), 1.52 (2H, s, NH_2), 2.73 (2H, q, $^3J_{\text{HH}} = 7$ Hz, CH_2), 5.49 (1H, d, $^3J_{\text{PH}} = 16$ Hz), 7.32–7.91 (15H, m, aromatic). ^{13}C NMR (125.8 MHz, CDCl_3): δ 10.3 (CH_3), 18.6 (CH_2), 28.2 and 28.7 (6 CH_3 of 2 *t*-Bu), 55.2 (d, $^1J_{\text{PC}} = 126$ Hz, C=P), 62.8 (d, $^2J_{\text{PC}} = 16$ Hz, CH), 76.4 and 80.7 (2 O–C(CH_3) $_3$), 127.3 (d, $^1J_{\text{PC}} = 94$ Hz), 129.4 ($^2J_{\text{PC}} = 12$ Hz), 132.7 (d, $^4J_{\text{PC}} = 2$ Hz), 133.8 (d, $^3J_{\text{PC}} = 10$ Hz), 147.0 (SC=N), 162.9 (NC=N), 169.7 (d, $^2J_{\text{PC}} = 12$ Hz, C=O), 171.1 (d, $^3J_{\text{PC}} = 17$ Hz, C=O). ^{31}P NMR (202.5 MHz, CDCl_3): δ 22.65.

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