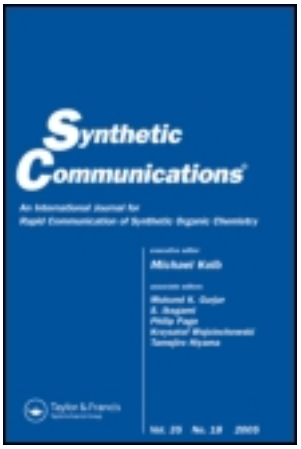

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Three-Component Reaction of Triphenylphosphine, Acetylenic Esters, and 6-Aminouracil or 6-Amino-N,N′-dimethyluracil

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THREE-COMPONENT REACTION OF TRIPHENYLPHOSPHINE, ACETYLENIC ESTERS, AND 6-AMINOURACIL OR 6-AMINO-*N,N′*-DIMETHYLURACIL

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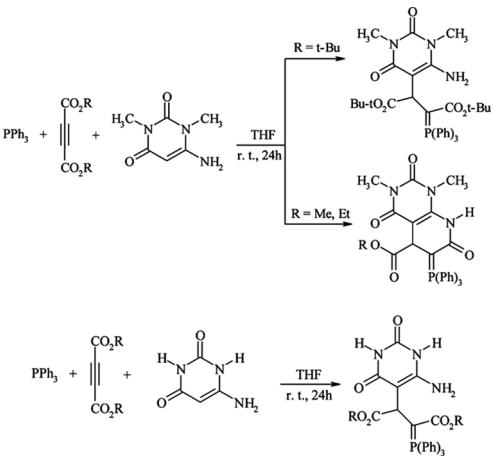
GRAPHICAL ABSTRACT



Abstract *Protonation of the reactive 1:1 intermediate produced in the reaction of acetylenic esters and triphenylphosphine by 6-aminouracil or 6-amino-*N,N′*-dimethyluracil leads to vinylphosphonium salts, which undergo Michael addition with the conjugate base of the NH acid to produce highly fanctionalized, salt-free phosphorus ylides in excellent yields.*

Keywords Acetylenic esters; 6-amino-*N,N′*-dimethyluracil; 6-aminouracil; phosphorus ylides; triphenylphosphine

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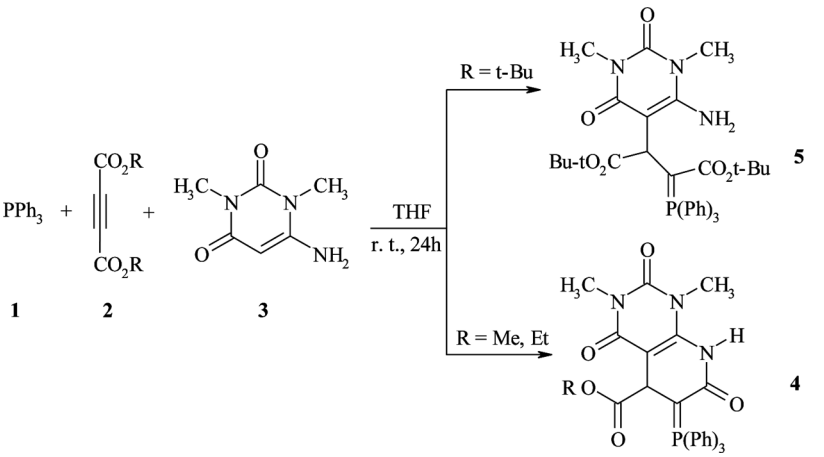
INTRODUCTION

Ylides are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products and compounds with biological and pharmacological activity.[1] Phosphorus ylides are reactive systems that take part in many reactions of value in organic synthesis.[2–8] 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.[2,3] Phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins.[2] Reaction of acetylenic esters with triphenylphosphine in the presence of an acidic hydrogen-containing organic compound has also been reported to produce phosphorus ylides.[9] In continuation of our work on the reactions between trivalent phosphorus nucleophiles and electron-deficient acetylenic compounds in the presence of organic N-H, O-H, or C-H acids,[10–21] here we report the results of our study on the reaction between triphenylphosphine and electron-deficient acetylenic esters in the presence of 6-amino-*N,N′*-dimethyluracil (Scheme 1).

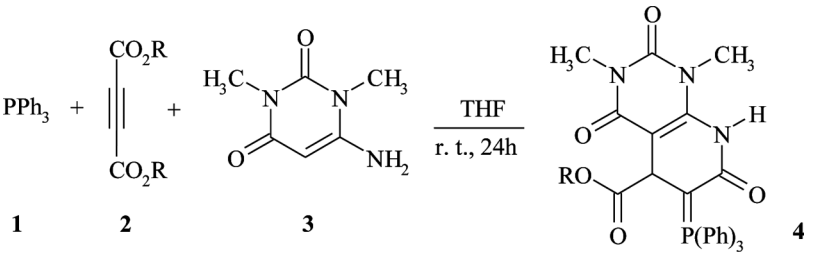
Thus the reaction between dimethyl acetylenedicarboxylate (DMAD) or diethyl acet ylenedicarboxylate (DEAD) with 6-amino-*N,N′*-dimethyl uracil in the presence of triphenylphosphine afforded alkyl-1,3-dimethyl-2,4,7-trioxo-6-(triphenyl-λ^5-phosph anylidene)-1,2,3,4,5,6,7,8-octahydropyrido[2,3-*d*]pyrimidine-5-carboxylate **4a** and **b** in good yields (Scheme 2).

The mass spectra of the ylides **4** are fairly similar and display molecular ion peaks.

The ^{1}H NMR spectrum of **4a** shows three sharp lines ($\delta = 3.06$, 3.20, and 3.15 ppm) corresponding to the protons of the methyl groups and methoxy protons respectively. There was a doubled signal for the methine proton at 3.57 ($^3J_{HP} = 10.5$ Hz). The aromatic protons resonated between 7.31 and 7.67 ppm. A broad signal was observed at $\delta = 9.32$ ppm for NH proton which disappeared after



Scheme 1. Condensation of acetylenic esters and 6-amino-*N,N′*-dimethyl uracil in the presence of triphenylphosphine.



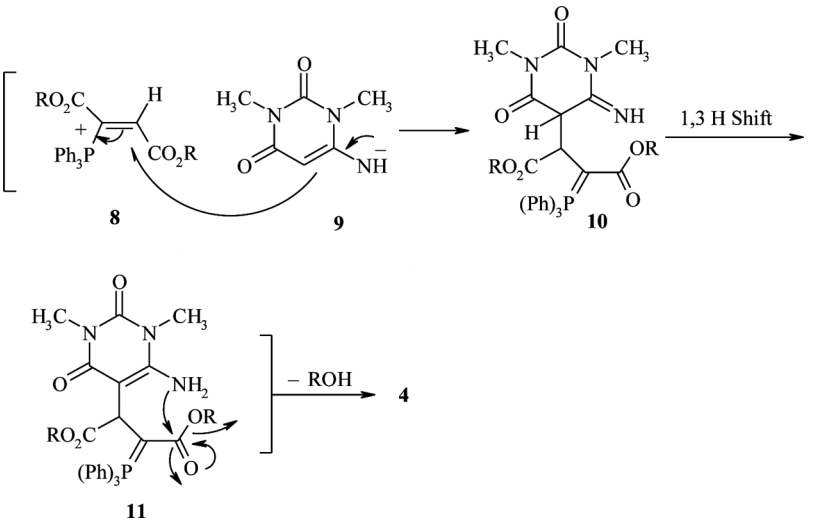
1 2 3 4

2,4	R	%Yield
a	Me	90
b	Et	85

Scheme 2. Condensation of DMAD or DEAD and 6-amino-*N,N′*-dimethyl uracil in the presence of triphenylphosphine.

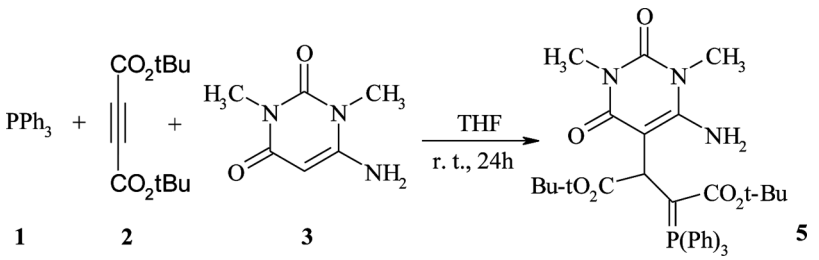
a few drops of D_2O were added to dimethylsulfoxide (DMSO) solution of **4a**. The ^{31}P NMR spectrum of compound **4a** consists of one signal at 20.15. This shift is similar to those observed for other stable phosphorus ylides.[22,23] 13 C NMR spectrum of compound **4a** shows 15 distinct signals, which is consistent with the proposed structure. The infrared (IR) spectrum of compound **4a** also supported the suggested structure, and strong absorption bands were observed at 3025, 1719, and 1690 cm^{-1} respectively for the NH and carbonyl groups.

On the basis of the well-established chemistry of trivalent phosphorus nucleophiles,[2,3,11] it is reasonable to assume that ylides **4** result from the initial addition of triphenylphosphine to the acetylenic ester and subsequent protonation of the 1:1 adduct by 6-amino-*N,N′*-dimethyluracil. Then, the positively charged ion intermediate



8 9 10 11 4

Scheme 3. Suggested mechanism for formation of ylides **4**.



Scheme 4. Condensation of DTAD and 6-amino-*N,N′*-dimethyl uracil in the presence of triphenylphosphine.

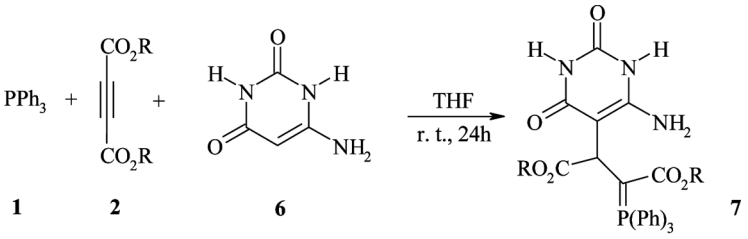
8 is attacked by the conjugate anion of 6-amino-*N,N′*-dimethyluracil **9** to form the intermediate **10**. Compound **11**, which followed by addition of the amino to carbonyl group, then loses an alkoxide ion and cyclizes to phosphorane **4** (Scheme 3).

Under similar conditions as these reactions, triphenylphosphine reacted with DTAD in the presence of 6-amino-*N,N′*-dimethyluracil to produce phosphorane **5** in 73% yield (Scheme 4).

NMR spectra of compound **5** in d_6-DMSO show that this compound, in contrast to ylides **4a** and **4b**, is stable and does not cyclize to phosphorane **4** even at refluxing acetonitrile because t-butyl group is a hindrance. The three-component reaction of acetylenic esters, triphenylphosphine, and 6-aminouracil also leads to the phosphorus ylides **7a** and **b** (Scheme 5).

The mass spectra of the ylides **7** are fairly similar and display molecular ion peaks.

The ^{1}H NMR spectrum of compound **7a** showed two singlets at 3.05 and 3.65 ppm for methoxy protons. There was a doubled signal for the methine proton at 4.82 ($^3J_{HP} = 10.5$ Hz). The aromatic protons resonated between 7.60 and 7.90 ppm. A broad singlet with the integral of 2 was observed at $\delta = 10.98$ ppm for NH_2 protons, and two single signals we observed at 7.59 and 7.89 ppm for NH protons that disappeared after addition of a few drops of D_2O to the d_6-DMSO solution of compound **7a**. The ^{31}P NMR spectrum of compound **7a** displays two signals at 24.29 ppm. ^{13}C NMR spectrum of compound **7a** showed 14 distinct signals in accordance with the proposed structure.



2,7	R	%Yield
a	Me	88
b	t-Bu	70

Scheme 5. Three-component reaction of triphenylphosphine, acetylenic esters, and 6-aminouracil.

In summary, phosphorus ylides may be prepared by a simple, one-pot, three-component reaction of acetylenic esters, triphenylphosphine, and 6-amino-*N,N′*-dimethyluracil or 6-aminouracil. The present method has the advantage that not only the reaction is performed under neutral conditions but also the substances can be mixed without any activation or modification.

EXPERIMENTAL

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed at the analytical laboratory of the Science and Research Unit of Islamic Azad University. Mass spectra were recorded on a Finnigan-Mat 8430 mass spectrometer operating at an ionization potential of 70 eV. Infrared (IR) spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on a Bruker DRX-500 Avance spectrometer in d_6-DMSO using tetramethylsilane (TMS) as internal standard or 85% H_3PO_4 as external standard. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General Procedure for Preparation of Compounds 4a, 4b, 5, 7a, and 7b

To a magnetically stirred solution of uracil derivatives **3** or **6** (1 mmol) in 10 mL tetrahydrofuran (THF) and triphenylphosphine **1** (1 mmol) was added a mixture of acetylenic esters **2** (1 mmol) in 2 mL THF at room temperature. The reaction mixture was then allowed to stir for 24 h. The solvent was evaporated at reduced pressure, and the residue was purified by silica-gel column chromatography using hexane–ethyl acetate as eluent. The solvent was removed under reduced pressure to afford the product.

Methyl 1,3-dimethyl-2,4,7-trioxo-6-(triphenyl-λ^5-phosphanylidene)-1,2,3,4,5,6,7,8-octahydro pyrido[2,3-*d*]pyrimidine-5-carboxylate (4a). White power; yield (90%); mp = 173–175 °C. IR (KBr) (ν_{max} cm^{-1}): 3025 (NH), 1719, and 1690 (C=O). Analyst: calcd. for $C_{29}H_{26}N_3O_5P$: C, 66.03; H, 4.97; N, 7.97%. Found: C, 65.87; H, 5.20; N, 8.06%, MS (*m/z*, %): 527 (M^+, 5). ^{1}H NMR (500 MHz, d_6-DMSO): δ 3.06 and 3.20 (6H, 2 s, $2NCH_3$), 3.15 (3H, s, OCH_3), 3.57 (1H, d, $^{3}J_{HP} = 10.5$ Hz, CH), 7.31–7.67 (15H, m, aromatic), 9.32 (1H, br, NH) ppm. ^{13}C NMR (125.7 MHz, d_6-DMSO): δ 29.71 and 29.90 ($2NCH_3$), 38.40 (CH, d, $^{2}J_{PC} = 14.9$ Hz), 40.44 (C=P, d, $^{1}J_{PC} = 125$ Hz), 51.34 (OCH_3), 84.41 (d,$^{3}J_{PC} = 12$ Hz, C=C-NH), 124.81 (C^{ipso}, d, $^{1}J_{PC} = 91.0$ Hz), 129.38 (C^{meta}, d, $^{3}J_{PC} = 11.8$ Hz), 132.90 (C^{paea}), 133.88 (C^{ortho}, d, $^{2}J_{PC} = 9.9$ Hz), 148.17 (C=C-NH), 151.17 and 160.76 (2C=O), 165.73 (d, $^{2}J_{PC} = 15.3$ Hz, C=O), 174.83 (C=O) ppm.^{31}P NMR (d_6-DMSO): δ = 20.15 ppm.

Ethyl 1,3-dimethyl-2,4,7-trioxo-6-(triphenyl-λ^5-phosphanylidene)-1,2,3,4,5,6,7,8-octahydro pyrido[2,3-*d*]pyrimidine-5-carboxylate (4b). White power; yield (85%); mp = 188–190 °C. IR (KBr) (ν_{max} cm^{-1}): 3055 (N-H), 1725, 1692 (C=O). Analyst: calcd. for $C_{30}H_{28}N_3O_5P$: C, 66.54; H, 5.21; N, 7.76%. Found: C, 66.75; H, 5.40; N, 7.59%, MS (*m/z*, %): 541 (M^+, 3). ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.73 (3 H, t,$^{3}J_{HH} = 7.1$ Hz, CH_3), 3.08 and 3.32 (6H, 2 s, $2NCH_3$), 3.55 (1H, d, $^{3}J_{HP} = 10.5$ Hz, CH), 3.77 (2H, m, $^{3}J_{HH} = 7$ Hz, OCH_2), 7.32–7.70 (15H,

m, aromatic), 9.18 (1H, br, NH) ppm. ^{13}C NMR (125.7 MHz, d_6-DMSO): δ 13.88 (CH_3), 27.95 and 30.17 ($2NCH_3$), 38.43 (CH, d, $^2J_{PC} = 14.9$ Hz), 41.32 (C=P, d, $^1J_{PC} = 125$ Hz), 60.40 (OCH_2), 84.40 (d,$^3J_{PC} = 12$ Hz, C=C-NH), 125.12 (C^{ipso}, d, $^1J_{PC} = 91.0$ Hz), 129.36 (C^{meta}, d, $^3J_{PC} = 11.8$ Hz), 132.91 (C^{paea}), 133.67 (C^{ortho}, d, $^2J_{PC} = 9.9$ Hz), 148.33 (C=C-NH), 151.27 and 160.78 (2C=O), 165.42 (d, $^2J_{PC} =$ 15.3 Hz, C=O), 174.89 (C=O) ppm.^{31}P NMR (d_6-DMSO): δ = 20.12 ppm.

Di-*tert*-butyl-2-(6-amino-1,3 dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-pyridin-5-yl)-3-(triphenyl-λ^5-phosphanylidene)succinate (5). White power; yield (73%); mp = 179–181 °C. IR (KBr) (ν_{max} cm^{-1}): 3345 (NH_2), 1722, 1605 (C=O). Analyst: calcd. for $C_{36}H_{42}N_3O_6P$: C, 67.17; H, 6.58; N, 6.53%. Found: C, 66.90; H, 6.71; N, 6.64%. MS (*m*/*z*, %): 643 (M^+, 8). ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.84 and 1.45 (18H, 2 s, 2 t-Bu), 3.06 and 3.38 (6H, 2 s, $2NCH_3$), 4.15 (1H, d, $^3J_{HP} = 18.8$ Hz, Hz, CH), 7.20 (2H, br, NH_2), 7.48–7.67 (15H, m, aromatic), ^{13}C NMR (125.7 MHz, d_6-DMSO): δ 25.99 and 28.78 ($6CH_3$), 28.24 and 29.00 ($2NCH_3$), 38.60 (CH, d, $^2J_{PC} = 13.8$ Hz), 40.90 (C=P, d, $^1J_{PC} = 125$ Hz), 77.06 and 79.35 (2C), 89.04 (C=C-NH_2), 127.52 (C^{ipso}, d, $^1J_{PC} = 90.2$ Hz), 129.38(C^{meta}, d, $^3J_{PC} = 11.6$ Hz), 132.03 (C^{para}), 133.88 (C^{ortho}, d, $^2J_{PC} = 9.9$ Hz), 151.64 (C=C-NH_2), 152.74 and 161.25 (C=O), 170.95 (C=O, d, $^2J_{PC} = 15.3$ Hz), 173.03 (C=O, d, $^3J_{PC} = 11.3$ Hz) ppm. ^{31}P NMR (d_6-DMSO): δ 20.85 ppm.

Dimethyl-2-(6-amino-2,4-dihydroxo-pyridin-5-yl)-3-(triphenyl-λ^5-phosphanylidene)succinate (7a). White power; yield (88%); mp 185–187 °C; IR (KBr) (ν_{max} cm^{-1}): 3345 (NH_2), 1733, 1688 (C=O). Analyst: calcd. for $C_{28}H_{26}N_3O_6P$: C, 63.27; H, 4.93; N, 7.91%. Found: C, 63.39; H, 5.08; N, 7.78%. MS (*m*/*z*, %): 531 (M^+, 5). ^{1}H NMR (500 MHz, d_6-DMSO): δ 3.05 and 3.65 (6H, 2 s, $2OCH_3$), 4.82 (1H, d, $^3J_{HP} = 16$ Hz, CH), 7.60 (1H, s, NH), 7.89 (1H, s, NH), 7.60–7.90 (15H, m, aromatic), 10.98 (2H, s, NH_2).^{13}C NMR (125.7 MHz, d_6-DMSO): δ 40.08 (C=P, d, $^1J_{PC} = 125$ Hz), 50.04 and 52.56 ($2OCH_3$), 58.48 (CH, d, $^2J_{PC} = 16.2$ Hz), 100.77 (C=C-NH_2), 125.35 (C^{ipso}, d, $^1J_{PC} = 91.2$ Hz), 129.30 (C^{meta}, d, $^3J_{PC} = 11.3$ Hz), 132.72 (C^{para}), 133.07 (C^{ortho}, d, $^2J_{PC} = 9.0$ Hz), 143.07 (C=C-NH_2), 150.32 and 150.39 (2C=O), 169.54 (C=O, d, $^3J_{PC} = 12.5$ Hz), 170.67(C=O) ppm. ^{31}P NMR (d_6-DMSO): δ 24.29 ppm.

Di-*tert*-butyl-2-(6-amino-2,4-dihydroxo-pyridin-5-yl)-3-(triphenyl-λ^5-phosphany lidene)succinate (7b). White power; yield (70%); mp 180–182 °C; IR (KBr) (ν_{max} cm^{-1}): 3345 (NH_2), 1722, 1692 (C=O). Analyst: calcd. for $C_{34}H_{38}N_3O_6P$: C, 66.33; H, 6.22; N, 6.83%. Found: C, 66.42; H, 6.07; N, 6.70%. MS (*m*/*z*, %): 615 (M^+, 7). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.43 and 1.45 (18H, 2 s, $6CH_3$), 4.72 (1H, d, $^3J_{HP} = 16.0$ Hz, CH), 7.59 (1H, s, NH), 7.48–7.67 (15H, m, aromatic), 7.91 (1H, s, NH), 11.02 (1H, s, NH_2). ^{13}C NMR (125.7 MHz, d_6-DMSO): δ 28.09 and 28.21 ($6CH_3$), 40.10 (C=P, d, $^1J_{PC} = 125$ Hz), 59.37 (CH, d, $^2J_{PC} = 16.8$ Hz,), 77.33 and 81.04 (2C), 100.65 (C=C-NH_2), 126.35 (C^{ipso}, d, $^1J_{PC} = 91.5$ Hz), 129.33 (C^{meta}, d, $^3J_{PC} = 11.1$ H,), 130.78 (C^{para}), 131.75 (C^{ortho}, d,$^2J_{PC} = 9.1$ Hz), 150.73 (C=C-NH_2), 151.95 and 164.75 (2C=O), 169.12 (C=O, d, $^2J_{PC} = 12.8$ Hz), 170.83 (C=O) ppm. ^{31}P NMR (d_6-DMSO): δ 24.08 ppm.

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