Diastereoselective synthesis of functionalised stable phosphorus ylides

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Stable phosphorus ylides were obtained in excellent yields from the reaction between electron-deficient acetylenic compounds, and 2-cyano-N-(aryl) or alkyl acetamide in the presence of triphenylphosphine in dichloromethane.

Keywords: acetylenic esters, triphenylphosphine, phosphorus ylides, 2-cyano-*N*-aryl acetamide, 2-cyano-*N*-alkyl acetamide, diastereoselective synthesis

Organic phosphorus compounds are widely used in organic chemistry.[1] An important group of this class is phosphorus ylides. Although phosphorus ylides have few direct end uses, they are important reagents in the synthesis of organic compounds such as allenes,[2–7] cyclic, and heterocyclic compounds.[8–10] The utility of metallated phosphorus ylides in organometallic chemistry has been well documented because stabilised phosphorus ylides are versatile ligands for heavy metal ions.[11,12] These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually prepared from the phosphine and an alkyl halide.[12–22] Among other methods, phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins.[23–30]

In continuation of our work on the reaction between trivalent phosphorus nucleophiles and electron-deficient acetylenic compounds in the presence of organic acidic compounds,[29–31] we now report an efficient synthetic route to stable phosphorus ylides using triphenylphosphine, dialkyl acetylenedicarboxylates and 2-cyano-*N*-(aryl) or alkyl acetamide.

The reaction of the 2-cyano-*N*-(aryl) or alkyl acetamide **3** with the dialkyl acetylenedicarboxylates **2** in the presence of triphenylphosphine **1** leads to the corresponding ylide **4a–f** in good yields (Scheme 1).

Because of the existence of two stereogenic centres in the products, mixtures of two diastereomers were expected but the ^{1}H NMR spectra of product **4** showed the existence of only one isomer. Comparing the coupling constant of two methine hydrogens with those for similar compounds reported previously,[32] we concluded that the obtained isomer is (*S,S*) **6** and its enantiomers (Fig. 1).

The structures of compounds **4a–f** were confirmed from their IR, ^{1}H, ^{13}C, and ^{31}P NMR spectra. The IR spectrum of **4a** showed absorption bands at 1735, 1677 cm^{-1} for the carbonyl

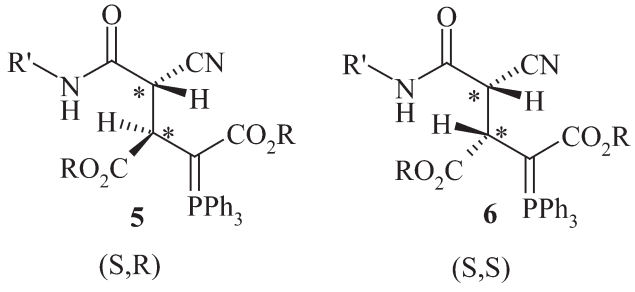
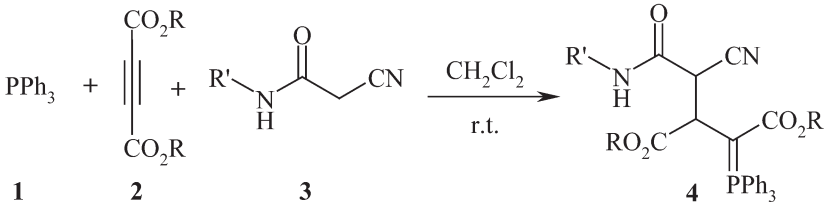


Fig. 1



4	R	R'	Yield* (%)
a	Me	Ben	88
b	Et	Ben	80
c	Me	*P*-CH_3-C_6H_4	85
d	Et	C_6H_5	80
e	Me	n-bu	86
f	Et	n-bu	82

* Isolated yields

Scheme 1

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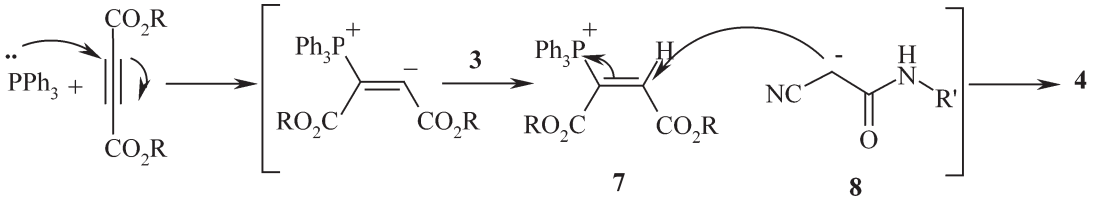

Scheme 2

groups at 2235 for C≡N group, and a NH group at 3320 cm^{-1}. The ^1H NMR spectrum of **4a** exhibited two sharp singl signals (δ = 3.09, 3.61 ppm) recognised as arising from two OCH_3 groups. The doublet of doublets at δ = 3.12 and the doublet at δ = 5.03 were attributed to CHC*H*C=P and C*H*CH=P protons respectively ($^3J_{HH}$ = 10.6 Hz and $^3J_{PH}$ = 16.5 Hz). There are two doublet of doublets at δ = 4.41, 4.64 ($^2J_{HH}$ = 15.2 H_Z, $^3J_{HH}$ = 5.2 H_Z) which were attributed to CH_2 group (diastereotopic protons). A broad signal at 6.32 ppm was recognised as arising from the NH group. The aromatic protons show multiplets at δ = 7.27–7.74 ppm. The ^{31}P NMR spectrum of compound **4a** displays a signal at 24.49 ppm. The ^{13}C NMR spectrum of **4a** exhibits 17 distinct resonances, which are in agreement with the proposed structure.

It is reasonable to assume that compounds **4** result from the initial addition of triphenylphosphine to acetylenic ester **2** and subsequent protonation of the 1:1 adduct by 2-cyano-*N*-(aryl) or alkyl acetamide **3** Then, the positively charged ion **7** is attacked by the anion of 2-cyano-*N*-(aryl) or alkyl acetamide **8** to form ylide **4** (Scheme 2).

Conclusion

In conclusion, the present method carries the advantage that, not only is the reaction performed under neutral conditions, but the substances can be mixed without any activation or modification. The procedure described here provides an acceptable one-pot method for the diastereoselective synthesis of phosphorus ylides derivatives.

Experimental

All melting points were determined on an Electrothermal 9100 apparatus and are uncorrected. Elemental Analysis were performed using a Costech ECS 4010 CHNS-O analyzer at the analytical laboratory of Islamic Azad University, Yazd Branch. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionisation 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 BRUKER DRX-300 AVANCE spectrometer at 300.1, 75.46, and 121.49 MHz, respectively. ^1H, ^{13}C and ^{31}P NMR spectra were obtained in $CDCl_3$ solution using TMS as internal standard (^1H, ^{13}C) or 85% H_3PO_4 as external standard (^{31}P). The chemicals used in this study were purchased from Fluka and were used without further purification.

General procedure

To a magnetically stirred solution of 2-cyano-*N*-(aryl)or alkyl acetamide (1 mmol) and dialkyl acetylenedicarboxylate (1 mmol) in CH_2Cl_2 (10 mL) was added drop wise a mixture of triphenylphosphine (1 mmol) in CH_2Cl_2 (3 mL) at room temperature over 2 min. The reaction mixture was then stirred for 3 h. The solvent was evaporated and the residue was crystallised from ethyl acetate–hexane mixture.

Spectral data

Dimethyl 2-[benzylcarbamoyl(cyano)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate (**4a**): Yield: 88%; colourless crystals; m.p. 201–202 °C. IR (KBr) (ν_{max}, cm^{-1}): 3320 (NH), 2235(C≡N), 1735, 1677 (C=O). Anal. Calcd for $C_{34}H_{31}N_2O_5P$: C, 70.58; H, 5.40; N, 4.84. Found: C, 70.76; H, 5.62; N, 4.65%. MS (*m/z*, %) : 578 (M^+, 9). ^1H NMR ($CDCl_3$): δ = 3.09 (s, 3H, OCH_3), 3.12 (dd,1H, $^3J_{PH}$ = 16.5 H_Z, $^3J_{HH}$ = 10.6 H_Z, CHC*H*C=P), 3.61 (s, 3H, OCH_3), 4.41 (dd, 1H, CH_2, $^2J_{HH}$ = 15.2 H_Z, $^3J_{HH}$ = 5.2 H_Z), 4.64 (dd,1H, CH_2, $^2J_{HH}$ = 15.2 H_Z, $^3J_{HH}$ = 5.2 H_Z), 5.03 (d, 1H, $^3J_{HH}$ = 10.8 H_Z, C*H*CHC=P), 6.32 (br, 1H, NH), 7.27–7.74 (m, 20H, arom) ppm.^{13}C NMR ($CDCl_3$): δ = 39.08 (d,$^3J_{PC}$ = 10.8 H_Z, *C*HCHC=P), 40.44 (d, $^1J_{PC}$ = 125 H_Z, C=P), 43.54 (CH_2), 45.50 (d,$^2J_{PC}$ = 13 H_Z,CH*C*HC=P), 50.11, 51.85 ($2OCH_3$), 118.71 (*C*≡N), 126.42 (d, $^1J_{PC}$ = 92 H_Z, C ^{ipso}), 128.65 (d,$^3J_{PC}$ = 12.0 H_Z, C ^{meta}), 132.15 (d, $^4J_{PC}$ = 2.4 H_Z, C ^{para}), 133.99 (d, $^2J_{PC}$ = 9 H_Z, C ^{ortho}), 127.18, 127.52, 128.79, 138.09 (C, arom), 165.10(C=O), 169.82 (d, $^2J_{PC}$ = 12.15 H_Z, C=O), 175.34 (d, $^3J_{PC}$ = 10.32 H_Z, C=O) ppm. ^{31}P NMR ($CDCl_3$): δ = 24.49 ppm.

Diethyl 2-[benzylcarbamoyl(cyano)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate (**4b**): Yield: 80%; colourless crystals; m.p. 179–182 °C. IR (KBr) (ν_{max}, cm^{-1}): 3405 (NH), 2235(C≡N), 1722, 1676 (C=O). Anal. Calcd for $C_{36}H_{35}N_2O_5P$: C, 71.27; H, 5.82; N, 4.62. Found: C, 71.41; H, 5.72; N, 4.78%. MS (*m/z*, %): 606 (M^+, 11). ^1H NMR ($CDCl_3$): δ = 0.44 (t, 3 H, $^3J_{HH}$ = 6.8 Hz, OCH_2CH_3), 1.17 (t, 3H, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 3.08 (dd, 1H, $^3J_{PH}$ = 16.4 H_Z, $^3J_{HH}$ = 10.4 H_Z, CHC*H*C=P), 3.63–3.68 (m, 2H, OCH_2CH_3), 4.13–4.20 (m, 2H, OCH_2CH_3), 4.31 (dd, 1H, CH_2, $^2J_{HH}$ = 15.6 H_Z, $^3J_{HH}$ = 5.2 H_Z), 4.64 (dd, 1H, CH_2, $^2J_{HH}$ = 15.6 H_Z, $^3J_{HH}$ = 5.2 H_Z), 4.99 (d,1H, $^3J_{HH}$ = 10.8 H_Z, C*H*CHC=P), 6.21 (br, 1H, NH), 7.17–7.75 (m, 20H, arom) ppm.^{13}C NMR ($CDCl_3$): δ =13.81, 14.05 ($2OCH_2CH_3$), 39.12 (d,$^3J_{PC}$ = 10.8 H_Z, *C*HCHC=P), 40.01 (d, $^1J_{PC}$ = 125 H_Z, C=P), 43.56 (CH_2), 45.43 (d,$^2J_{PC}$ = 14 H_Z, CH*C*HC=P), 58.10, 60.96 ($2OCH_2CH_3$), 119.20 (*C*≡N), 125.62 (d, $^1J_{PC}$ = 85 H_Z, C ^{ipso}), 128.65 (d,$^3J_{PC}$ = 12.0 H_Z,C ^{meta}), 132.15 (d, $^4J_{PC}$ = 3.0 H_Z,C ^{para}), 134.11 (d, $^2J_{PC}$ = 9 H_Z, C ^{ortho}), 127.12, 127.23, 129.05, 138.09 (C,arom), 165.30 (C=O), 169.31 (d, $^2J_{PC}$ = 11.45 H_Z, C=O), 172.64 (d, $^3J_{PC}$ = 10.11 H_Z, C=O) ppm. ^{31}P NMR ($CDCl_3$): δ = 24.57 ppm.

Dimethyl2-[cyano(-p-tolylcarbamoyl)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate (**4c**): Yield: 85%; colourless crystals; m.p. 197–200 °C. IR (KBr) (ν_{max}, cm^{-1}): 3265 (NH), 2235(C≡N), 1734, 1682, (C=O). Anal. Calcd for $C_{34}H_{31}N_2O_5P$: C, 70.58; H, 5.40; N, 4.84. Found: C, 70.69; H, 5.32; N, 4.97%. MS (*m/z*, %): 578 (M^+, 12). ^1H NMR ($CDCl_3$): δ = 2.41 (s, 3H, CH_3), 3.03 (s, 3H, OCH_3), 3.71 (s, 3H, OCH_3), 3.16 (m, 1H, CHC*H*C=P), 5.18 (d, 1H, $^3J_{HH}$ = 10.4 H_Z, C*H*CHC=P), 7.06–7.80 (m, 19H, arom), 8.80 (br, 1H, NH) ppm.^{13}C NMR ($CDCl_3$): δ = 21.04 (*C*H_3), 39.08 (d,$^3J_{PC}$ = 10.8 H_Z, *C*HCHC=P), 40.44 (d, $^1J_{PC}$ = 125 H_Z, C=P), 45.50 (d,$^2J_{PC}$ = 13 H_Z, CH*C*HC=P), 48.88, 52.00 ($2OCH_3$), 119.81(*C*≡N), 126.42 (d, $^1J_{PC}$ = 92 H_Z, C ^{ipso}), 128.65 (d,$^3J_{PC}$ = 12.0 H_Z, C ^{meta}), 132.35 (d, $^4J_{PC}$ = 2.2 H_Z, C ^{para}), 133.99 (d, $^2J_{PC}$ = 10 H_Z, C ^{ortho}), 127.18, 127.52, 129.45, 136.46 (C,arom), 161.40 (C=O), 163.12 (d, $^2J_{PC}$ = 12.09 H_Z, C=O), 173.34 (d, $^3J_{PC}$ = 10.12 H_Z, C=O) ppm. ^{31}P NMR ($CDCl_3$): δ = 25.14 ppm.

Diethyl 2-[cyano(phenylcarbamoyl)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate (**4d**): Yield: 80%; colourless crystals; m.p. 195–198 °C. IR (KBr) (ν_{max}, cm^{-1}): 3315 (NH), 2230(C≡N) 1731, 1677 (C=O). Anal. Calcd for $C_{35}H_{33}N_2O_5P$: C, 70.93; H, 5.61; N, 4.73. Found: C, 70.81; H, 5.49; N, 4.82%. MS(*m/z*, %): 592 (M^+, 9). ^1H NMR ($CDCl_3$): δ = 0.38 (t, 3H, $^3J_{HH}$ = 6.8 Hz, OCH_2CH_3), 1.18 (t, 3H, $^3J_{HH}$ = 6.8 Hz, OCH_2CH_3), 3.07 (dd, 1H, $^3J_{PH}$ = 16.4 H_Z, $^3J_{HH}$ = 10.4H_Z, CHC*H*C=P), 3.67–3.75 (m, 2H, OCH_2CH_3), 4.18–4.20 (m, 2H, OCH_2CH_3), 5.24 (d,1H, $^3J_{HH}$ = 10.4 H_Z, C*H*CHC=P), 7.21–7.64 (m, 20H, arom), 9.08 (br, 1H, NH) ppm. ^{13}C NMR ($CDCl_3$): δ =13.80, 14.07 ($2OCH_2CH_3$), 40.56 (d,$^3J_{PC}$ = 6 H_Z, *C*HCHC=P), 40.91 (d, $^1J_{PC}$ = 125 H_Z, C=P), 45.43 (d,$^2J_{PC}$ = 14 H_Z, CH*C*HC=P), 58.28, 61.22 ($2OCH_2CH_3$), 118.71 (*C*≡N), 126.02 (d, $^1J_{PC}$ = 92 H_Z, C ^{ipso}), 128.48 (d,$^3J_{PC}$ = 12.0 H_Z, C ^{meta}), 132.09 (d, $^4J_{PC}$ = 2.0 H_Z, C ^{para}), 133.95 (d, $^2J_{PC}$ = 10 H_Z, C ^{ortho}), 120.08, 124.22, 129.02, 138.48 (C, arom), 163.67 (C=O), 169.97 (d, $^2J_{PC}$ = 10.32 H_Z, C=O), 172.51 (d, $^3J_{PC}$ = 10.21 H_Z, C=O) ppm. ^{31}P NMR ($CDCl_3$): δ = 25.69 ppm.

Dimethyl 2-[butylcarbamoyl(cyano)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate(**4e**): Yield: 86%; colourless crystals; m.p. 201–202 °C. IR (KBr) (ν_{max}, cm^{-1}): 3310 (NH), 2235(C≡N), 1740,

1680 (C=O). Anal. Calcd for $C_{31}H_{33}N_2O_5P$: C, 68.37; H, 6.11; N, 5.14. Found: C, 68.22; H, 6.23; N, 5.31%. MS (*m/z*, %) : 544 (M^+, 7). ^{1}H NMR ($CDCl_3$): δ = 0.92 (t, 3H, $^3J_{HH}$ = 7 Hz, C_3H_6-C*H*$_3$), 1.21–1.50 (m, 4H, N-CH_2-C_2H_4-CH_3), 3.06 (s, 3H, OC*H*$_3$), 3.39–3.49 (m, 2H, N-C*H*$_2$), 3.55 (m, 1H, CHC*H*C=P), 3.70 (s, 3H, OCH_3), 4.86 (d, 1H, $^3J_{HH}$ = 10.5 H_Z, C*H*CHC=P), 6.56 (br, 1H, NH), 7.35–7.78 (m, 15H, arom) ppm.^{13}C NMR ($CDCl_3$): δ =13.81, 20.14, 31.26 (3C, N-CH_2-*C*$_3$H$_7$), 39.30 (d,$^3J_{PC}$ = 6.8 H_Z, *C*HCHC=P), 39.90 (N-*C*H_2), 40.58 (d, $^1J_{PC}$ = 117 H_Z, C=P), 45.30 (d, $^2J_{PC}$ = 13.4 H_Z, CH*C*HC=P), 49.10, 51.87 (2OCH_3), 118.9 (*C*≡N), 126.52 (d, $^1J_{PC}$ = 88 H_Z, C ^{ipso}), 128.52 (d,$^3J_{PC}$ = 12.5 H_Z, C ^{meta}), 132.21 (d, $^4J_{PC}$ = 2.1 H_Z, C ^{para}), 133.99 (d, $^2J_{PC}$ = 10 H_Z, C ^{ortho}), 165.20 (C=O), 169.85 (d, $^2J_{PC}$ = 12.15 H_Z, C=O), 173.21 (d, $^3J_{PC}$ = 10.32 H_Z, C=O) ppm.^{31}P NMR ($CDCl_3$): δ = 25.29 ppm.

Diethyl 2-[butylcarbamoyl(cyano)methyl]-3-(triphenyl-λ^5-phosphanylidene)succinate (**4f**)*:* Yield: 82%; colourless crystals; m.p. 205–207 °C. IR (KBr) (ν_{max}, cm^{-1}): 3270(NH), 2235(C≡N), 1729, 1684 (C=O). Anal. Calcd for $C_{33}H_{37}N_2O_5P$: C, 69.22; H, 6.51; N, 4.89. Found: C, 69.41; H, 6.38; N, 4.78%. MS (*m/z*, %) : 572 (M^+, 7). ^{1}H NMR ($CDCl_3$): δ = 0.43 (t, 3H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 0.92 (t, 3H, $^3J_{HH}$ = 6.5 Hz, C_3H_6-C*H*$_3$), 1.20 (t, 3H, $^3J_{HH}$ = 6.5 Hz, OCH_2CH_3), 1.29–1.50 (4H, m, N-CH_2-*C*$_3$H$_7$), 3.03 (2H, m, OC*H*$_2$$CH_3$), 3.45–3.49 (m, 2H, N-C*H*$_2$), 3.63 (m, 1H, CHC*H*C=P), 4.16 (m, 2H, OC*H*$_2$$CH_3$), 4.89 (d, 1H,$^3J_{HH}$ = 10 H_Z, C*H*CHC=P), 6.56 (br, 1H, NH), 7.48–7.78 (m, 15H, arom) ppm.^{13}C NMR ($CDCl_3$): δ =13.84, 14.07 (2OCH_2*C*H_3), 13.80, 20.16, 31.33 (3C, N-CH_2-*C*$_2$H$_4$-*C*H$_3$), 39.33 (d,$^3J_{PC}$ = 6 H_Z, *C*HCHC=P), 39.88 (N-*C*H_2), 40.10 (d, $^1J_{PC}$ = 128 H_Z, C=P), 45.37 (d, $^2J_{PC}$ = 14 H_Z, CH*C*HC=P), 57.84, 61.03 (2OCH_3), 119.04 (*C*≡N), 126.52 (d, $^1J_{PC}$ = 92 H_Z, C ^{ipso}), 128.52 (d,$^3J_{PC}$ = 12.5 H_Z, C ^{meta}), 132.11 (d, $^4J_{PC}$ = 2.1 H_Z, C ^{para}), 134.06 (d, $^2J_{PC}$ = 10 H_Z, C ^{ortho}), 165.04 (C=O), 169.35 (d, $^2J_{PC}$ = 13H_Z, C=O), 172.60 (d, $^3J_{PC}$ = 10.21 H_Z, C=O) ppm. ^{31}P NMR ($CDCl_3$): δ = 25.32 ppm.

Received 29 August 2010; accepted 23 November 2010
Paper 1000324 doi: 10.3184/174751911X556747
Published online: 21 January 2011

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