Three-component reaction between trivalent phosphorus nucleophiles, dialkyl acetylenedicarboxylates and (2,4-dinitrophenyl) acetic acid

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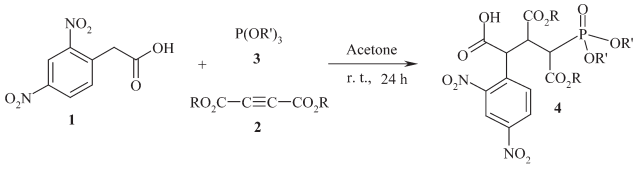
A three-component reaction between dialkyl acetylenedicarboxylates (DAADs) and phosphites in the presence of (2,4-dinitrophenyl)acetic acid leads to dialkyl 2-(dialkoxyphosphoryl)-3-[3-acetyl-2-(2,4-dinitrophenyl)-4-oxosuccinates in excellent yields. A similar reaction with triphenylphosphine, instead of phosphites, produces the stabilised phosphoranes in good yields.

Keywords: (2,4-dinitrophenyl)acetic acid, dialkyl acetylenedicarboxylate, phosphites, stereoselective synthesis, phosphorus ylides, triphenylphosphine.

Phosphorus ylides take part in many valuable reactions in organic synthesis.[1–6] Phosphorus ylides are usually prepared by treatment of a phosphonium salt with a base and the salts are usually prepared from the phosphine and an alkyl halide.[2] Phosphonium salts can also prepared by Michael addition of phosphine to activated olefins.[2] Michael addition of phosphorus(III) compounds such as triphenylphosphine to acetylenic esters leads to reactive 1,3-dipolar intermediate betaines which are not detected even at low temperature.[6] These unstable species can be trapped by a protic reagent, such as methanol, amide and imide, to produce various compounds *e.g.* ylides.

The reaction of trimethyl phosphite with dimethylacetylenedicarboxylate (DMAD) in the presence of alcohols are reported to produce phosphite ylide derivatives which are stable at low temperatures, but convert to phosphonate derivatives by warming or by treatment with water.[7] The reaction of trimethyl phosphite with DMAD in the presence of 2-naphthol has been reported to afford stable 2,2-dimethoxy-3,4-dihydro-1-oxa-2-phosphaphenanthrene-3,4-dicarboxylates in good yield.[8] In continuation of our previous work on three-component reactions between trivalent phosphorus nucleophiles, acetylenic esters and organic acidic compounds,[9–15] we report here the results of our study on the reaction between acetylenic esters and phosphites or triphenylphosphine in the presence of (2,4-dinitrophenyl)acetic acid.



4	R	R'	Yield*
a	Me	Me	90
b	Et	Me	86
c	Me	Ph	80
d	Me	Et	83
e	Me	Bu	81

*Isolated yield (%).

Scheme 1 Three-component reaction between dialkyl acetylenedicarboxylates and phosphites in the presence of (2,4-dinitrophenyl)acetic acid.

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Results and discussion

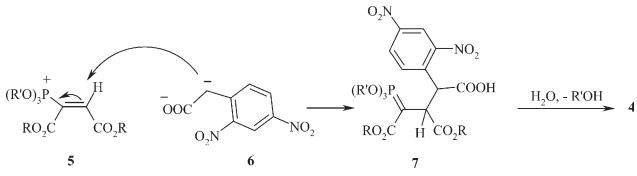
Reaction of dialkyl acetylenedicarboxylates with phosphites in the presence of (2,4-dinitrophenyl)acetic acid leads to dialkyl 2-[4-(dialkoxyphosphoryl)-2-(2,4-dinitrophenyl)-3,4-di(methoxycarbonyl)butanoic acid in excellent yields (Scheme 1).

The ^{1}H NMR spectrum of compound **4a** displayed two doublets (J_{HP} = 12 Hz) at 3.35 and 3.55 ppm for two $POCH_3$ groups and two singlets at 3.67 and 3.95 ppm for two methoxycarbonyl groups. Three signals resonate at 4.33 (dd, $^3J_{HH}$ = 12 Hz, $^2J_{HP}$ = 21 Hz, CH), 4.70 ppm (dd, $^3J_{HH}$ = 12 Hz, $^3J_{HP}$ = 5 Hz, CH) and 5.32 ppm (m, CH) for three vicinal methine protons. The aromatic protons resonated at δ 7.27–7.97 ppm. One signal observed at 8.16 ppm disappeared after addition of a few drops of D_2O to $CDCl_3$ solution of compound **4a**. This signal is related to the OH proton. The ^{13}C NMR spectrum of compound **4a** showed 16 distinct resonances in agreement with the proposed structure. The structural assignments made on the basis of the NMR spectra of compound **4a** were supported by its IR spectrum, Strong absorption bands were observed at 3095–3505 cm^{-1} for the OH and at 1735 cm^{-1} for the carbonyl group. The ^{31}P NMR spectrum of compound **4a** displays a signal at 21.65 ppm.

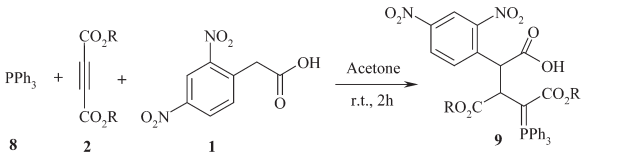
It is reasonable to assume that compound **4** results from the initial addition of phosphites **3** to dialkyl acetylenedicarboxylates **2** and subsequent protonation of the 1:1 adduct by (2,4-dinitrophenyl)acetic acid **1** (Scheme 2). Then, the positively charged ion **5** is attacked by the anion **6** to form ylide **7** that is then tautomerised and hydrolysed to phosphonate **4**.

The reaction of acetylenic ester **2** with triphenylphosphine **8** in the presence of (2,4-dinitrophenyl)acetic acid **1** leads to phosphorus ylides **9** in excellent yields (Scheme 3).

The IR spectrum of **9a** showed absorption bands at 1725 cm^{-1} for the carbonyl group and OH group at 3510 cm^{-1}. The nitro stretching vibrations observed absorption bonds at 1342 and 1530 cm^{-1}. The ^{1}H NMR spectrum of **9a** exhibited two sharp signals at δ 3.45 and 3.58 ppm arising from two OCH_3 groups. The doublet at δ 2.62 ($^3J_{HH}$ = 10.8 Hz and $^3J_{PH}$ = 16.4 Hz) and the doublet at δ 3.61 ($^3J_{HH}$ = 10.8 Hz) were attributed to CHCHC=P and CHCH=P protons respectively. The aromatic protons show multiplets at δ 7.32–8.39 ppm. One signal is



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



Entry	R	Product	Yield (%)*
1	Me	**9a**	90
2	Et	**9b**	92
3	t-Bu	**9c**	82

*Isolated yields

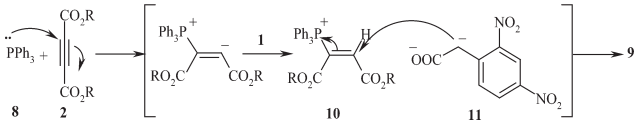
Scheme 3 Three-component reaction between triphenylphosphine, dialkyl acetylenedicarboxylates and (2,4-dinitrophenyl)acetic acid.

observed at 8.64 ppm that disappeared after addition of a few drops of D_2O to d_6-DMSO solution of compound **9a**. This signal is related to the OH proton. ^{13}C NMR spectra of compound **9a** shows 18 distinct signals, which is consistent with the proposed structure. The ^{31}P NMR spectrum of compound **9a** displays a signal at 24.49 ppm.

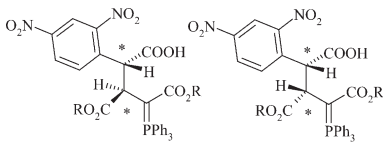
It is reasonable to assume that compound **9** results from the initial addition of triphenylphosphine **8** to acetylenic ester **2** and subsequent protonation of the 1:1 adduct by (2,4-dinitrophenyl)acetic acid **1**. Then, the positively charged ion **10** is attacked by the anion **11** to form ylide **9** (Scheme 4).

Because of the existence of two stereogenic centres in the products, mixtures of two diastereomers were expected but the ^1H NMR spectra of product **9** showed the existence of only one isomer. Comparing the coupling constant of two methine hydrogens with those for similar compounds reported previously,[16] we concluded that the obtained isomer is (2R,3R)**-9** and its enantiomer (Scheme 5).

In summary, we report that the three-component reaction between phosphites, dialkyl acetylenedicarboxylates and (2,4-dinitrophenyl)acetic acid provides a simple and efficient one-pot route for the synthesis of dialkyl 2-[2-(2,4-dinitrophenyl)-4-(dialkoxyphosphoryl)-3,4-di(methoxycarbonyl) butanoic acid in good yields and the reaction between dialkyl acetylenedicarboxylates and triphenylphosphine by (2,4-dinitrophenyl)acetic acid produces highly functionalised, salt-free phosphorus ylides in excellent yields. The present method has the advantage that the reaction is performed in neutral conditions and the starting materials can be mixed without any activation or modification.



Scheme 4 Suggested mechanism for formation of compound **9**.



Scheme 5 Two diastereomers.

Experimental

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed at the analytical laboratory of Science and Researches Unit of Islamic Azad University. 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-500 Avance spectrometer in d_6-DMSO or $CDCl_3$ using 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.

Preparation of compounds **4a–e**; *general procedure*

A mixture of phosphite (2 mmol) in 2 mL acetone was added to a magnetically stirred solution of (2,4-dinitrophenyl)acetic acid (2 mmol) and DAAD (2 mmol) in 10 mL acetone at room temperature. The reaction mixture was then stirred for 24 h. The solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel (60, 230–400 mesh) using hexane–ethyl acetate (3:1) mixture as eluent.

Dimethyl 2-[2-(3,4-di(methoxycarbonyl)-4-(dimethoxyphosphoryl) 2,4-dinitrophenyl)]butanoic acid (**4a**): Yield: 90%; Yellow oil. IR (KBr) (ν_{max}, cm^{-1}): 3095–3505 (OH), 1735 (C=O). Calcd for $C_{16}H_{19}N_2O_{13}P$: C, 40.18; H, 4.00; N, 5.86. Found: C, 40.07; H, 3.86; N, 5.96%. MS (*m/z*, %): 478 (M^+, 7). ^1H NMR (500 MHz, $CDCl_3$): δ 3.35 and 3.55 (6 H, d $^3J_{PH}$ = 12 Hz, 2$POCH_3$), 3.67 and 3.95 (6 H, 2 s, 2 OCH_3), 4.33 (1 H, dd, $^2J_{HP}$ = 21 Hz, $^3J_{HH}$ = 12 Hz, P–CH), 4.70 (1 H, dd, $^3J_{HP}$ = 5 Hz, $^3J_{HH}$ = 12 Hz, P–C–CH), 5.32 (1 H, m, P–C–CH), 7.27–7.97 (3 H, m, 3 CH aromatic), 8.16 (1H, s, OH) ppm. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 41.13 (CH), 48.64 (d, $^1J_{cp}$ = 145 Hz, P–C), 53.05 and 53.46 (2 OCH_3), 54.67 (d, $^2J_{CP}$ = 3 Hz, CH), 53.54–54.67 (m, 2 $POCH_3$), 120.15, 126.75, 33.85, 140.56, 143.72, and 150.09 (C, aromatic), 168.27 (d, $^2J_{CP}$ = 5 H_Z, C=O), 170.18 (d, $^3J_{CP}$ = 21 Hz, C=O), 175.32 (C=O) ppm. ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 21. 65 ppm.

Diethyl 2-[2-(3,4-di(methoxycarbonyl)-4-(dimethoxyphosphoryl) 2,4-dinitrophenyl)]butanoic acid (**4b**): Yield: 86%; Yellow oil. IR (KBr) (ν_{max}, cm^{-1}): 3143-3580 (OH), 1739 (C=O). Calcd for $C_{18}H_{23}N_2O_{13}P$: C, 42.70; H, 4.58; N, 5.53. Found: C, 42.89; H, 4.40; N, 5.60%. MS (*m/z*, %): 506 (M^+, 5). ^1H NMR (500 MHz, $CDCl_3$): δ 1.01 and 1.18 (6 H, 2 t, $^3J_{HH}$ = 7 Hz, 2 CH_3), 3.66–3.81 (6 H, m, 2 $POCH_3$), 3.91–4.68 (6 H, m, 2 OCH_2 and 2H), 5.04 (1 H, m, P–C–CH), 7.27–7.38 (3 H, m, 3 CH aromatic), 8.42 (1H, s, OH) ppm. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 13.84 and 14.38 (2 CH_3), 41.60 (CH), 48.42 (d, $^1J_{cp}$ = 145 Hz, P–C), 54.02 (d, $^2J_{CP}$ = 3 Hz, CH), 53.75 and 53.87 (2 d, $^2J_{CP}$ 7 Hz, 2 $POCH_3$), 62.48 and 63.39 (2 OCH_2), 120.48, 126.23, 133.84, 140.27, 143.84, and 152.14 (C, aromatic), 166.95 (d, $^2J_{CP}$ = 5 Hz, C=O), 170.31 (d, $^3J_{CP}$ = 21 Hz, C=O), 175.57 (C=O) ppm. ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 21.78 ppm.

Dimethyl 2-[3,4-di(methoxycarbonyl)-2-(2,4-dinitrophenyl)-4-(dip henoxyphosphoryl)]butanoic acid (**4c**): Yield: 80%; Yellow oil. IR (KBr) (ν_{max}, cm^{-1}): 3100–3630 (OH), 1735 (C=O). Calcd for $C_{26}H_{23}N_2O_{13}P$: C, 51.84; H, 3.85; N, 4.65. Found: C, 51.90; H, 3.70; N, 4.50%. MS (*m/z*, %): 602 (M^+, 10). ^1H NMR (500 MHz, $CDCl_3$): δ 3.69 and 3.75 (6 H, 2 s, 2 OCH_3), 4.35 (1 H, dd, $^2J_{HP}$ = 22 Hz, $^3J_{HH}$ = 12 Hz, P–CH), 4.70 (1 H, d, $^3J_{HH}$ = 1.5 Hz, CH), 4.76 (1 H, m, P–C–CH), 7.16–7.39 (13 H, m, 13 CH aromatic), 8.18 (1H, s, OH) ppm. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 43.06 (CH), 44.05 (d, $^1J_{cp}$ = 145 Hz, P–C), 53.45 (d, $^2J_{CP}$ = 3 Hz, CH), 53.67 and 53.75 (2 OCH_3), 120.12, 126.42, 133.62, 140.14, 143.55, and 150.38 (C, aromatic), 120.64 (d, $^3J_{CP}$ = 5 Hz, 4 CH_{ortho}), 126.70 (s, 2 CH_{para}), 130.18 (d, $^4J_{cp}$ = 8 Hz, 4 CH_{meta}), 150.35 (d, $^2J_{cp}$ = 10 Hz, C_{ipso}), 150.72 (d, $^2J_{cp}$ = 10 Hz, C_{ipso}), 168.25 (d, $^2J_{CP}$ = 5 Hz, C=O), 171.88 (d, $^3J_{CP}$ = 21 Hz, C=O), 175.12 (C=O) ppm. ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 21.51 ppm.

Dimethyl 2-[4-(diethoxyphosphoryl)-3,4-di(methoxycarbonyl)-2-(2,4-dinitrophenyl)butanoic acid (**4d**): Yield: 83%; Yellow oil. IR (KBr) (ν_{max}, cm^{-1}): 3141-3583 (OH), 1735 (C=O). Anal. Calcd for $C_{18}H_{23}N_2O_{13}P$: C, 42.70; H, 4.58; N, 5.53. Found: C, 42.89; H, 4.40; N, 5.60%. MS (*m/z*, %): 506 (M^+, 3). ^1H NMR (500 MHz, $CDCl_3$): δ 1.21 and 1.32 (6 H, 2 t $^3J_{HH}$ = 7 Hz, 2 CH_3), 3.74 and 3.97 (6 H, 2 s, 2 OCH_3), 4.02–4.16 (4H, m, 2 OCH_2), 4.35 (1 H, dd, $^2J_{HP}$ = 21 Hz, $^3J_{HH}$ = 12 Hz, P–CH), 4.77 (1 H, dd, $^3J_{HP}$ = 5 Hz, $^3J_{HH}$ = 12 Hz, P–C–CH), 5.38 (1 H, m, P–C–CH), 7.31 -7.84 (3 H, m, 3 CH aromatic), 8.23 (1H, s, OH) ppm. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 16.51 and 16.63 (2 CH_3), 41.15 (CH), 48.58 (d, $^1J_{cp}$ = 145 Hz, P–C), 53.12 and 53.49 (2 OCH_3), 54.65 (d, $^2J_{CP}$ = 3 Hz, CH), 63.54–63.97 (m, 2 $POCH_2$),

120.12, 126.70, 33.87, 140.62, 143.75, and 150.18 (C, aromatic), 168.33 (d, $^2J_{CP}$ = 5 H_Z, C=O), 170.24 (d, $^3J_{CP}$ = 21 Hz, C=O), 175.36 (C=O) ppm. ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 21. 48 ppm.

Dimethyl 2-[4-(dibutoxyphosphoryl)-3,4-di(methoxycarbonyl)-2-(2,4-dinitrophenyl)] butanoic acid (**4e**): Yield: 81%; Yellow oil. IR (KBr) (ν_{max} cm^{-1}): 3148–3593 (OH), 1745 (C=O). Anal. Calcd for $C_{22}H_{31}N_2O_{13}P$: C, 46.98; H, 5.56; N, 4.98. Found: C, 46.90; H, 5.45; N, 5.14%. MS (*m/z*, %): 562 (M^+, 11). ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.84, and 0.89 (6 H, 2 t, 2 CH_3), 1.37 (4 H, m, 2 CH_2), 1.54 (4 H, m, 2 CH_2), 3.72 and 3.90 (6 H, 2 s, 2 OCH_3), 3.96- 4.07 (4 H, m, 2 OCH_2), 4.40 (1 H, dd, $^2J_{HP}$ = 21 Hz, $^3J_{HH}$ = 12 Hz, P–CH), 4.76 (1 H, dd, $^3J_{HP}$ = 5 Hz, $^3J_{HH}$ = 12 Hz, P–C–CH), 5.39 (1 H, m, P–C–CH), 7.31–7.95 (3 H, m, 3 CH aromatic), 8.23 (1H, s, OH) ppm. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 13.86 and 13.94 (2 CH_3), 18.90 and 18.97 (2 CH_2), 32.61 and 32.76 (2d, $^3J_{CP}$ = 7Hz, 2 CH_2), 48.66 (d, $^1J_{cp}$ = 145 Hz, P–C), 53.12 and 53.42 (2 OCH_3), 54.68 (d, $^2J_{CP}$ = 3 Hz, CH), 66.82–67.57 (m, 2 PO*C*H_2), 120.19, 126.81, 33.77, 140.62, 143.70, and 150.19 (C, aromatic), 168.17 (d, $^2J_{CP}$ = 5 H_Z, C=O), 170.23 (d, $^3J_{CP}$ = 21 Hz, C=O), 175.36 (C=O) ppm. ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 21.61 ppm.

Synthesis of compounds **9a–c***; general procedure*

A mixture of triphenylphosphine (2 mmol) in 2 mL acetone was added to a magnetically stirred solution of (2,4-dinitrophenyl) acetic acid (2 mmol) and DAAD (2 mmol) in 10 mL acetone at room temperature. The reaction mixture was then stirred for 2 h. The solvent was evaporated at reduced pressure. The residue was precipitated in a solution of diethyl ether–hexane. The solid was filtered and washed with diethyl ether to give the pure product.

Dimethyl 2-[3,4-di(methoxycarbonyl)-2-(2,4-dinitrophenyl)]-3-(triphenyl-λ^5-phosphanylidene)]butanoic acid (**9a**): Yield: 90%; Yellow powder; m.p. 195–197 °C. IR (KBr) (ν_{max}, cm^{-1}): 3510 (OH), 1725 (C=O), 1342 and 1530 (NO_2). Calcd for $C_{32}H_{27}N_2O_{10}P$: C, 60.96; H, 4.32; N, 4.44. Found: C, 60.81; H, 4.50; N, 4.36%. MS (*m/z*, %): 630 (M^+, 4). ^{1}H NMR (500 MHz, d_6-DMSO): δ 2.62 (dd, 1H, $^3J_{PH}$ = 16.4 H_Z, $^3J_{HH}$ = 10.8 H_Z, CHCHC=P), 3.45 (s, 3H, OCH_3), 3.58 (s, 3H, OCH_3), 3.61 (d, 1H, $^3J_{HH}$ = 10.8 Hz, CHCHC=P), 7.32–8.39 (m, 18H, aromatic), 8.64(1 H, broad s, OH) ppm.^{13}C NMR (125.8 MHz, d_6-DMSO): δ 33.49 (d, $^3J_{PC}$ = 10.8 Hz, CHCHC=P), 39.21 (d, $^1J_{PC}$ = 125 Hz C=P), 45.42 (d, $^2J_{PC}$ = 14 H_Z, CHCHC=P), 49.34, 52.38 (2OCH_3), 126.69 (d, $^1J_{PC}$ = 85 Hz, C ^{ipso}), 129.46 (d, $^3J_{PC}$ = 12.0 Hz,C ^{meta}), 132.91 (d, $^4J_{PC}$ = 3.0 Hz, C ^{para}), 134.08 (d, $^2J_{PC}$ = 9 Hz, C ^{ortho}), 120.53, 127.61, 136.48, 144.08, 146.69 and 149.71 (6C, aromatic), 168.55 (d, $^2J_{PC}$ = 12.95 H_Z, C=O), 170.08 (d, $^3J_{PC}$ = 17.98 Hz, C=O), 175.64 (C=O) ppm.^{31}P NMR (202.5 MHz, d_6-DMSO): δ 24.49 ppm.

Diethyl 2-[3,4-di(methoxycarbonyl)-2-(2,4-dinitrophenyl)]-3-(triphenyl-λ^5-phosphanylidene)]butanoic acid (**9b**): Yield: 92%; Yellow powder; m.p. 145–147 °C. IR (KBr) (ν_{max}, cm^{-1}): 3505 (OH), 1723 (C=O), 1344 and 1529 (NO_2). Calcd for $C_{34}H_{31}N_2O_{10}P$: C, 62.01; H, 4.74; N, 4.25. Found: C, 61.90; H, 4.65; N, 4.17%. MS (*m/z*, %): 658 (M^+, 8). ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.37 (t, 3 H, $^3J_{HH}$ = 7.1 Hz, CH_3), 1.21(t, 3H, $^3J_{HH}$ = 7.1 Hz, CH_3), 2.60 (dd, 1H, $^3J_{PH}$ = 16.4 Hz, $^3J_{HH}$ = 10.8 Hz, CHCHC=P), 3.50 (m, 2H, OCH_2), 3.68 (d, 1H, $^3J_{HH}$ = 10.8 Hz, CHCHC=P), 4.04 (m, 2H, OCH_2), 7.35–8.37 (m, 18H, aromatic), 8.65(1H, broad s, OH) ppm. ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 14.66 and 14.97 (2CH_3), 33.54 (d, $^3J_{PC}$ = 10.8 H_Z, CHCHC=P), 39.10 (d, $^1J_{PC}$ = 125 Hz, C=P), 45.30 (d, $^2J_{PC}$ = 14 Hz, CHCHC=P), 57.48, 60.97 (2OCH_2), 126.82 (d, $^1J_{PC}$ = 85 Hz, C ^{ipso}), 129.35 (d, $^3J_{PC}$ = 12.0 Hz, C ^{meta}), 132.84 (d, $^4J_{PC}$ = 3.0 Hz, C ^{para}), 134.15 (d, $^2J_{PC}$ = 9 Hz, C ^{ortho}), 120.52, 127.56, 136.48, 144.35, 146.66 and 149.73 (6C, aromatic), 168.41 (d, $^2J_{PC}$ = 12.95 Hz, C=O), 170.10 (d, $^3J_{PC}$ = 17.98 Hz, C=O), 175.08 (C=O) ppm. ^{31}P NMR (202.5 MHz, d_6-DMSO): δ 24.57 ppm.

Di-tert-butyl-3,4-di(methoxycarbonyl)-2-[2-(2,4-dinitrophenyl)]-3-(triphenyl-λ^5-phosphanylidene)]butanoic acid (**9c**): Yield: 82%; Yellow powder; m.p. 197–199 °C. IR (KBr) (ν_{max}, cm^{-1}): 3505 (OH), 1712 (C=O), 1339 and 1530 (NO_2). Calcd for $C_{38}H_{39}N_2O_{10}P$: C, 63.86; H, 5.50; N, 3.92. Found: C, 64.02; H, 5.35; N, 3.74%. MS (*m/z*, %): 714 (M^+, 3). ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.88 (9H, s, 3CH_3), 1.39 (9H, s, 3CH_3), 2.43 (dd, 1H, $^3J_{PH}$ = 16.4 Hz, $^3J_{HH}$ = 10.8 H_Z, CHCHC=P), 3.66 (d, 1H, $^3J_{HH}$ = 10.8 Hz, CHCHC=P), 7.34–8.35 (m, 18H, aromatic), 8.62 (1H, broad s, OH) ppm. ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 28.76 and 28.87 (6CH_3), 33.61 (d, $^3J_{PC}$ = 10.8 Hz, CHCHC=P), 39.19 (d, $^1J_{PC}$ = 125 Hz, C=P), 46.14 (d, $^2J_{PC}$ = 14 Hz, CHCHC=P), 76.69 and 80.19 (2C), 126.92 (d, $^1J_{PC}$ = 85 Hz, C ^{ipso}), 129.19 (d, $^3J_{PC}$ = 12.0 Hz, C ^{meta}), 132.76 (d, $^4J_{PC}$ = 3.0 Hz, C ^{para}), 134.22 (d, $^2J_{PC}$ = 9 Hz, C ^{ortho}), 120.51, 127.42, 136.25, 144.83, 146.56 and 149.78 (6C, aromatic), 168.28 (d, $^2J_{PC}$ = 12.95 Hz, C=O), 170.16 (d, $^3J_{PC}$ = 17.98 Hz, C=O), 174.38 (C=O) ppm.^{31}P NMR (202.5 MHz, d_6-DMSO): δ 23.94 ppm.

We gratefully acknowledge financial support from the Research Council of Islamic Azad University of Yazd and The Islamic Azad University of Zahedan of Iran.

Received 19 April 2012; accepted 13 June 2012
Paper 1201275 doi: 10.3184/174751912X13403013285904
Published online: 8 August 2012

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