

## Triphenylphosphite as a good reagent for the diastereoselective synthesis of phosphonate esters

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### Abstract

The reaction between dialkyl acetylenedicarboxylates and N-H acids such as pyrazole and indazole in the presence of triphenylphosphite at room temperature led to stable phosphonate ester derivatives **4a-f**. The configuration of compounds **4a-f** (**2S**<sup>\*</sup>, **3R**<sup>\*</sup>) was determined on the basis of coupling constants predicted from the Karplus equation.

**Keywords:** N-H acids, acetylenic esters, triphenylphosphite, Karplus equation, diastereomer, phosphonate esters

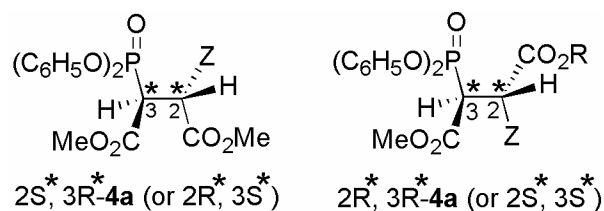
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### Introduction

In recent years there has been an increasing interest in the synthesis of organophosphorus compounds, in particular those bearing a carbon atom bound to a phosphorus atom. This interest has resulted from the recognition of the value of such compounds in a wide range of industrial, biological and chemical synthetic aspects.<sup>1-4,20-22</sup> The successful attack by nucleophilic trivalent phosphorus on a carbon atom is facilitated when the latter is part of, or conjugated with, a carbonyl group, or when it is part of an unsaturated bond otherwise activated.<sup>2-10</sup> There are many studies on the reaction between trivalent phosphorus nucleophiles and  $\alpha$ ,  $\beta$ -unsaturated carbonyl compounds in the presence of a proton source such as an alcohol or a phenol.<sup>2,10,11</sup> New or improved methods for phosphonate synthesis continue to attract much attention because phosphonates have biologically important properties and serve as natural products, analogues of phosphates, phosphonopeptides, amino acid analogues and pro drugs. Also the phosphonate esters have physiological activity within the cell.<sup>12-14, 17-19</sup>



these protons. Since compound **4a** possess two stereogenic centers, two diastereoisomers with anti HCCH arrangements are possible. The three-bond carbon-phosphorus coupling,  $^3J_{CP}$ , depends on the configuration, as expected, the transoid coupling being larger than the cisoid ones. The Karplus relation can be derived from the data for organophosphorus compounds with tetra and pentavalent phosphorus.<sup>16</sup> The observation of  $^3J_{CP}$  of 18.6 Hz for the ester C=O group (see Experimental section), is in a good agreement with the  $2S^*,3R^*$ -**4a** and its mirror image  $2R^*,3S^*$ -**4a** geometries (see Scheme 2). Although the presence of the  $^{31}P$  nucleus complicates both the  $^1H$  and  $^{13}C$  NMR spectra of **4a**, it helps in the assignment of the signals by long-range couplings with the  $^1H$  and  $^{13}C$  nuclei (see Experimental section). The  $^1H$  and  $^{13}C$  NMR spectra of (**4b-f**) are similar to those of **4a**, except for the ester groups, which exhibited characteristic resonances with appropriate chemical shifts (see Experimental section). The structural assignments made on the basis of the  $^1H$  and  $^{13}C$  NMR spectra of compounds (**4a-f**) were supported by the IR spectra. The carbonyl region of the spectra exhibited two distinct absorption bands for each compound (see Experimental section). Of special interest is the ester absorption at  $1745-1719\text{ cm}^{-1}$  for these compounds.



Scheme 2

## Experimental Section

**General Procedures.** Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a Shimadzu IR-460 spectrometer respectively. Also, the  $^1H$ ,  $^{13}C$ , and  $^{31}P$  NMR spectra were obtained from a BRUKER DRX-500 AVANCE instrument with  $CDCl_3$  as solvent at 500.1, 125.8, and 202.4 MHz respectively. In addition, the mass spectra were recorded on a Shimadzu QP 1100 EX mass spectrometer operating at an ionization potential of 70 eV. Elemental analysis for C, H and N were performed using a Heraeus CHN-O-Rapid analyzer. Dialkyl acetylenedicarboxylates, triphenylphosphite, pyrazole and indazole were purchased from Fluka, (Buchs, Switzerland) and used without further purifications.

**Preparation of (2*S*,3*R*\*)-dimethyl-2-(pyrazole-1-yl)-3-(diphenoxyphosphonato)butane dioate (4a).** To a magnetically stirred solution of triphenylphosphite (0.31g, 1mmol) and pyrazole (0.07g, 1mmol) in diethyl ether (10ml) was added dropwise a mixture of dimethyl acetylenedicarboxylate (0.14g, 1mmol) in diethyl ether (5ml) at  $-10\text{ }^\circ\text{C}$  over 10 min. After

approximately 24 hours stirring at room temperature, the solvent was removed under reduced pressure and product washed with cold diethyl ether (2×5mL).

White powder, 97%, mp=125-127 °C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1745 and 1719 (C=O), 1271 (P=O). Anal. Calcd for  $\text{C}_{21}\text{H}_{21}\text{N}_2\text{O}_7\text{P}$  (444): C, 56.63; H, 4.80; N, 6.25, Found: C, 56.76; H, 4.73; N, 6.31.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 3.71 and 3.79 (6H, 2s, 2 $\text{CH}_3$ ), 4.62 (1H, dd,  $^2J_{\text{PH}}=21.2$  Hz,  $^3J_{\text{HH}}=10.8$  Hz, P-CH-CH), 5.82 (1H, dd,  $^3J_{\text{PH}}=8.4$  Hz,  $^3J_{\text{HH}}=10.8$  Hz, P-CH-CH), 6.28 -7.66 (13H, m,  $\text{H}_{\text{aro}}$ ).  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 47.96 (d,  $^1J_{\text{CP}}=134.8$  Hz, P-CH), 53.05 and 53.20 (s, 2 $\text{OCH}_3$ ), 61.72 (d,  $^2J_{\text{CP}}=3.5$  Hz, P-C-CH), 106.20 (1C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 120.04 120.30 (2d,  $^3J_{\text{PC}}=4.7$  Hz  $\text{C}_{\text{ortho}}$  of 2 $\text{C}_6\text{H}_5$ ), 125.29 and 125.39 ( $\text{C}_{\text{para}}$  of 2 $\text{C}_6\text{H}_5$ ), 129.64 and 129.73 ( $\text{C}_{\text{meta}}$  of 2 $\text{C}_6\text{H}_5$ ), 131.92 and 141.17 (2C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 149.47 and 149.88 (2d,  $^2J_{\text{CP}}=9.0$  Hz,  $\text{C}_{\text{ipso}}$  of 2 $\text{C}_6\text{H}_5$ ), 166.64 (d,  $^3J_{\text{CP}}=6.2$  Hz, C=O), 167.04 (d,  $^2J_{\text{CP}}=18.6$  Hz, C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ , 10.13 [s,  $(\text{PhO})_2\text{P}(\text{=O})$ ].

**(2S\*,3R\*)-Diethyl-2-(pyrazole-1-yl)-3-(diphenoxyphosphonato)butanedioate (4b).** White powder, 96%, mp=124-126 °C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1741 and 1719 (C=O), 1269 (P=O). Anal. Calcd for  $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_7\text{P}$  (472): C, 58.35; H, 5.35; N, 6.02, Found: C, 58.47; H, 5.30; N, 5.93.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 1.14 and 1.25 (6H, 2t,  $^3J_{\text{HH}}=7.1$  Hz, 2 $\text{CH}_3$ ), 4.16 and 4.25 (4H, m, 2ABX<sub>3</sub>system, 2 $\text{OCH}_2$ ), 4.60 (1H, dd,  $^2J_{\text{PH}}=21.2$  Hz,  $^3J_{\text{HH}}=11.0$  Hz, P-CH-CH), 5.77 (1H, dd,  $^3J_{\text{PH}}=8.8$  Hz,  $^3J_{\text{HH}}=11.0$  Hz, P-CH-CH), 6.24 -7.64 (13H, m,  $\text{H}_{\text{aro}}$ ).  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 13.77 and 13.82(2 $\text{CH}_3$ ), 48.06 (d,  $^1J_{\text{CP}}=134.6$  Hz, P-CH), 62.01 (d,  $^2J_{\text{CP}}=3.3$  Hz, P-C-CH), 62.41 and 62.55 (2 $\text{OCH}_2$ ), 106.07 (1C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 120.24 and 120.44 (2d,  $^3J_{\text{PC}}=4.6$  Hz  $\text{C}_{\text{ortho}}$  of 2 $\text{C}_6\text{H}_5$ ), 125.39 and 125.45 ( $\text{C}_{\text{para}}$  of 2 $\text{C}_6\text{H}_5$ ), 129.64 and 129.66 ( $\text{C}_{\text{meta}}$  of 2 $\text{C}_6\text{H}_5$ ), 131.97 and 141.22 (2C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 149.72 and 150.07 (2d,  $^2J_{\text{CP}}=9.6$  Hz,  $\text{C}_{\text{ipso}}$  of 2 $\text{C}_6\text{H}_5$ ), 166.22 (d,  $^3J_{\text{CP}}=6.0$  Hz, C=O), 167.08 (d,  $^2J_{\text{CP}}=19.0$  Hz, C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ , 10.85 [s,  $(\text{PhO})_2\text{P}(\text{=O})$ ].

**(2S\*,3R\*)-Di-tert-butyl -2-(pyrazole-1-yl)-3-(diphenoxyphosphonato)butanedioate (4c).** White powder, 92%, mp=124-126 °C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1739 and 1722 (C=O), 1273 (P=O). Anal. Calcd for  $\text{C}_{27}\text{H}_{33}\text{N}_2\text{O}_7\text{P}$  (528): C, 61.50; H, 6.19; N, 5.37, Found: C, 61.36; H, 6.25; N, 5.30.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 1.36 and 1.47 (18H, 2 $\text{CMe}_3$ ), 4.52 (1H, dd,  $^2J_{\text{PH}}=20.9$  Hz,  $^3J_{\text{HH}}=11.1$  Hz, P-CH-CH), 5.64 (1H, dd,  $^3J_{\text{PH}}=8.6$  Hz,  $^3J_{\text{HH}}=11.1$  Hz, P-CH-CH), 6.24 -7.61 (13H, m,  $\text{H}_{\text{aro}}$ ).  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 27.62 and 27.70 (2 $\text{CMe}_3$ ), 48.98 (d,  $^1J_{\text{CP}}=135.7$  Hz, P-CH), 62.78 (d,  $^2J_{\text{CP}}=3.4$  Hz, P-C-CH), 83.33 and 83.41 (2 $\text{CMe}_3$ ), 105.97 (1C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 120.38 and 120.69 (2d,  $^3J_{\text{PC}}=4.5$  Hz  $\text{C}_{\text{ortho}}$  of 2 $\text{C}_6\text{H}_5$ ), 125.33 and 125.49 ( $\text{C}_{\text{para}}$  of 2 $\text{C}_6\text{H}_5$ ), 129.26 and 129.64 ( $\text{C}_{\text{meta}}$  of 2 $\text{C}_6\text{H}_5$ ), 131.62 and 140.86 (2C,  $\text{C}_3\text{H}_3\text{N}_2$ ), 149.81 and 149.95 (2d,  $^2J_{\text{CP}}=9.4$  Hz,  $\text{C}_{\text{ipso}}$  of 2 $\text{C}_6\text{H}_5$ ), 163.64 (d,  $^3J_{\text{CP}}=6.0$  Hz, C=O), 165.98 (d,  $^2J_{\text{CP}}=19.7$  Hz, C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ , 10.85 [s,  $(\text{PhO})_2\text{P}(\text{=O})$ ].

**(2S\*3R\*)-Dimethyl-2-(indazole-1-yl)-3-(diphenoxyphosphonato)butanedioate(4d).** White powder, 98%, mp=132-134 °C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1745 and 1719 (C=O), 1271 (P=O). Anal. Calcd for  $\text{C}_{25}\text{H}_{23}\text{N}_2\text{O}_7\text{P}$  (494): C, 61.03; H, 4.51; N, 5.73, Found: C, 60.73; H, 4.66; N, 5.67.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 3.52 and 3.77 (6H, 2s, 2 $\text{CH}_3$ ), 4.87 (1H, dd,  $^2J_{\text{PH}}=21.0$  Hz,  $^3J_{\text{HH}}=11.2$  Hz, P-CH-CH), 6.24 (1H, dd,  $^3J_{\text{PH}}=8.9$  Hz,  $^3J_{\text{HH}}=11.2$  Hz, P-CH-CH), 6.63 -8.02 (15H,

m, H<sub>aro</sub>). <sup>13</sup>C NMR (125.8 MHz, δ, CDCl<sub>3</sub>): 47.84 (d, <sup>1</sup>J<sub>CP</sub>=135.9 Hz, P-CH), 53.28 and 53.41 (s, 2OCH<sub>3</sub>), 58.66 (d, <sup>2</sup>J<sub>CP</sub>=3.9 Hz, P-C-CH), 109.71 and 119.62 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 120.21 and 120.40 (2d, <sup>3</sup>J<sub>PC</sub>= 4.4 Hz C<sub>ortho</sub> of 2C<sub>6</sub>H<sub>5</sub>), 121.60 and 124.23 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 125.56 and 125.64 (C<sub>para</sub> of 2C<sub>6</sub>H<sub>5</sub>), 127.38 (1C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 129.38 and 129.69 (C<sub>meta</sub> of 2C<sub>6</sub>H<sub>5</sub>), 135.69 and 140.76 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 149.36 and 149.74 (2d, <sup>2</sup>J<sub>CP</sub>=9.8 Hz, C<sub>ipso</sub> of 2C<sub>6</sub>H<sub>5</sub>), 167.05 (d, <sup>3</sup>J<sub>CP</sub>= 5.9 Hz, C=O), 168.23 (d, <sup>2</sup>J<sub>CP</sub>=19.3 Hz, C=O). <sup>31</sup>P NMR (202.4 MHz, δ, 11.20 [s, (PhO)<sub>2</sub>P=O].

**(2S\*,3R\*)-Diethyl-2-(indazol -1-yl)-3-(diphenoxyphosphonato)butanedioate (4e).** White powder, 95%, mp=128-130 °C. IR (KBr) (ν<sub>max</sub>, cm<sup>-1</sup>): 1738 and 1720 (C=O), 1272 (P=O). Anal. Calcd for C<sub>27</sub>H<sub>27</sub>N<sub>2</sub>O<sub>7</sub>P (522): C, 62.21; H, 5.11; N, 5.41, Found: C, 62.07; H, 5.17; N, 5.36. <sup>1</sup>H NMR (500.1 MHz, δ, CDCl<sub>3</sub>): 1.14 and 1.25 (6H, 2t, <sup>3</sup>J<sub>HH</sub>=7.1 Hz, 2CH<sub>3</sub>), 4.13 and 4.25 (4H, m, 2ABX<sub>3</sub>system, 2OCH<sub>2</sub>), 4.59 (1H, dd, <sup>2</sup>J<sub>PH</sub>=21.5 Hz, <sup>3</sup>J<sub>HH</sub>=11.1 Hz, P-CH-CH), 5.67 (1H, dd, <sup>3</sup>J<sub>PH</sub>=8.8 Hz, <sup>3</sup>J<sub>HH</sub>=11.1 Hz, P-CH-CH), 6.20 -7.56 (15H, m, H<sub>aro</sub>). <sup>13</sup>C NMR (125.8 MHz, δ, CDCl<sub>3</sub>): 14.13 and 14.39 (2CH<sub>3</sub>), 45.33 (d, <sup>1</sup>J<sub>CP</sub>=132.3 Hz, P-CH), 62.50 (d, <sup>2</sup>J<sub>CP</sub>=3.7 Hz, P-C-CH), 62.67 and 62.94 (2OCH<sub>2</sub>), 109.54 and 119.78 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 120.68 and 120.91 (2d, <sup>3</sup>J<sub>PC</sub>= 4.3 Hz C<sub>ortho</sub> of 2C<sub>6</sub>H<sub>5</sub>), 121.15 and 124.56 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 125.61 and 125.68 (C<sub>para</sub> of 2C<sub>6</sub>H<sub>5</sub>), 127.25 (1C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 129.56 and 129.71 (C<sub>meta</sub> of 2C<sub>6</sub>H<sub>5</sub>), 135.84 and 141.04 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 149.69 and 150.05 (2d, <sup>2</sup>J<sub>CP</sub>=9.6 Hz, C<sub>ipso</sub> of 2C<sub>6</sub>H<sub>5</sub>), 166.16 (d, <sup>3</sup>J<sub>CP</sub>= 6.1 Hz, C=O), 167.21 (d, <sup>2</sup>J<sub>CP</sub>=19.5 Hz, C=O). <sup>31</sup>P NMR (202.4 MHz, δ, 10.85 [s, (PhO)<sub>2</sub>P=O].

**(2S\*,3R\*)-Di-tert-butyl -2-(indazole -1-yl)-3-(diphenoxyphosphonato)butanedioate (4f).**

White powder, 91%, mp=112-115 °C. IR (KBr) (ν<sub>max</sub>, cm<sup>-1</sup>): 1736 and 1725 (C=O), 1283 (P=O). Anal. Calcd for C<sub>31</sub>H<sub>35</sub>N<sub>2</sub>O<sub>7</sub>P (578): C, 64.51; H, 5.97; N, 4.86, Found: C, 64.36; H, 6.06; N, 4.84. <sup>1</sup>H NMR (500.1 MHz, δ, CDCl<sub>3</sub>): 1.26 and 1.52 (18H, 2CMe<sub>3</sub>), 4.73 (1H, dd, <sup>2</sup>J<sub>PH</sub>=22.1 Hz, <sup>3</sup>J<sub>HH</sub>=11.2 Hz, P-CH-CH), 6.02 (1H, dd, <sup>3</sup>J<sub>PH</sub>=8.9 Hz, <sup>3</sup>J<sub>HH</sub>=11.2 Hz, P-CH-CH), 6.67 -8.01 (15H, m, H<sub>aro</sub>). <sup>13</sup>C NMR (125.8 MHz, δ, CDCl<sub>3</sub>): 27.62 and 27.75 (2CMe<sub>3</sub>), 48.46 (d, <sup>1</sup>J<sub>CP</sub>=134.5 Hz, P-CH), 59.70 (d, <sup>2</sup>J<sub>CP</sub>=4.0 Hz, P-C-CH), 83.13 and 83.37 (2CMe<sub>3</sub>), 109.82 and 119.58 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 120.32 and 120.72 (2d, <sup>3</sup>J<sub>PC</sub>= 4.7 Hz C<sub>ortho</sub> of 2C<sub>6</sub>H<sub>5</sub>), 122.19 and 124.17 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 125.20 and 125.49 (C<sub>para</sub> of 2C<sub>6</sub>H<sub>5</sub>), 126.88 (1C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 129.28 and 129.36 (C<sub>meta</sub> of 2C<sub>6</sub>H<sub>5</sub>), 135.38 and 140.72 (2C, C<sub>7</sub>H<sub>5</sub>N<sub>2</sub>), 149.68 and 149.79 (2d, <sup>2</sup>J<sub>CP</sub>=9.5 Hz, C<sub>ipso</sub> of 2C<sub>6</sub>H<sub>5</sub>), 163.89 (d, <sup>3</sup>J<sub>CP</sub>= 6.5 Hz, C=O), 165.63 (d, <sup>2</sup>J<sub>CP</sub>=20.3 Hz, C=O). <sup>31</sup>P NMR (202.4 MHz, δ, 10.85 [s, (PhO)<sub>2</sub>P=O].

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