

# Stable phosphorus ylides and heterocyclic phosphonate esters derivatives synthesised from stereoselective reactions between triphenyl phosphite and activated acetylenic esters

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One-pot synthesis of stable heterocyclic phosphorus ylides **4a–j** is reported in fairly good yields by the reaction between dialkyl acetylenedicarboxylates and triphenyl phosphite in the presence of strong NH-acids such as 2-benzoxazolinone, 2-indolinone and 2-mercaptobenzoxazole in aqueous media as an environmentally friendly solvent. The hydrolysis of compounds **4a–f** led to stable phosphonate ester derivatives **5h–l**. The configuration of compounds **5h–l** (2*S*\*,3*R*\*) was determined on the basis of coupling constant predicted from the Karplus equation.

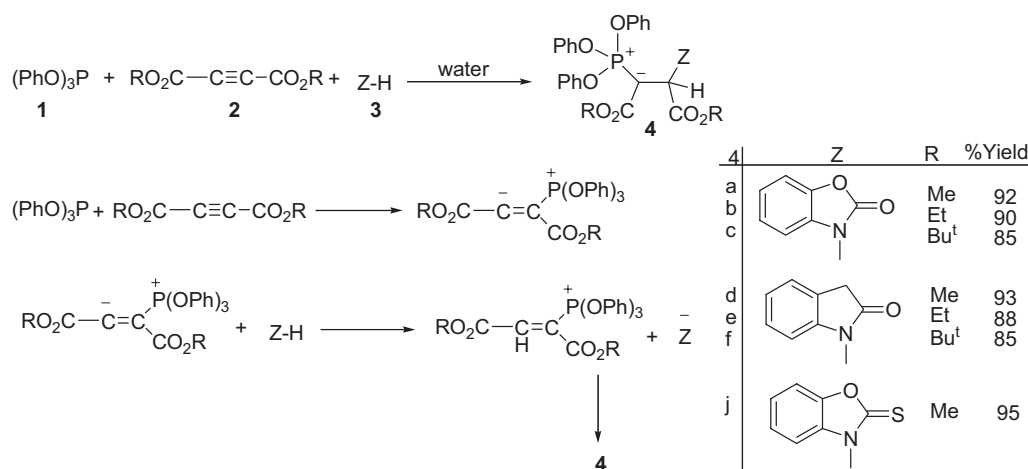
**Keywords:** dialkyl acetylenedicarboxylates, NH-acids, triphenyl phosphite, heterocyclic phosphorus ylide, phosphonate esters

Phosphorus ylides are important reactants in organic chemistry because of their applications in the synthesis of organic products,<sup>1–6</sup> especially naturally occurring products with biological and pharmacological activity.<sup>7–19</sup> Recently, organic reactions in water have attracted much attention because water is the most readily available safe solvent.<sup>20–23</sup> The use of water as a solvent has been uncommon in organic reactions for several reasons, among them the insolubility of the reactant, the incompatibility of intermediates with water and the competition of the desired reaction with hydrolysis.<sup>24,25</sup> During our investigations to develop the synthesis of organophosphorus compounds,<sup>26–33</sup> we found that water as a solvent provides an efficient system to help the one-pot synthesis of heterocyclic phosphorus ylides.<sup>34</sup> This system involved three components, dialkyl acetylenedicarboxylates, triphenyl phosphite and NH-acids. For this reason, synthesis of a new class of stable phosphorus ylides **4a–j** was undertaken from the reaction between triphenyl phosphite and acetylenic esters in the presence of NH-acids such as 2-benzoxazolinone, 2-indolinone and 2-mercaptobenzoxazole in aqueous media. The hydrolysis of compounds **4a–j** led to *S*\*,*R*\* phosphonate esters **5h–l** in stereoselective reaction. The phosphonate esters 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>35–38</sup>

## Results and discussion

To generate new class of stable phosphorus ylide **4a–j** (see Scheme 1) the reactions between dialkylacetylenedicarboxylates **2** and benzoxazolin-2-one, indolin-2-one or 2-mercaptobenzoxazole **3** in the presence of triphenyl phosphite **1** were proceeded in distilled water as solvent at room temperature and finished after approximately 20–50 minutes. The <sup>1</sup>H and <sup>13</sup>C NMR spectra of the crude product clearly indicated the formation of ylide **4**. Any product other than **4** could not be detected by NMR spectroscopy. The structures of compounds **4a–j** were deduced from their IR, <sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P NMR spectra (see Experimental). The mass spectra of them displayed ion peaks at appropriate *m/z* values. Any initial fragmentations involve loss of the side chains.

The <sup>1</sup>H NMR 500 MHz spectrum of compound **4a** displayed two sharp lines ( $\delta = 3.58$  and 3.64) arising from methoxy protons along with signal for methine proton at  $\delta = 6.02$  ppm, which appear as one doublet ( $^3J_{\text{PH}} = 16.9$  Hz). The <sup>13</sup>C NMR spectrum of **4a** exhibited 17 distinct resonances that is in a good agreement with the structure of **4a**. Although the presence of the <sup>31</sup>P nucleus complicates both the <sup>1</sup>H and <sup>13</sup>C NMR spectra of **4a**, it helps in assignment of the signals by long-range couplings with the <sup>1</sup>H and <sup>13</sup>C nuclei (see Experimental). The <sup>1</sup>H and <sup>13</sup>C NMR spectra of **4b–j** are similar to those of **4a**, except for the ester groups, which exhibited characteristic resonances with appropriate chemical



Scheme 1

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shifts (see Experimental). The structural assignments made on the basis of the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compounds **4a–j** were supported by the IR spectra. The carbonyl region of the spectra exhibited two or three distinct absorption bands for each compound (see Experimental). Of special interest is the carbonyl absorption at  $1766\text{--}1656\text{ cm}^{-1}$  for these compounds. Conjugation with the negative charge appears to be a plausible factor in the reduction of the wave numbers of the carbonyl absorption bands.

On the other hand, a simple, short, neutral stereoselective synthesis of phosphonate esters **5h–l** is reported from the hydrolysis of compounds **4a–e** at room temperature (see Scheme 2). The  $^1\text{H}$  NMR 500 MHz spectra of compound **5h** displayed two sharp lines ( $\delta = 3.75, 3.85$ ) arising from methoxy protons, along with signals from methine protons at  $\delta = 4.66$  ( $^2J_{\text{PH}} = 20.3\text{ Hz}$ ,  $^3J_{\text{HH}} = 11.4\text{ Hz}$ ) and  $\delta = 5.71$  which appear as doublet of doublet and broad, for the  $\text{O}=\text{P}-\text{CH}-\text{CH}$  and  $\text{O}=\text{P}-\text{CH}-\text{CH}$  groups respectively. The vicinal proton–proton coupling constant ( $^3J_{\text{HH}}$ ) as a function of the torsion angle can be obtained from the Karplus equation.<sup>37,38</sup> Typically,  $J_{\text{gauche}}$  varies between 1.5 and 5 Hz and  $J_{\text{anti}}$  between 10 and 14 Hz. Observation of  $^3J_{\text{HH}} = 11.4\text{ Hz}$  for the vicinal protons in compound **5h** (see Experimental) indicates an anti arrangement for these protons.<sup>13,16,17</sup> Since compound **5h** possess two stereogenic centres, two diastereoisomers with anti HCCH arrangements are possible. The three-bond carbon–phosphorus coupling  $^3J_{\text{PC}}$ , depends on configuration, as expected, for 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>39</sup> The observation of  $^3J_{\text{PC}} = 19.2\text{ Hz}$  for the ester  $\text{C}=\text{O}$  group (see Experimental), is in a good agreement with the (2*S*,3*R*)-**5h** and its mirror image (2*R*,3*S*)-**5h** geometries (see Scheme 3). The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **5i–l** are similar to those of **5h**, except for the ester groups.

Briefly, we have developed new method to present a one-pot synthesis of novel phosphorus ylides in aqueous media for first time. The present method carries the advantage that not only is the reaction performed under neutral conditions, but also that the substances can be mixed without any activation or modifications. The phosphorus ylides **4a–j** may be considered as potentially useful synthetic intermediates. It seems that the

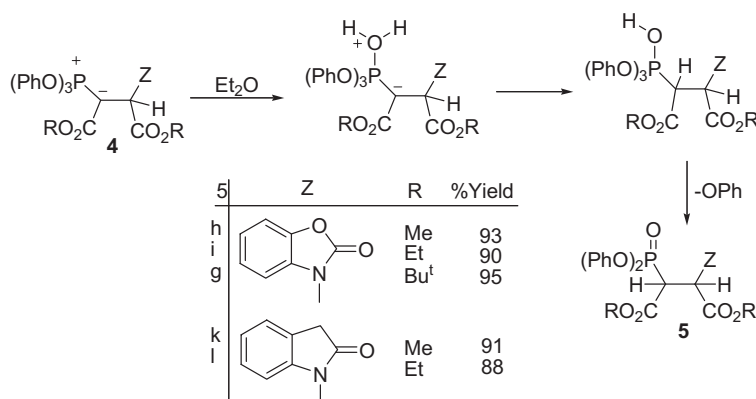
procedure described here may be employed as an acceptable method for the preparation of phosphorus ylides **4a–j** in a friendly environmental approach with variable functionalities. On the other hand, the hydrolysis of compounds **4a–j** led to phosphonate esters **5h–l**. The simplicity of the present procedure makes it an interesting alternative to other approaches.

## Experimental

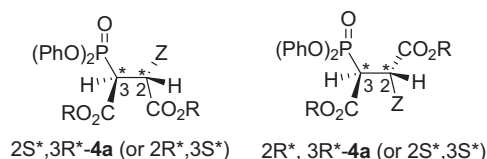
Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a Shimadzu IR-460 spectrometer respectively. Also the  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{31}\text{P}$  NMR spectra were obtained from a Bruker DRX-500 Avance instrument with  $\text{CDCl}_3$  as applied solvent at 500.1, 125.8, and 202.4 MHz respectively. Elemental analyses for C, H, N were performed using a Heraeus CHN–O–Rapid analyser (for **4a**, **4b**, **4c**, **4d**, **4e**, **4f**, **4j**, **5i**, **5g**, **5k** and **5l**) and also new equipment (CHNF–O–Perkin element 2004 II) for **5h** compound. In addition, the mass spectra were recorded on a Shimadzu QP 1100 EX mass spectrometer operating at an ionisation potential of 70 eV. Triphenyl phosphite, dialkyl acetylene dicarboxylates, 2-benzoxazolinone, 2-indolinone and 2-mercaptobenzoxazole were purchased from Fluka (Buchs, Switzerland) and used without further purification.

### General procedures (exemplified by **4a**)

**Dimethyl 2-(2-oxo-2,3-dihydro-1,3-benzoxazol-3-yl)-3-(triphenoxyphosphanylidene) butanedioate (4a)**: To a magnetically stirred solution of benzoxazolin-2-one (0.14 g, 1 mmol) and triphenyl phosphite (1 mmol) in 20 ml of water was added, dropwise, dimethyl acetylenedicarboxylate (0.14 g, 1 mmol) at  $10^\circ\text{C}$  for 10 minutes. After approximately 20–50 minutes stirring at room temperature, the product was filtered and washed with cold diethyl ether ( $3 \times 5\text{ ml}$ ) and it was obtained as white powder, yield 92%, 0.54 g; m.p.  $126\text{--}128^\circ\text{C}$ , IR ( $\nu_{\text{max}}$ ,  $\text{cm}^{-1}$ ): 1766, 1748 and 1661 ( $\text{C}=\text{O}$ ). MS, ( $m/z$ , %): 587 ( $\text{M}^+$ , 4), 494 ( $\text{M}^+-\text{OPh}$ , 87), 401 ( $\text{M}^+-2\text{OPh}$ , 74), 310 ( $\text{P}(\text{OPh})_3$ , 59). Anal. Calcd for  $\text{C}_{31}\text{H}_{26}\text{N}_2\text{O}_9\text{P}$  (587): C, 63.35; H, 4.46; N, 2.38. Found: C, 63.82; H, 4.53; N, 2.45.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  3.58 and 3.64 (6H, 2 s,  $2\text{OCH}_3$ ), 6.02 (1H, d,  $^3J_{\text{PH}} = 16.9\text{ Hz}$ ,  $\text{P}^+-\text{C}-\text{CH}$ ), 6.92–7.26 (19H<sub>arom</sub>,  $3\text{C}_6\text{H}_5$  and  $\text{C}_7\text{H}_4\text{NO}_2$ ).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  44.79 (d,  $^1J_{\text{PC}} = 230.2\text{ Hz}$   $\text{P}^+-\text{C}$ ), 50.44 and 52.60 ( $2\text{OCH}_3$ ), 55.18 (d,  $^2J_{\text{PC}} = 13.8\text{ Hz}$ ,  $\text{P}^+-\text{C}-\text{CH}$ ), 109.07 and 112.98 ( $2\text{C}$ ,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 120.08 (d,  $^3J_{\text{PC}} = 4.5\text{ Hz}$ ,  $\text{C}_{\text{ortho}}$ ), 121.71 and 123.82 ( $2\text{C}$ ,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 126.08 ( $\text{C}_{\text{para}}$ ), 129.34 ( $1\text{C}$ ,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 129.83 ( $\text{C}_{\text{meta}}$ ), 142.23 ( $1\text{C}$ ,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 149.68 (d,  $^2J_{\text{PC}} = 7.4\text{ Hz}$ ,  $\text{C}_{\text{ipso}}$ ), 154.32 ( $1\text{C}$ ,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 168.27 (d,  $^2J_{\text{PC}} = 21.4\text{ Hz}$ ,  $\text{C}=\text{O}$ ), 169.24 (d,  $^3J_{\text{PC}} = 17.6\text{ Hz}$ ,  $\text{C}=\text{O}$ ):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  41.88 ( $(\text{PhO})_3\text{P}^+-\text{C}$ ).



Scheme 2



Scheme 3

**Diethyl 2-(2-oxo-2,3-dihydro-1,3-benzoxazol-3-yl)-3-(triphenoxyphosphanylidene)butanedioate (4b):** White powder, yield 90%, 0.55 g; m.p. 139–141°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1766, 1740 and 1659 (C=O). Anal. Calcd for  $\text{C}_{33}\text{H}_{30}\text{NO}_9\text{P}$  (615): C, 64.37; H, 4.91; N, 2.28. Found: C, 64.77; H, 5.01; N, 2.32.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.14 and 1.18 (6H, 2t,  $^3J_{\text{HH}} = 6.5$  Hz  $2\text{OCH}_2\text{CH}_3$ ), 4.03 and 4.15 (4H, m, 2ABX<sub>3</sub> system  $2\text{OCH}_2\text{CH}_3$ ), 6.03 (1H, d,  $^3J_{\text{PH}} = 16.6$  Hz, P<sup>+</sup>-C-CH), 6.90–7.18 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  14.11 and 14.84 ( $2\text{OCH}_2\text{CH}_3$ ), 44.90 (d,  $^1J_{\text{PC}} = 228.8$  Hz, P<sup>+</sup>-C), 55.28 (d,  $^2J_{\text{PC}} = 13.5$  Hz, P<sup>+</sup>-C-CH), 59.12 and 61.51 ( $2\text{OCH}_2\text{CH}_3$ ), 108.98 and 113.14 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 119.93 (d,  $^3J_{\text{PC}} = 5.3$  Hz C<sub>ortho</sub>), 121.65 and 123.75 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 126.07 (C<sub>para</sub>), 129.43 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 129.83 (C<sub>meta</sub>), 142.23 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 149.66 (d,  $^2J_{\text{PC}} = 7.2$  Hz, C<sub>ipso</sub>), 154.55 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 167.98 (d,  $^2J_{\text{PC}} = 18.9$  Hz, C=O), 168.72 (d,  $^3J_{\text{PC}} = 18.1$  Hz, C=O):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  42.90 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

**Di-tert-butyl 2-(2-oxo-2,3-dihydro-1,3-benzoxazol-3-yl)-3-(triphenoxyphosphanylidene)butanedioate (4c):** White powder, yield 85%, 0.57 g; m.p. 145–147°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1766, 1736 and 1661 (C=O). MS, ( $m/z$ , %): 671 (M<sup>+</sup>, 2), 578 (M<sup>+</sup>-OPh, 35), 485 (M<sup>+</sup>-2OPh, 43), 310 ((PhO)<sub>3</sub>P, 52). Anal. Calcd for  $\text{C}_{37}\text{H}_{38}\text{NO}_9\text{P}$  (671): C, 66.14; H, 5.70; N, 2.09. Found: C, 65.94; H, 5.80; N, 2.12.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.38 and 1.42 (18H, 2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 5.96 (1H, d,  $^3J_{\text{PH}} = 16.2$  Hz, P<sup>+</sup>-C-CH), 6.91–7.26 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  28.12 and 28.64 (2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 45.72 (d,  $^1J_{\text{PC}} = 227.5$  Hz, P<sup>+</sup>-C), 55.81 (d,  $^2J_{\text{PC}} = 13.9$  Hz, P<sup>+</sup>-C-CH), 78.81 and 81.67 (2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 108.92 and 113.41 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 119.76 (d,  $^3J_{\text{PC}} = 5.5$  Hz, C<sub>ortho</sub>), 121.53 and 123.71 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 125.74 (C<sub>para</sub>), 29.55 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 129.77 (C<sub>meta</sub>), 142.19 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 149.64 (d,  $^2J_{\text{PC}} = 6.7$  Hz, C<sub>ipso</sub>), 154.68 (1C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 167.30 (d,  $^2J_{\text{PC}} = 20.2$  Hz, C=O), 167.49 (d,  $^3J_{\text{PC}} = 18.7$  Hz, C=O):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  41.09 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

**Dimethyl 2-(2-oxo-2,3-dihydroindolin-1-yl)-3-(triphenoxyphosphanylidene)butanedioate (4d):** White powder, yield 93%, 0.54 g; m.p. 136–138°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1747, 1698 and 1662 (C=O). MS, ( $m/z$ , %): 585 (M<sup>+</sup>, 3), 492 (M<sup>+</sup>-OPh, 81), 399 (M<sup>+</sup>-2OPh, 72), 306 (M<sup>+</sup>-3OPh, 62). Anal. Calcd for  $\text{C}_{32}\text{H}_{28}\text{NO}_8\text{P}$  (585): C, 65.62; H, 4.82; N, 2.39. Found: C, 66.01; H, 4.79; N, 2.43.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  3.20 and 3.42 (2H, 2d,  $^2J_{\text{HH}} = 22.2$ , CH<sub>2</sub>), 3.52 and 3.62 (6H, 2 s,  $2\text{OCH}_3$ ), 6.21 (1H, d,  $^3J_{\text{PH}} = 18.3$  Hz, P<sup>+</sup>-C-CH), 6.89–7.22 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>8</sub>H<sub>6</sub>NO).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  35.89 (s, CH<sub>2</sub>), 42.28 (d,  $^1J_{\text{PC}} = 230.6$  Hz P<sup>+</sup>-C), 50.31 and 51.83 ( $2\text{OCH}_3$ ), 51.83 (d,  $^2J_{\text{PC}} = 12.4$  Hz P<sup>+</sup>-C-CH), 112.97 (1C, C<sub>8</sub>H<sub>6</sub>NO), 120.94 (d,  $^3J_{\text{PC}} = 4.4$  Hz, C<sub>ortho</sub>), 121.69, 123.30 and 123.43 (3C, C<sub>8</sub>H<sub>6</sub>NO), 125.88 (C<sub>para</sub>), 127.81 (1C, C<sub>8</sub>H<sub>6</sub>NO<sub>2</sub>), 129.71 (C<sub>meta</sub>), 143.05 (1C, C<sub>8</sub>H<sub>6</sub>NO), 149.86 (d,  $^2J_{\text{PC}} = 7.5$  Hz, C<sub>ipso</sub>), 168.48 (d,  $^2J_{\text{PC}} = 21.5$  Hz, C=O), 169.98 (d,  $^3J_{\text{PC}} = 17.6$  Hz, C=O), 174.51 (1C, C<sub>8</sub>H<sub>6</sub>NO):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  42.66 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

**Diethyl 2-(2-oxo-2,3-dihydroindolin-1-yl)-3-(triphenoxyphosphanylidene)butanedioate (4e):** White powder, yield 88%, 0.54 g; m.p. 139–141°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1740, 1692 and 1656 (C=O). MS, ( $m/z$ , %): 613 (M<sup>+</sup>, 3), 520 (M<sup>+</sup>-OPh, 83), 427 (M<sup>+</sup>-2OPh, 49), 310 (P(OPh)<sub>3</sub>, 61). Anal. Calcd for  $\text{C}_{34}\text{H}_{32}\text{NO}_8\text{P}$  (613): C, 66.53; H, 5.26; N, 2.28. Found: C, 66.59; H, 5.30; N, 2.31.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.14 and 1.18 (6H, 2t,  $^3J_{\text{HH}} = 7.1$  Hz  $2\text{OCH}_2\text{CH}_3$ ), 3.19 and 3.42 (2H, 2d,  $^2J_{\text{HH}} = 21.9$  Hz, CH<sub>2</sub>), 3.97 and 4.14 (4H, m, 2ABX<sub>3</sub> system  $2\text{OCH}_2\text{CH}_3$ ), 6.24 (1H, d,  $^3J_{\text{PH}} = 17.7$  Hz, P<sup>+</sup>-C-CH), 6.88–7.25 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>8</sub>H<sub>6</sub>NO).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  14.14 and 14.18 ( $2\text{OCH}_2\text{CH}_3$ ), 35.92 (s, CH<sub>2</sub>), 44.39 (d,  $^1J_{\text{PC}} = 227.2$  Hz, P<sup>+</sup>-C), 51.86 (d,  $^2J_{\text{PC}} = 13.5$  Hz, P<sup>+</sup>-C-CH), 58.92 and 61.12 ( $2\text{OCH}_2\text{CH}_3$ ), 113.19 (1C, C<sub>8</sub>H<sub>6</sub>NO), 119.97 (d,  $^3J_{\text{PC}} = 5.3$  Hz C<sub>ortho</sub>), 121.61, 123.23 and 123.40 (3C, C<sub>8</sub>H<sub>6</sub>NO), 125.77 (C<sub>para</sub>), 127.72 (1C, C<sub>8</sub>H<sub>6</sub>NO), 129.69 (C<sub>meta</sub>), 143.12 (1C, C<sub>8</sub>H<sub>6</sub>NO), 149.76 (d,  $^2J_{\text{PC}} = 7.3$  Hz, C<sub>ipso</sub>), 168.16 (d,  $^2J_{\text{PC}} = 20.6$  Hz, C=O), 169.47 (d,  $^3J_{\text{PC}} = 18.2$  Hz, C=O), 174.51 (1C, C<sub>8</sub>H<sub>6</sub>NO):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  42.90 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

**Di-tert-butyl 2-(2-oxo-2,3-dihydroindolin-1-yl)-3-(triphenoxyphosphanylidene)butanedioate (4f):** White powder, yield 85%, 0.57 g; m.p. 118–120°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1730, 1695 and 1662 (C=O). Anal. Calcd for  $\text{C}_{38}\text{H}_{40}\text{NO}_8\text{P}$  (669): C, 68.13; H, 6.02; N, 2.09. Found: C, 68.28; H, 6.11; N, 2.05.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  1.34 and 1.44 (18H, 2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 3.20 and 3.41 (2H, 2d,  $^2J_{\text{HH}} = 21.8$  Hz, CH<sub>2</sub>), 6.20 (1H, d,  $^3J_{\text{PH}} = 16.7$  Hz, P<sup>+</sup>-C-CH), 6.95–7.26 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>8</sub>H<sub>6</sub>NO).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  28.21 and 28.63 (2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 36.05 (s, CH<sub>2</sub>), 45.23 (d,  $^1J_{\text{PC}} = 224.5$  Hz, P<sup>+</sup>-C), 52.32 (d,  $^2J_{\text{PC}} = 16.2$  Hz, P<sup>+</sup>-C-CH), 78.56 and 81.16 (2 s,  $2\text{OC}(\text{CH}_3)_3$ ), 113.64 (1C, C<sub>8</sub>H<sub>6</sub>NO), 119.87 (d,  $^3J_{\text{PC}} = 5.1$  Hz,

C<sub>ortho</sub>), 121.63, 123.21 and 123.37 (3C, C<sub>8</sub>H<sub>6</sub>NO), 125.58 (C<sub>para</sub>), 127.73 (1C, C<sub>8</sub>H<sub>6</sub>NO), 129.69 (C<sub>meta</sub>), 143.26 (1C, C<sub>8</sub>H<sub>6</sub>NO), 149.77 (d,  $^2J_{\text{PC}} = 6.8$  Hz, C<sub>ipso</sub>), 167.52 (d,  $^2J_{\text{PC}} = 20.4$  Hz, C=O), 168.29 (d,  $^3J_{\text{PC}} = 19.1$  Hz, C=O), 174.72 (1C, C<sub>8</sub>H<sub>6</sub>NO):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  40.88 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

**Dimethyl 2-(2-thioxo-2,3-dihydro-1,3-benzoxazole-3-yl)-3-(triphenoxyphosphanylidene)butanedioate (4j):** White powder, yield 95%; m.p. 136–138°C, IR ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1751 and 1666 (C=O). MS, ( $m/z$ , %): 603 (M<sup>+</sup>, 2), 510 (M<sup>+</sup>-OPh, 76), 417 (M<sup>+</sup>-2OPh, 65), 310 (P(OPh)<sub>3</sub>, 50). Anal. Calcd for  $\text{C}_{31}\text{H}_{26}\text{NO}_8\text{PS}$  (603): C, 61.65; H, 4.31; N, 2.32. Found: C, 61.57; H, 4.26; N, 2.27.  $^1\text{H}$  NMR (500.1 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{H}}$  3.58 and 3.68 (6H, 2 s,  $2\text{OCH}_3$ ), 6.84 (1H, d,  $^3J_{\text{PH}} = 19.8$  Hz, P<sup>+</sup>-C-CH), 6.90–7.32 (19H<sub>arom</sub>, m, 3C<sub>6</sub>H<sub>5</sub> and C<sub>7</sub>H<sub>4</sub>NOS).  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ),  $\delta_{\text{C}}$  45.11 (d,  $^1J_{\text{PC}} = 225.2$  Hz, P<sup>+</sup>-C), 53.16 and 53.47 ( $2\text{OCH}_3$ ), 53.63 (d,  $^2J_{\text{PC}} = 11.3$  Hz, P<sup>+</sup>-C-CH), 110.28 and 110.51 (2C, C<sub>7</sub>H<sub>4</sub>NOS), 120.66 (d,  $^3J_{\text{PC}} = 4.7$  Hz, C<sub>ortho</sub>), 124.51 and 125.20 (2C, C<sub>7</sub>H<sub>4</sub>NOS), 125.84 (C<sub>para</sub>), 129.92 (C<sub>meta</sub>), 130.12 and 147.16 (2C, C<sub>7</sub>H<sub>4</sub>NOS), 150.08 (d,  $^2J_{\text{PC}} = 7.3$  Hz, C<sub>ipso</sub>), 167.33 (d,  $^3J_{\text{PC}} = 20.5$  Hz, C=O), 168.53 (d,  $^3J_{\text{PC}} = 17.9$  Hz, C=O), 179.87 (1C, C=S):  $^{31}\text{P}$  NMR (202.5 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{P}}$  42.43 ((PhO)<sub>3</sub>P<sup>+</sup>-C).

#### General procedures (exemplified by 5h)

**Dimethyl (2R<sup>\*</sup>,3S<sup>\*</sup>)-2-(2-oxobenzoxazin-1(2H)-yl)-3-(diphenoxyphosphoryl)butanedioate (5h):** To generate ester phosphonate 5 (see Scheme 2), 1 mmol or 0.51 g of compound 4a in 25 ml diethyl ether stirred magnetically a few hours it was allowed to stand for 12 hours. Next the solvent removed under reduced pressure at room temperature and compound 5 was obtained as white powder in excellent yield (93% (0.48 g). White powder, 93%, 0.48 g; m.p. = 115–118°C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1773 and 1735 (C=O), 1257 (P=O). MS, ( $m/z$ , %): 511 (M<sup>+</sup>, 18), 418 (M-OPh, 45), 77 (Ph, 34). Anal. Calcd for  $\text{C}_{25}\text{H}_{22}\text{NO}_9\text{P}$  (511): C, 58.69; H, 4.34; N, 2.74. Found: C, 58.72; H, 4.11; N, 2.78.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 3.75 and 3.85 (6H, 2 s,  $2\text{OCH}_3$ ), 4.66 (1H, dd,  $^2J_{\text{PH}} = 20.3$  Hz,  $^3J_{\text{HH}} = 11.4$  Hz, P-CH-CH), 5.08 (1H<sub>bro</sub>, P-CH-CH), 6.81–7.21 (14H<sub>arom</sub>, m,  $2\text{OC}_6\text{H}_5$  and C<sub>6</sub>H<sub>4</sub>),  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 44.81 (d,  $^1J_{\text{CP}} = 135.6$  Hz, P-CH), 53.41 and 53.74 (2 s,  $2\text{OCH}_3$ ), 53.86 (d,  $^2J_{\text{CP}} = 4.7$  Hz, P-C-CH), 109.40 and 110.38 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 120.21 and 120.29 (2d,  $^3J_{\text{PC}} = 4.4$  Hz C<sub>ortho</sub> of  $2\text{C}_6\text{H}_5$ ), 122.96 and 124.11 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 125.57 (C<sub>para</sub> of  $2\text{C}_6\text{H}_5$ ), 129.66 and 129.70 (C<sub>meta</sub> of  $2\text{C}_6\text{H}_5$ ), 130.33 and 142.83 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 149.91 and 149.63 (2d,  $^2J_{\text{CP}} = 9.3$  Hz, C<sub>ipso</sub> of  $2\text{C}_6\text{H}_5$ ), 153.71 (C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 166.52 (d,  $^2J_{\text{CP}} = 6.5$  Hz, C=O), 167.53 (d,  $^3J_{\text{CP}} = 19.2$  Hz, C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 10.06 [s, (PhO)<sub>2</sub>P(=O)].

**Diethyl (2R<sup>\*</sup>,3S<sup>\*</sup>)-2-(2-oxobenzoxazin-1(2H)-yl)-3-(diphenoxyphosphoryl)butanedioate (5i):** White powder, 90%, 0.49 g; m.p. = 124–126°C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1763 and 1735 (C=O), 1250 (P=O). MS, ( $m/z$ , %): 539 (M<sup>+</sup>, 3), 446 (M-OPh, 80), 77 (Ph, 46). Anal. Calcd for  $\text{C}_{27}\text{H}_{26}\text{NO}_9\text{P}$  (539): C, 60.09; H, 4.86; N, 2.60. Found: C, 59.83; H, 4.95; N, 2.54.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 1.20 and 1.32 (6H, 2t,  $^3J_{\text{HH}} = 7.1$  Hz,  $2\text{OCH}_2\text{CH}_3$ ), 4.22 and 4.32 (4H, 2 m,  $2\text{OCH}_2\text{CH}_3$ ), 4.62 (1H, dd,  $^2J_{\text{PH}} = 20.3$  Hz,  $^3J_{\text{HH}} = 11.5$  Hz, P-CH-CH), 5.70 (1H<sub>bro</sub>, P-CH-CH), 6.83–7.26 (14H<sub>arom</sub>, m,  $2\text{OC}_6\text{H}_5$  and C<sub>6</sub>H<sub>4</sub>),  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 13.93 (s,  $2\text{CH}_3$ ), 44.97 (d,  $^1J_{\text{CP}} = 135.1$  Hz, P-CH), 53.97 (d,  $^2J_{\text{CP}} = 4.7$  Hz, P-C-CH), 62.65 and 63.14 (2 s,  $2\text{OCH}_2\text{CH}_3$ ), 109.45 and 110.73 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 120.26 and 120.34 (2d,  $^3J_{\text{PC}} = 4.6$  Hz C<sub>ortho</sub> of  $2\text{C}_6\text{H}_5$ ), 122.86 and 124.04 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 125.49 and 125.53 (C<sub>para</sub> of  $2\text{C}_6\text{H}_5$ ), 129.63 and 129.68 (C<sub>meta</sub> of  $2\text{C}_6\text{H}_5$ ), 129.86 and 142.85 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 149.55 and 149.64 (2d,  $^2J_{\text{CP}} = 9.5$  Hz, C<sub>ipso</sub> of  $2\text{C}_6\text{H}_5$ ), 153.78 (C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 166.00 (d,  $^2J_{\text{CP}} = 7.0$  Hz, C=O), 167.97 (d,  $^3J_{\text{CP}} = 19.2$  Hz, C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 10.27 [s, (PhO)<sub>2</sub>P(=O)].

**Di-tert-butyl (2R<sup>\*</sup>,3S<sup>\*</sup>)-2-(2-oxobenzoxazin-1(2H)-yl)-3-(diphenoxyphosphoryl)butanedioate (5g):** White powder, 95%, 0.51 g; m.p. = 128–130°C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1779 and 1716 (C=O), 1250 (P=O). MS, ( $m/z$ , %): 595 (M<sup>+</sup>, 3), 502 (M-OPh, 80), 77 (Ph, 46). Anal. Calcd for  $\text{C}_{31}\text{H}_{34}\text{NO}_9\text{P}$  (595): C, 62.49; H, 5.76; N, 2.35. Found: C, 62.72; H, 5.69; N, 2.43.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 1.18 and 1.37 (18H, 2 s,  $2\text{OCMe}_3$ ), 4.22 and 4.32 (4H, 2 m,  $2\text{OCH}_2\text{CH}_3$ ), 4.64 (1H, dd,  $^2J_{\text{PH}} = 22.6$  Hz,  $^3J_{\text{HH}} = 11.5$  Hz, P-CH-CH), 5.62 (1H<sub>bro</sub>, P-CH-CH), 7.10–7.32 (14H<sub>arom</sub>, m,  $2\text{OC}_6\text{H}_5$  and C<sub>6</sub>H<sub>4</sub>),  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 27.27 and 27.70 (2 s,  $2\text{OCMe}$ ), 46.97 (d,  $^1J_{\text{CP}} = 135.0$  Hz, P-CH), 55.33 (s, P-C-CH), 83.71 and 84.24 (2 s,  $2\text{OCMe}$ ), 110.03 and 110.60 (2C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 120.60 and 120.63 (2d,  $^3J_{\text{PC}} = 4.3$  Hz C<sub>ortho</sub> of  $2\text{C}_6\text{H}_5$ ), 123.01, 124.01 and 129.32 (3C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 125.51 and 125.54 (C<sub>para</sub> of  $2\text{C}_6\text{H}_5$ ), 129.72 and 129.78 (C<sub>meta</sub> of  $2\text{C}_6\text{H}_5$ ), 142.46 (C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 150.00 and 150.11 (2d,  $^2J_{\text{CP}} = 9.2$  Hz, C<sub>ipso</sub> of  $2\text{C}_6\text{H}_5$ ), 153.60 (C, C<sub>7</sub>H<sub>4</sub>NO<sub>2</sub>), 163.64 (d,  $^2J_{\text{CP}} = 6.9$  Hz, C=O), 165.27 (C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 11.17 [s, (PhO)<sub>2</sub>P(=O)].

*Dimethyl (2R\*,3S\*)-2-(2-oxo-2,3-dihydroindol-1-yl)-3-(diphenoxyphosphoryl)butandioate (5k)*: White powder, 91%, 0.46 g; m.p. = 126–128°C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1770 and 1741 (C=O), 1265 (P=O). Anal. Calcd for  $\text{C}_{26}\text{H}_{24}\text{NO}_8$  (509): C, 61.27; H, 4.75; N, 2.75. Found: C, 60.85; H, 4.67; N, 2.81.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 3.45 (2H,  $\text{CH}_2$ ), 3.71 and 3.85 (6H, 2 s,  $2\text{OCH}_3$ ), 4.76 (1H,  $\text{H}_{\text{bro}}$ , P–CH–CH), 5.54 (1H,  $\text{H}_{\text{bro}}$ , P–CH–CH), 6.81–7.21 (14H,  $\text{H}_{\text{aro}}$ , m,  $2\text{OC}_6\text{H}_5$  and  $\text{C}_6\text{H}_4$ ),  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 35.18 (s,  $\text{CH}_2$ ), 44.51 (d,  $^1J_{\text{CP}} = 135.1$  Hz, P–CH), 52.62 (P–C– $\text{C}_{\text{bro}}$ ), 53.24 and 53.38 (2 s,  $2\text{OCH}_3$ ), 109.14 and 120.12 (2C,  $\text{C}_8\text{H}_6\text{NO}$ ), 120.40 ( $\text{C}_{\text{ortho}}$  of  $2\text{C}_6\text{H}_5$ ), 122.70 and 124.74 (2C,  $\text{C}_8\text{H}_6\text{NO}$ ), 125.57 and 125.48 ( $\text{C}_{\text{para}}$  of  $2\text{C}_6\text{H}_5$ ), 129.61 ( $\text{C}_{\text{meta}}$  of  $2\text{C}_6\text{H}_5$ ), 127.90 and 143.34 (2C,  $\text{C}_8\text{H}_6\text{NO}$ ), 149.49 and 149.66 (2d,  $^2J_{\text{CP}} = 9.6$  Hz,  $\text{C}_{\text{ipso}}$  of  $2\text{C}_6\text{H}_5$ ), 153.71 (C,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 166.95 (d,  $^2J_{\text{CP}} = 6.5$  Hz, C=O), 167.73 (d,  $^3J_{\text{CP}} = 20.0$  Hz, C=O), 175.79 (1C, N–C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 10.01 [s,  $(\text{PhO})_2\text{P}(=\text{O})$ ].

*Diethyl (2R\*,3S\*)-2-(2-oxo-2,3-dihydroindol-1-yl)-3-(diphenoxyphosphoryl)butandioate (5l)*: White powder, 88%, 0.47 g; m.p. = 145–147°C. IR (KBr) ( $\nu_{\max}$ ,  $\text{cm}^{-1}$ ): 1754 and 1746 (C=O), 1248 (P=O). Anal. Calcd for  $\text{C}_{28}\text{H}_{28}\text{NO}_8$  (537): C, 62.54; H, 5.25; N, 2.61. Found: C, 63.03; H, 5.29; N, 2.58.  $^1\text{H}$  NMR (500.1 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 0.91 and 1.30 (6H, 2t,  $^3J_{\text{HH}} = 7.0$  Hz,  $2\text{OCH}_2\text{CH}_3$ ), 3.52 (2H,  $\text{CH}_2$ ), 3.91 and 4.18 (4H, 2 m,  $2\text{OCH}_2\text{CH}_3$ ), 4.90 (1H, dd,  $^3J_{\text{PH}} = 21.4$  Hz,  $^3J_{\text{HH}} = 10.4$  Hz, P–CH–CH), 5.70 (1H,  $\text{H}_{\text{bro}}$ , P–CH–CH), 6.76–7.29 (14H,  $\text{H}_{\text{aro}}$ , m,  $2\text{OC}_6\text{H}_5$  and  $\text{C}_6\text{H}_4$ ),  $^{13}\text{C}$  NMR (125.8 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 13.49 and 13.89 (2 s,  $2\text{CH}_3$ ), 35.15 (s,  $\text{CH}_2$ ), 45.82 (d,  $^1J_{\text{CP}} = 134.6$  Hz, P–CH), 52.49 (P–C– $\text{C}_{\text{bro}}$ ), 62.29 and 62.71 (2 s,  $2\text{OCH}_2\text{CH}_3$ ), 109.23 and 119.70 (2C,  $\text{C}_8\text{H}_6\text{NO}$ ), 120.40 and 120.62 (2d,  $^3J_{\text{PC}} = 4.4$  Hz  $\text{C}_{\text{ortho}}$  of  $2\text{C}_6\text{H}_5$ ), 124.04 and 124.64 (2C,  $\text{C}_8\text{H}_6\text{NO}$ ), 125.61 and 125.65 ( $\text{C}_{\text{para}}$  of  $2\text{C}_6\text{H}_5$ ), 127.95 (C,  $\text{C}_8\text{H}_6\text{NO}$ ), 129.35 and 129.81 ( $\text{C}_{\text{meta}}$  of  $2\text{C}_6\text{H}_5$ ), 143.40 (C,  $\text{C}_8\text{H}_6\text{NO}$ ), 149.35 and 150.05 (2d,  $^2J_{\text{CP}} = 9.6$  Hz,  $\text{C}_{\text{ipso}}$  of  $2\text{C}_6\text{H}_5$ ), 153.78 (C,  $\text{C}_7\text{H}_4\text{NO}_2$ ), 165.15 (d,  $^2J_{\text{CP}} = 6.0$  Hz, C=O), 167.14 (C=O), 175.42 (1C, N–C=O).  $^{31}\text{P}$  NMR (202.4 MHz,  $\delta$ ,  $\text{CDCl}_3$ ): 11.57 [s,  $(\text{PhO})_2\text{P}(=\text{O})$ ].

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