Study of reaction between triphenylphosphine and activated acetylenic esters in the presence of benzanilide and some its derivatives

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Triphenylphosphine reacts with dialkyl acetylenedicarboxylates in the presence of NH-acids, such as benzanilide, 2-cyanobenzanilide, *N*-(2-acetylphenyl)benzamide, 3-nitrobenzanilide and methyl 2-benzamidobenzoate to generate stable phosphorus ylides. These stable ylides exist in solution as a mixture of two geometrical isomers as a result of restricted rotation around the carbon–carbon partial double bond resulting from conjugation of the ylide moiety with the adjacent carbonyl group.

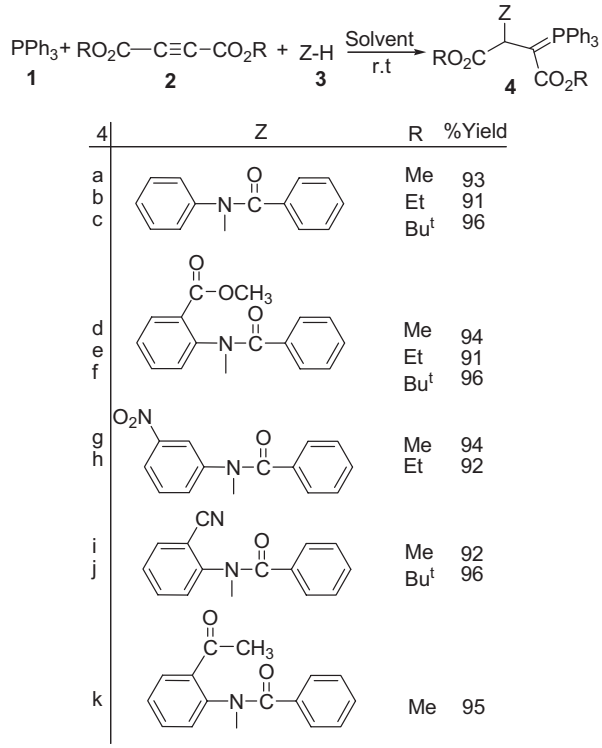
Keywords: activated acetylenic esters, NH-acids, triphenylphosphine, stable phosphorus ylides, geometrical isomers

Trivalent phosphorus compound is known to be a nucleophile, whereas it behaves as an electron donor toward good electron acceptor both in the ground and excited state.[1,2] In recent years, there has been increasing interest in the synthesis of organophosphorus compounds, that is, those bearing a carbon atom bound directly to a phosphorus atom.[3-38] This interest has resulted from the recognition of the value of such compounds in variety of biological, industrial and chemical synthetic uses.[3-7] A large number of methods have appeared describing novel synthesis of organophosphorus compounds.[6,7]

There are many studies on the reaction between trivalent phosphorus nucleophiles and α, β-unsaturated carbonyl compounds in the presence of a proton source such as alcohol or phenol.[7] As part of our current studies on the development of new routes in stable phosphorus ylides synthesis,[19-24] we describe the reaction between triphenylphosphine **1** and activated acetylenic esters **2** in the presence of benzanilide and some its derivatives **3** which leads to the corresponding stable phosphorus ylides **4** in fairly high yield (see Scheme 1).

Results and discussion

We now report a simple synthesis of stable phosphorus ylides from reaction between triphenylphosphine **1** and dialkyl acetylenedicarboxylate **2** in the presence of N-H acids **3**, such as benzanilide, 2-cyanobenzanilide, *N*-(2-acetylphenyl)benzamide, 3-nitrobenzanilide and methyl 2-benzamidobenzoate which led to **4** in fairly high yield (see Scheme 1). The reactions **4a–k** were carried out in ethyl acetate solvent at room temperature and were finished within a few minutes. The ^{1}H and ^{13}C NMR spectra of the crude product clearly indicated the formation of compounds **4a–k**. Any products other than **4a–k** could not be detected by NMR spectroscopy. The structures of compounds **4a–k** were deduced from their IR, ^{1}H, ^{13}C and ^{31}P NMR spectra. Although the presence of the ^{31}P nucleus complicates both the ^{1}H and ^{13}C NMR spectra of **4a**, it helps in assignment of the signals by long-range couplings with the ^{1}H and ^{13}C nuclei (see Experimental). The ^{1}H, ^{13}C, and ^{31}P NMR spectra of ylides **4a–k** are consistent with the presence of two isomers. The ylides moiety of these compounds are strongly conjugated with the adjacent carbonyl group and rotation around the partial double bond in (*E*)-**4** and (*Z*)-**4** geometrical isomers is slow on the NMR timescale at ambient temperature (see Scheme 2). As can be seen, only one geometrical isomer was observed for di-*tert*-butyl derivatives of **4**, presumably, because of the bulky *tert*-butyl groups. On the basis of the well established chemistry of trivalent phosphorus nucleophiles,[3-7]



Scheme 1

Scheme 2

it is reasonable to assume that phosphorus ylide **4** results from the initial addition of triphenylphosphine to the acetylenic esters and subsequent protonation of the 1 : 1 adduct by the NH-acid to form phosphoranes **4** (see Scheme 3). The ^{1}H NMR spectrum of **4a** showed four sharp lines at δ = 3.18, 3.85, 2.95 and 3.89 ppm arising from methoxy protons. Methine protons appeared as two doublet peaks at δ = 5.57 ppm (1H, d, $^3J_{PH}$ = 20.1 Hz, P–C–C*H*) and 5.66 ppm (1H, d, $^3J_{PH}$ = 18.4 Hz, P–C–C*H*) respectively for the E and Z geometrical isomers. The aromatic protons appeared as a multiplet at δ = 6.82–7.91 ppm. The ^{13}C NMR spectrum of **4a**

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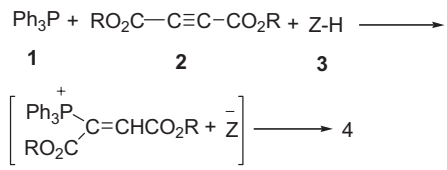

Scheme 3

displayed **46** distinct resonances in a good agreement with the mixture of two conformational isomers. The ^{1}H and ^{13}C NMR spectra of compounds **4b–k** are similar to those of **4a**, except for signals from the ester group which appear as characteristic resonance lines with the corresponding chemical shifts. The structural assignments for compounds **4a–k** were made on the basis of the ^{1}H and ^{13}C NMR spectra that were supported by their IR spectra. The carbonyl region of the spectra exhibits absorption bands for each compound. Of special interest is the ester absorption of these compounds at 1761–1612 cm^{-1}. Conjugation with negative charge accounts for the reduction of the wave unmbers of the carbonyl absorption bands.

Briefly, we have prepared novel phosphorus ylides using a one-pot reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of strong NH-acids such as benzanilide, 2-cyanobenzanilide, *N*-(2-acetylphenyl)benzamide, 3-nitrobenzanilide, and methyl 2-benzamidobenzoate. The present method carries the advantage that, not only the reaction is performed under neutral conditions, but also the substances can be mixed without any activation or modifications. Benzanilide and some of its derivatives containing phosphorus ylides **4a–k** 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 phosphoranes with variable functionalities.

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}H, ^{13}C, and ^{31}P NMR spectra were obtained from a Bruker DRX-500 AVANCE instrument with $CDCl_3$ as an solvent at 500.1, 202.5, and 125.8 MHz respectively. Elemental analyses for C, H, N were performed using a Heraeus CHN–O–Rapid analyser (for **4a**, **4b**, **4c**, **4d**, **4f**, **4g**, **4h**, **4i**, **4j** and **4k**) and also new equipment (CHNF–O–Perkin elemer 2004 II) for **4e**. In addition, the mass spectra were recorded on a Shimadzu GCMS-QP 5050A mass spectrometer operating at an ionisation potential of 70 eV. Benzanilide and some of its derivatives **3** were prepared using benzoylchlorid, aniline, 2-cyanoaniline, 3-nitroaniline, 2-aminoacetophenone and methyl-2-aminobenzoate. Dialkyl acetylenedicarboxylates and triphenylphosphine, were purchsed from Fluka, (Buchs, Switzerland) and used without further purifications.

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

Dimethyl 2-(N-phenylbenzamido)-3-(triphenylphosphanylidene) butanedioate (**4a**): To a magnetically stirred solution of triphenylphosphine (0.26 g or 1 mmol) and benzanilide (0.2 g or 1 mmol) in 10 ml of dry ethyl acetate was added, dropwise, a mixture of dimethyl acetylenedicarboxylate (0.14 g or 1 mmol) in 4 ml of dry ethyl acetate over 10 min. After a few minutes stirring at room temperature, the product was filtered and washed with cold diethyl ether (3 × 5 ml).

White powder. m.p 182–184°C, yield 0.56 g, 93%. IR (KBr) (ν_{max}, cm^{-1}) 1753, 1731 and 1620 (C=O). MS (*m/z*, %): 601 (M^{+},4), 483 (M-2CO_2Me, 34), 405 (M-$C_{13}H_{10}NO$, 23), 262 (PPh_3, 28), 183 (PPh_2, 37), 108 (PPh, 19), 77 (Ph, 100). Anal. calcd. for $C_{37}H_{32}NO_5P$ (601): C, 73.85; H, 5.36; N, 2.33%. Found: C, 74.27; H, 5.21; N, 2.40%.

Major rotamer: (%52), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 3.18 and 3.85 (6H, 2 s, 2OCH_3), 5.57 (1H, d, $^{3}J_{PH}$ = 20.1 Hz, P =C–C*H*), 6.82–7.91 (25H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 38.69 (d, $^{1}J_{PC}$ = 126.3 Hz, P =C), 49.86 and 52.53 (2OCH_3), 59.79 (d, $^{2}J_{PC}$ = 17.4 Hz, P=C–*CH*), 124.08, 126.03 and 126.76 (3C, $C_{13}H_{10}NO$), 126.81 (d, $^{1}J_{PC}$ = 91.4 Hz, C_{ipso}), 127.33, 128.01 and 128,49 (3C, $C_{13}H_{10}NO$), 128.65 (d, $^{3}J_{PC}$ = 12.7 Hz, C_{meta}), 132.19 (C_{para}), 128.75, 131.75 and 132.06 (3C, $C_{13}H_{10}NO$), 133.74 (d, $^{2}J_{PC}$ = 9.8 Hz, C_{ortho}), 137.30, 140.44 and 165.87 (3C, $C_{13}H_{10}NO$), 169.92 (N-*C*=*O*), 170.36 (d, $^{3}J_{PC}$ = 18.2 Hz, C=O ester), 172.90 (d, $^{2}J_{PC}$ = 14.2 Hz, P-C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 24.06 (Ph_3P^{+}-C).

Minor rotamer: (%48), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.95 and 3.89 (6H, 2 s, 2OCH_3), 5.66 (1H, d, $^{3}J_{PH}$ = 18.4 Hz, P =C–C*H*), 6.82–7.91 (25H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 40.97 (d, $^{1}J_{PC}$ = 134.1 Hz, P =C), 49.52 and 52.18 (2OCH_3), 60.49 (d, $^{2}J_{PC}$ = 16.5 Hz, P =C–*CH*), 124.12, 126.06 and 126.80 (3C, $C_{13}H_{10}NO$), 126.95 (d, $^{1}J_{PC}$ = 91.9 Hz, C_{ipso}), 127.28, 128.14, 128.53 and 128.76 (4C, $C_{13}H_{10}NO$), 128.80 (d, $^{3}J_{PC}$ = 12.2 Hz, C_{meta}), 131.48 and 132.04 (2C, $C_{13}H_{10}NO$), 132.12 (C_{para}), 133.55 (d, $^{2}J_{PC}$ = 9.7 Hz, C_{ortho}), 137.55, 140.97 and 165.91 (3C, $C_{13}H_{10}NO$), 168.90 (d, $^{3}J_{PC}$ = 12.9 Hz, C=O), 169.66 (N–*C*=*O*), 173.15 (d, $^{2}J_{PC}$ = 14.8 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 24.87 (Ph_3P^{+}–C).

Diethyl 2-(N-phenylbenzamido)-3-(triphenylphosphanylidene)butane dioate (**4b**): White powder, m.p 183–185°C, yield 0.57 g, 91%. IR (KBr) (ν_{max}, cm^{-1}): 1749, 1736 and 1629 (C=O). MS (*m/z*, %): 629 (M^{+}, 3), 567 (M-2OCH_3, 39), 433 (M-$C_{13}H_{10}NO$, 42), 262 (PPh_3, 33), 183 (PPh_2, 30), 108 (PPh, 17), 77 (Ph, 100). Anal. calcd. for $C_{39}H_{36}NO_5P$ (629): C, 74.37; H, 5.77; N, 2.22%. Found: C, 73.95; H, 5.79; N, 2.18%.

Major rotamer: (%53). ^{1}H NMR (500.1 Hz, $CDCl_3$ δ 0.36 and 0.97 (6H, 2t, $^{3}J_{HH}$ = 7.0 Hz, 2OCH_2CH_3), 3.47 and 4.32 (4 H, 2 m, 2ABX_3system, 2OCH_2CH_3), 5.65 (1H, d, $^{3}J_{PH}$ = 18.7 Hz, P =C–*CH*), 7.06–7.73 (25H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 14.35 and 14.44 (2OCH_2CH_3), 39.56 (d, $^{1}J_{PC}$ = 124.8 Hz, P =C), 59.78 and 59.92 (2OCH_2CH_3), 61.13 (d, $^{2}J_{PC}$ = 13.8 Hz, P =C–CH), 120.82 and 123.77(2C, $C_{13}H_{10}NO$), 125.92 (d, $^{1}J_{PC}$ = 92.0 Hz, C_{ipso}), 126.60, 127.51, 127.69 and 128.09 (4C, $C_{13}H_{10}NO$), 128.19 (d, $^{3}J_{PC}$ = 12.1 Hz, C_{meta}), 128.58 and 131.06 (2C, $C_{13}H_{10}NO$), 132.49 (C_{para}), 133.64 (d, $^{2}J_{PC}$ = 9.7 Hz, C_{ortho}), 137.67, 138.84, 141.01 and 166.06 (4C, $C_{13}H_{10}NO$), 169.39 (d, $^{3}J_{PC}$ = 13.4 Hz, C=O), 169.67(N-*C*=*O*), 172.50 (d, $^{2}J_{PC}$ = 14.6 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 25.07 (Ph_3P^{+}–C).

Minor rotamer: (%47). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 1.34 and 1.44 (6H. 2t. $^{3}J_{HH}$ = 7.1, 2OCH_2CH_3), 3.75 and 4.45 (4H, 2 m, 2ABX_3system, 2OCH_2 CH_3), 5.54 (1H, d, $^{3}J_{PH}$ = 20.5 Hz, P =C–C*H*), 7.06–7.73 (25H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 13.86 and 13.97 (2OCH_2CH_3), 40.06 (d, $^{1}J_{PC}$ = 135.1 Hz, P =C), 60.53 and 60.66 (2OCH_2CH_3), 61.24 (d, $^{2}J_{PC}$ = 14.3 Hz, P =C–*C*H), 120.68 and 123. 84 (2C, $C_{13}H_{10}NO$), 125.87 (d, $^{1}J_{PC}$ = 92.2 Hz, C_{ipso}), 126.76, 127.33, 128.09 and 128.24 (4C, $C_{13}H_{10}NO$), 128.31 (d, $^{3}J_{PC}$ = 12.0 Hz, C_{meta}), 128.77 and 131.23 (2C, $C_{13}H_{10}NO$), 132.49 (C_{para}), 133.64 (d, $^{2}J_{PC}$ = 9.7 Hz, C_{ortho}), 137.39, 138.84, 140,49 and 164.94 (4C, $C_{13}H_{10}NO$), 169.97 (N–*C*=*O*), 170.19 (d, $^{3}J_{PC}$ = 18.1 Hz, C=O), 172.16 (d, $^{2}J_{PC}$ = 14.1 Hz, P–C=C), ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 23.94 (Ph_3P^{+}–C).

Di-tert-butyl 2-(N-phenylbenzamido)-3-(triphenylphosphanylidene) butanedioate (**4c**): White powder, m.p 190–192°C, yield 0.66 g, 96%. IR (KBr) (ν_{max}, cm^{-1}) 1751, 1735 and 1629 (C=O). MS (*m/z*, %): 685 (M^{+}, 3), 584 (M–CO_2Me_3, 29), 571 (M–2CO_2CMe_3, 21), 262 (PPh_3, 64), 183 (PPh_2, 78), 108 (PPh, 56). Anal. calcd. for $C_{43}H_{44}NO_5P$ (685): C, 75.29; H, 6.47; N, 2.04%. Found: C, 75.64; H, 6.51; N, 1.98%.

Major rotamer: ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 0.82 and 1.66 (18H, 2 s, 2OCMe_3), 5.51 (1H, d, $^{3}J_{PH}$ = 19.5 Hz, P =C–*CH*) 7.04–7.70 (25H_{arom}, m, 5C_6H_5). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 28.14 and 28.48 (2OCMe_3), 38.15 (d, $^{1}J_{PC}$ = 125.6 Hz, P =C), 61.06 (d, $^{2}J_{PC}$ = 17.1 Hz, P =C–*CH*), 76.70 and 80.37 (2OCMe_3), 125.68 and 126.65 (2C, $C_{13}H_{10}NO$), 126.70 (d, $^{1}J_{PC}$ = 92.1 Hz, C_{ipso}), 127.15, 127.23, 127.76, 127.94 and 128.09 (5C, $C_{13}H_{10}NO$), 128.32 (d, $^{3}J_{PC}$ = 11.9 Hz, C_{meta}), 129.86, 130.14 and 130.40 (3C, $C_{13}H_{10}NO$), 131.77 (C_{para}), 133.78 (d, $^{2}J_{PC}$ = 9.2 Hz, C_{ortho}), 137.80 and 141.46 (2C, $C_{13}H_{10}NO$), 167.68 (d, $^{3}J_{PC}$ = 12.4 Hz, C=O), 170.91 (d, $^{2}J_{PC}$ = 14.4 Hz, P-C=C), 169.19 (N–*C*=*O*).^{31}P NMR (202.5 MHz, CDCl3):δ 23.39 (Ph_3P^{+}–C).

Dimethyl 2-[N-(2-methoxycarbonylphenyl)benzamido]-3-(triphenyl-phosphanylidene)butanedioate (**4d**): White powder. m.p 202–204°C, yield 0.60 g, 94%. IR (KBr) (ν_{max}, cm^{-1}) 1750, 1725 and 1620 (C=O). MS (*m/z*, %): 643 (M^{+}, 3), 584 (M–CO_2Me, 41), 389 (M–$C_{15}H_{12}NO3$, 27), 262 (PPh_3, 64), 183 (PPh_2, 59), 108 (PPh, 21), 77 (Ph, 100). Anal. calcd. for $C_{39}H_{34}NO_7P$ (659): C, 70.99; H, 5.20; N, 2.12%. Found: C, 70.81; H, 5.19; N, 2.20%.

Major rotamer: (%60), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.69, 3.18 and 3.91 (9H, 3 s, 3OCH_3), 5.73 (1H, d, $^{3}J_{PH}$ = 19.4 Hz, P =C–C*H*),

6.97–8.34 (24H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 40.16 (d, $^1J_{PC}$ = 127.6 Hz, P =C), 48.45, 51.25 and 52.22 (3OCH_3), 60.01 (d, $^2J_{PC}$ = 18.1 Hz, P =C–*CH*), 126.04 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 127.34 and 128.02 (2C, $C_{13}H_9NO$), 128.46 (C_{meta}), 128.76, 129.14, 130.06 and 130.99 (4C, $C_{13}H_9NO$), 131.79 (d, $^4J_{PC}$ = 2.2 Hz, C_{para}), 131.98 (1C, $C_{13}H_{10}NO$), 133.70 (d, $^2J_{PC}$ = 9.6 Hz, C_{ortho}), 134.83, 137.58, 142.19, 164.71 and 165.69 (5C, $C_{13}H_9NO$), 169.06 (N–*C*=*O*), 169.47 (d, $^3J_{PC}$ = 13.9 Hz, C=O ester), 174.01 (d, $^2J_{PC}$ = 12.7 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.00 (Ph_3P^+-C).

Minor rotamer: (%40), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 3.04, 3.16 and 3.85 (9H, 3 s, 3OCH_3), 5.88 (1H, d, $^3J_{PH}$ = 21.2 Hz, P =C–*CH*), 6.97–8.34 (24H_{arom}, m, 5C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 41.48 (d, $^1J_{PC}$ = 134.7 Hz, P =C), 49.28, 52.18 and 51.96 (3OCH_3), 59.92 (d, $^2J_{PC}$ = 18.4 Hz, P =C–*CH*), 127.25 (1C, $C_{13}H_9NO$), 126.19 (d, $^1J_{PC}$ = 91.8 Hz, C_{ipso}), 128.24 (1C, $C_{13}H_9NO$), 128.62 (C_{meta}), 128.83, 129.02, 130.38 and 131.07 (4C, $C_{13}H_9NO$), 131.79 (d, $^4J_{PC}$ = 2.2 Hz, C_{para}), 132.12 (1C, $C_{13}H_9NO$), 133.85 (d, $^2J_{PC}$ = 9.8 Hz, C_{ortho}), 134.66, 137.75, 141.84, 164.42 and 165.34 (5C, $C_{13}H_9NO$), 169.06 (N–*C*=*O*), 170.09 (d, $^3J_{PC}$ = 9.1 Hz, C=O), 174.05 (d, $^2J_{PC}$ = 12.3 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.48 (Ph_3P^+-C).

Diethyl 2-[N-(2-methoxycarbonylphenyl)benzamido]-3-(triphenylphosphanylidene)butanedioate (**4e**): White powder, m.p 141–143°C, yield 0.63 g, 91%. IR (KBr) (ν_{max}, cm^{-1}): 1758, 1690 and 1656 (C=O). MS (*m/z*, %): 687 (M^+, 4), 629 (M–2Et, 30), 628 (M–OCH_3, 40), 614 (M–CO_2Et, 53), 262 (PPh_3, 67), 183 (PPh_2, 77), 108 (PPh, 37), 77 (Ph, 100). Anal. calcd. for $C_{41}H_{38}NO_7P$ (687): C, 71.58; H, 5.57; N, 2.04%. Found: C, 71.97; H, 5.57; N, 1.97%.

Major rotamer: (%61). ^{1}H NMR (500.1 Hz, $CDCl_3$ δ 0.24 and 1.39 (6H, 2t, $^3J_{HH}$ = 7.1 Hz, 2OCH$_2CH_3$), 3.15 (1 s, OCH_3), 3.47 and 4.30 (4 H, 2 m, 2ABX$_3$system, 2OCH_2CH$_3$), 5.73 (1H, d, $^3J_{PH}$ = 19.6 Hz, P =C–*CH*), 6.86–8.44 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 13.82 and 14.47 (2OCH$_2CH_3$), 40.13 (d, $^1J_{PC}$ = 125.9 Hz, P =C), 51.23 (OCH_3), 57.23 (OCH_2CH$_3$), 61.00 (d, $^2J_{PC}$ = 18.2 Hz, P =C–*C*H), 61.05 (OCH_2CH$_3$), 126.22(1C, $C_{13}H_9NO$), 126.67 (d, $^1J_{PC}$ = 91.9 Hz, C_{ipso}), 127.24, 127.98 and 128.19 (3C, $C_{13}H_9NO$), 128.40 (d, $^3J_{PC}$ = 11.9 Hz, C_{meta}), 128.73, 129.22, 129.91, 130.04 and 131.14 (5C, $C_{13}H_9NO$), 131.87 (C_{para}), 133.64 (d, $^2J_{PC}$ = 9.3 Hz, C_{ortho}), 134.81, 137.72 and 142.22(3C, $C_{13}H_9NO$), 164.71(CH_3O–*C*=*O*), 168.95 (d, $^3J_{PC}$ = 13.9 Hz, C=O), 170.14 (N–*C*=*O*), 173.38 (d, $^2J_{PC}$ = 12.6 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 25.68 (Ph_3P^+–C).

Minor rotamer: (%39). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 1.09 and 1.42 (6H. 2t. $^3J_{HH}$ = 7.1, 2OCH$_2CH_3$), 3.01(1 s, OCH_3), 4.21 and 4.44 (4H, 2 m, 2ABX$_3$system, 2OCH_2CH$_3$), 5.85 (1H, d, $^3J_{PH}$ = 21.4 Hz, P =C–*CH*), 6.86–8.44 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 14.40 and 14.76 (2OCH$_2CH_3$), 40.27 (d, $^1J_{PC}$ = 134.8 Hz, P =C), 51.18 (OCH_3), 57.60 (OCH_2CH$_3$), 59.91 (d, $^2J_{PC}$ = 18.3 Hz, P =C–*C*H), 61.02 (OCH_2CH$_3$), 126.30 (1C, $C_{13}H_9NO$), 126.85 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 127.29 (1C, $C_{13}H_9NO$), 128.54 (d, $^3J_{PC}$ = 12.0 Hz, C_{meta}), 128.93, 128.26, 128.73, 129.33, 129.91, 130.12 and 131.19 (7C, $C_{13}H_9NO$), 131.87 (C_{para}), 133.85 (d, $^2J_{PC}$ = 9.1 Hz, C_{ortho}), 134.88, 137.85 and 142.93 (3C, $C_{13}H_9NO$), 164.46 (CH_3O–*C*=*O*), 169.04 (d, $^3J_{PC}$ = 13.2 Hz, C=O), 169.88 (N–*C*=*O*), 173.51 (d, $^2J_{PC}$ = 12.5 Hz, P–C=C), ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.68 (Ph_3P^+–C).

Di-tert-butyl 2-[N-(2-methoxycarbonylphenyl)benzamido]-3-(triphenylphosphanylidene)butanedioate (**4f**): White powder, m.p 47–149°C, yield 0.71 g, 96%. IR (KBr) (ν_{max}, cm^{-1}) 1761, 1737 and 1624 (C=O). MS (*m/z*, %): 743 (M^+, 5), 684 (M–CO_2Me, 47), 560 (M–PPh_2, 20), 489 (M–$C_{15}H_{12}NO_3$, 17), 262 (PPh_3, 80), 183 (PPh_2, 73), 108 (PPh, 57). Anal. calcd. for $C_{45}H_{46}NO_7P$ (743): C, 72.64; H, 6.24; N, 1.88%. Found: C, 72.95; H, 6.29; N, 2.01%.

Major rotamer: ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 0.69 and 1.63 (18H, 2 s, 2OCMe_3), 5.47 (1H, d, $^3J_{PH}$ = 20.2 Hz, P =C–*CH*) 6.93–8.56 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 28.07 and 28.55 (2OCMe_3), 39.21 (d, $^1J_{PC}$ = 127.4 Hz, P =C), 51.28 (OMe), 61.95 (d, $^2J_{PC}$ = 18.4 Hz, P =C-*CH*), 76.69 and 80.34 (2O*C*Me$_3$), 126.36 and 126.42 (2C, $C_{13}H_9NO$), 126.51 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 127.43 (1C, $C_{13}H_9NO$), 128.13 (d, $^3J_{PC}$ = 11.8 Hz, C_{meta}), 128.60, 129.96, and 131.58 (3C, $C_{13}H_9NO$), 133.15 (C_{para}), 133.87 (d, $^2J_{PC}$ = 9.2 Hz, C_{ortho}), 134.87, 137.73 and 142.59 (3C, $C_{13}H_9NO$), 164.80 (MeO–*C*=*O*), 168.08 (d, $^3J_{PC}$ = 13.3 Hz, C=O), 169.87 (N–*C*=*O*), 171.68 (d, $^2J_{PC}$ = 12.4 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$):δ 25.06 (Ph_3P^+–C).

Dimethyl 2-[N-(2-nitrophenyl)benzamido]-3-(triphenylphosphanylidene)butanedioate (**4g**): Yellow powder. m.p 198–200°C, yield 0.61 g, 94%. IR (KBr) (ν_{max}, cm^{-1}) 1750, 1734 and 1632 (C=O). MS (*m/z*, %): 646 (M^+, 3), 615 (M–OMe, 44), 528 (M–2CO_2Me), 405 (M–$C_{13}H_9N_2O3$, 27), 262 (PPh_3, 60), 183 (PPh_2, 40), 108 (PPh, 27), 77 (Ph, 100). Anal. calcd. for $C_{37}H_{31}N_2O_7P$ (646): C, 68.70; H, 4.83; N, 4.33%. Found: C, 68.98; H, 4.78; N, 4.37%.

Major rotamer: (%60), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.91 and 3.89 (6H, 2 s, 2OCH_3), 5.58 (1H, d, $^3J_{PH}$ = 20.5 Hz, P =C–*CH*), 7.08–8.04 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 38.85 (d, $^1J_{PC}$ = 123.9 Hz, P =C), 49.09 and 52.72 (2OCH_3), 60.01 (d, $^2J_{PC}$ = 18.1 Hz, P =C–*CH*), 121.22, 121.78 and 125.60 (3C, $C_{13}H_9NO$), 125.91 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 127.84 and 127.96 (2C, $C_{13}H_9NO$), 128.89 (d, $^3J_{PC}$ = 12.0 Hz, C_{meta}), 129.23 (1C, $C_{13}H_9NO$), 132.45 (C_{para}), 133.40 (d, $^2J_{PC}$ = 9.7 Hz, C_{ortho}), 136.26, 136.33, 137.08, 142.21, 142.50 and 147.04 (6C, $C_{13}H_9NO$), 169.06 (N–*C*=*O*), 169.73 (d, $^3J_{PC}$ = 18.5 Hz, C=O ester), 172.59 (d, $^2J_{PC}$ = 13.4 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 23.96 (Ph_3P^+-C).

Minor rotamer: (%40), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 3.12 and 3.82 (6H, 2 s, 2OCH_3), 5.64 (1H, d, $^3J_{PH}$ = 18.9 Hz, P =C–*CH*), 7.08–8.04 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 40.89 (d, $^1J_{PC}$ = 133.7 Hz, P =C), 49.72 and 52.45 (2OCH_3), 59.92 (d, $^2J_{PC}$ = 18.4 Hz, P =C–*CH*), 121.26, 121.84 and 125.66 (3C, $C_{13}H_9NO$), 126.37 (d, $^1J_{PC}$ = 91.7 Hz, C_{ipso}), 127.79 and 128.02 (2C, $C_{13}H_9NO$), 129.05 (d, $^3J_{PC}$ = 11.6 Hz, C_{meta}), 132.45 (C_{para}), 133.61 (d, $^2J_{PC}$ = 9.9 Hz, C_{ortho}), 136.21, 136.37, 136.14, 137.14, 142.25, 142.56 and 147.08 (7C, $C_{13}H_9NO$), 169.06 (N–*C*=*O*), 169.03 (d, $^3J_{PC}$ = 12.8 Hz, C=O), 169.97 (d, $^2J_{PC}$ = 18.3 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 25.14 (Ph_3P^+-C).

Diethyl 2-[N-(2-nitrophenyl)benzamido]-3-(triphenyl phosphanylidene) butanedioate (**4h**): Yellow powder, m.p 179–181°C, yield 0.62 g, 92%. IR (KBr) (ν_{max}, cm^{-1}): 1745, 1731 and 1617 (C=O). MS (*m/z*, %): 674 (M^+, 3), 601 (M–CO_2Et, 38), 584 (M–2OEt, 31), 433 (M–$C_{13}H_9N_2O_3$t, 26), 262 (PPh_3, 53), 183 (PPh_2, 80), 108 (PPh, 45), 77 (Ph, 100). Anal. calcd. for $C_{39}H_{35}N_2O_5P$ (674): C, 69.41; H, 5.23; N, 4.15%. Found: C, 70.01; H, 5.15; N, 4.20%.

Major rotamer: (%65). ^{1}H NMR (500.1 Hz, $CDCl_3$ δ 0.34 and 1.39 (6H, 2t, $^3J_{HH}$ = 7.1 Hz, 2OCH$_2CH_3$), 3.45 and 4.3o (4 H, 2 m, 2ABX$_3$system, 2OCH_2CH$_3$), 5.62 (1H, d, $^3J_{PH}$ = 18.7 Hz, P =C–*CH*), 7.08–8.04 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 13.89 and 14.38 (2OCH$_2CH_3$), 39.07 (d, $^1J_{PC}$ = 125.7 Hz, P =C), 57.75 (OCH_2CH$_3$), 60.63 (d, $^2J_{PC}$ = 17.7 Hz, P =C–*C*H), 61.36 (OCH_2CH$_3$), 115.43, 121.26 and 121.76 (3C, $C_{13}H_9NO$), 126.63 (d, $^1J_{PC}$ = 91.8 Hz, C_{ipso}), 127.64, 127.78, 127.98 and 128.42 (4C, $C_{13}H_9NO$), 128.81 (d, $^3J_{PC}$ = 12.4 Hz, C_{meta}), 129.19 and 129.82 (2C, $C_{13}H_9NO$), 132.37 (C_{para}), 133.52 (d, $^2J_{PC}$ = 9.7 Hz, C_{ortho}), 133.71, 142.26 and 142.56 (3C, $C_{13}H_9NO$), 169.63 (N–*C*=*O*), 169.92 (d, $^3J_{PC}$ = 17.8 Hz, C=O), 172.08 (d, $^2J_{PC}$ = 13.8 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 24.01 (Ph_3P^+–C).

Minor rotamer: (%35). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 1.27 and 1.42 (6H. 2t. $^3J_{HH}$ = 7.1, 2OCH$_2CH_3$), 4.16 and 4.37 (4H, 2 m, 2ABX$_3$system, 2OCH_2CH$_3$), 5.53 (1H, d, $^3J_{PH}$ = 21.7 Hz, P =C–*CH*), 7.08–8.04 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 14.27 and 14.38 (2OCH$_2CH_3$), 40.98 (d, $^1J_{PC}$ = 132.5 Hz, P =C), 58.16 (OCH_2CH$_3$), 60.02 (d, $^2J_{PC}$ = 17.3 Hz, P =C–*C*H), 61.27 (OCH_2CH$_3$), 116.21, 121.54 and 122.03 (3C, $C_{13}H_9NO$), 126.24 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 126.42, 127.91, 128.18 and 128.64 (4C, $C_{13}H_9NO$), 128.89 (d, $^3J_{PC}$ = 12.6 Hz, C_{meta}), 129.37 and 130.11 (2C, $C_{13}H_9NO$), 132.37 (C_{para}), 133.65 (d, $^2J_{PC}$ = 9.7 Hz, C_{ortho}), 133.84, 142.48 and 142.93 (3C, $C_{13}H_9NO$), 168.53 (d, $^3J_{PC}$ = 13.1 Hz, C=O), 169.88 (N–*C*=*O*), 171.83 (d, $^2J_{PC}$ = 16.6 Hz, P–C=C), ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 25.50 (Ph_3P^+–C).

Dimethyl 2-[N-(2-cyanophenyl)benzamido]-3-(triphenylphosphanylidene) butanedioate (**4i**): Yellow powder. m.p 205–207°C, yield 0.58 g, 92%. IR (KBr) (ν_{max}, cm^{-1}) 1729, 1641 and 1612 (C=O). MS (*m/z*, %): 626 (M^+, 5), 595 (M–OMe, 29), 508 (M–2CO_2Me, 32), 262 (PPh_3, 78), 183 (PPh_2, 86), 108 (PPh, 54), 77 (Ph, 100). Anal. calcd. for $C_{38}H_{31}N_2O_5P$ (626): C, 72.81; H, 4.99; N, 4.47%. Found: C, 72.65; H, 5.04; N, 4.53%.

Major rotamer: (%65), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.81 and 3.92 (6H, 2 s, 2OCH_3), 5.56 (1H, d, $^3J_{PH}$ = 19.0 Hz, P =C–*CH*), 7.09–8.45 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 40.23 (d, $^1J_{PC}$ = 125.3 Hz, P =C), 48.80 and 52.70 (2OCH_3), 61.63 (d, $^2J_{PC}$ = 17.6 Hz, P =C–*CH*), 114.48 (1C, $C_{13}H_9NO$), 117.71 (CN), 122.22, 124.55 and 125.20 (3C, $C_{13}H_9NO$), 125.84 (d, $^1J_{PC}$ = 92.0 Hz, C_{ipso}), 127.73 and 128.60 (2C, $C_{13}H_9NO$), 128.84 (d, $^3J_{PC}$ = 12.5 Hz, C_{meta}), 129.18 (1C, $C_{13}H_9NO$), 132.26 (C_{para}), 133.55 (d, $^2J_{PC}$ = 9.5 Hz,

C_{ortho}), 133.87, 136.37, 144.21 and 165.62 (4C, $C_{13}H_9NO$), 169.22 (d, $^3J_{PC}$ = 13.4 Hz, C=O ester), 169.76 (N–*C*=*O*), 173.36 (d, $^2J_{PC}$ = 13.7 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.10 (Ph_3P^+-C).

Minor rotamer: (%35), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 3.24 and 3.87 (6H, 2 s, 2O*CH*$_3$), 5.68 (1H, d, $^3J_{PH}$ = 20.5 Hz, P =C–C*H*), 7.09–8.45 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 41.68 (d, $^1J_{PC}$ = 132.4 Hz, P =C), 49.40 and 52.51 (2O*CH*$_3$), 60.53 (d, $^2J_{PC}$ = 18.6 Hz, P =C–*CH*), 115.67 (1C, $C_{13}H_9NO$), 117.67 (CN), 122.28, 125.28 and 125.39 (4C, $C_{13}H_9NO$), 125.96 (d, $^1J_{PC}$ = 91.2 Hz, C_{ipso}), 127.41 and 128.70 (2C, $C_{13}H_9NO$), 128.75 (d, $^3J_{PC}$ = 12.1 Hz, C_{meta}), 129.78 (1C, $C_{13}H_9NO$), 132.26 (C_{para}), 132.51(1C, $C_{13}H_9NO$), 133.81 (d, $^2J_{PC}$ = 9.8 Hz, C_{ortho}), 134.10, 136.42, 143.49 and 165.39 (4C, $C_{13}H_9NO$), 169.70 (N–*C*=*O*), 169.84 (d, $^3J_{PC}$ = 12.1 Hz, C=O), 173.44 (d, $^2J_{PC}$ = 12.9 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.46 (Ph_3P^+–C).

Di-tert-butyl 2-[N-(2-cyanophenyl)benzamido]-3-(triphenylphosphanylidene)butanedioate (**4j**): White powder, m.p 189–191°C, yield 0.71 g, 96%. IR (KBr) (ν_{max}, cm^{-1}) 1741, 1735 and 1620 (C=O). MS (*m/z*, %): 710 (M^+, 4), 609 (M–CO_2CMe_3, 34), 560 (M–2OCMe_3, 53), 262 (PPh_3, 77), 183 (PPh_2, 80), 108 (PPh, 69). Anal. calcd. for $C_{44}H_{43}N_2O_5P$ (710): C, 74.33; H, 6.10; N, 3.94%. Found: C, 74.68; H, 5.99; N, 4.02%.

Major rotamer: ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 0.74 and 1.63 (18H, 2 s, 2O*CMe*$_3$), 5.38 (1H, d, $^3J_{PH}$ = 19.7 Hz, P =C–*CH*), 7.04–8.61 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ 28.07 and 28.47 (2O*CMe*$_3$), 39.58 (d, $^1J_{PC}$ = 126.0 Hz, P =C), 62.55 (d, $^2J_{PC}$ = 17.9 Hz, P =C–*CH*), 76.96 and 80.61 (2O*C*Me$_3$), 114.48 (1C, $C_{13}H_9NO$), 117.91 (CN), 121.73 and 124.45 (3C, $C_{13}H_9NO$), 126.62 (d, $^1J_{PC}$ = 91.8 Hz, C_{ipso}), 126.85 and 127.33 (2C, $C_{13}H_9NO$), 128.52 (d, $^3J_{PC}$ = 12.1 Hz, C_{meta}), 129.01 and 129.16 (2C, $C_{13}H_9NO$), 132.01 (C_{para}), 132.32 (1C, $C_{13}H_9NO$), 132.84 (d, $^2J_{PC}$ = 9.2 Hz, C_{ortho}), 133.18, 133.67, 136.60 and 144.67 (4C, $C_{13}H_9NO$), 167.88 (d, $^3J_{PC}$ = 12.6 Hz, C=O), 169.32 (N–*C*=*O*), 171.29 (d, $^2J_{PC}$ = 13.4 Hz, P–C=C). ^{31}P NMR (202.5 MHz, $CDCl_3$):δ 25.19 (Ph_3P^+–C).

Dimethyl 2-[N-(2-acetylphenyl)benzamido-3-(triphenylphosphanylidene)butanedioate (**4k**): Yellow powder. m.p 150–152°C, yield 0.61 g, 95%. IR (KBr) (ν_{max}, cm^{-1}) 1743, 1634 and 1618 (C=O). MS (*m/z*, %): 643 (M^+, 4), 581 (M–2OMe, 21), 525 (M–2CO_2Me, 36), 262 (PPh_3, 33), 183 (PPh_2, 38), 108 (PPh, 100). Anal. calcd. for $C_{39}H_{34}NO_6P$ (643): C, 72.75; H, 5.32; N, 2.18%. Found: C, 73.12; H, 5.38; N, 2.22%.

Major rotamer: (%56), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.73 (1H, s, CH_3), 3.07 and 3.80 (6H, 2 s, 2O*CH*$_3$), 5.72 (1H, d, $^3J_{PH}$ = 19.4 Hz, P =C–C*H*), 7.00–8.38 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 28.66 (CH_3), 40.08 (d, $^1J_{PC}$ = 123.9 Hz, P =C), 48.48 and 52.35 (2O*CH*$_3$), 61.22 (d, $^2J_{PC}$ = 16.8 Hz, P =C–*CH*), 120.79, 122.53, 125.15 and 127.48 (4C, $C_{13}H_9NO$), 127.63 (d, $^1J_{PC}$ = 90.2 Hz, C_{ipso}), 128.22 (1C, $C_{13}H_9NO$), 128.50 (d, $^3J_{PC}$ = 12.1 Hz, C_{meta}), 129.01, 130.15, 131.03, 131.88 and 133.24 (5C, $C_{13}H_9NO$), 133.41 (C_{para}), 134.15 (d, $^2J_{PC}$ = 9.0 Hz, C_{ortho}), 141.43 and 166.13 (2C, $C_{13}H_9NO$), 169.35 (d, $^3J_{PC}$ = 13.6 Hz, C=O ester), 169.62 (N–*C*=*O*), 172.18 (d, $^2J_{PC}$ = 13.9 Hz, P–C=C), 203.33 (O=C–CH_3). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.11 (Ph_3P^+–C).

Minor rotamer: (%44), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ 2.71 (1H, s, CH_3), 3.82 and 3.88 (6H, 2 s, 2O*CH*$_3$), 5.89 (1H, d, $^3J_{PH}$ = 20.0 Hz, P =C–C*H*), 7.00–8.38 (24H_{arom}, m, 4C_6H_5 and C_6H_4). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 28.66 (CH_3), 41.25 (d, $^1J_{PC}$ = 135.4 Hz, P =C), 49.36 and 52.35 (2O*CH*$_3$), 60.41 (d, $^2J_{PC}$ = 16.5 Hz, P =C–*CH*), 120.63, 121.96 and 125.19 (3C, $C_{13}H_9NO$), 126.83 (d, $^1J_{PC}$ = 91.3 Hz, C_{ipso}), 127.54 and 128.42 (2C, $C_{13}H_9NO$), 128.50 (d, $^3J_{PC}$ = 12.1 Hz, C_{meta}), 129.27, 130.33, 131.22, 131.92 and 133.05 (5C, $C_{13}H_9NO$), 133.41 (C_{para}), 134.15 (d, $^2J_{PC}$ = 9.0 Hz, C_{ortho}), 140.73 and 165.38 (2C, $C_{13}H_9NO$), 168.76 (d, $^3J_{PC}$ = 14.2 Hz, C=O), 169.45 (N–*C*=*O*), 171.92 (d, $^2J_{PC}$ = 14.0 Hz, P–C=C), 203.33 (O=C–CH_3). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 26.52 (Ph_3P^+–C).

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