Water–acetone media enforced chemoselective synthesis of 2-substituted pyrrole stable phosphorus ylides from reaction between pyrrole and acetylenic esters in the presence of triphenylphosphine

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Pyrrole undergoes a smooth reaction with dialkyl acetylenedicarboxylates in the presence of triphenylphosphine in a mixture of water–acetone (50:50) as a solvent pathway to produce phosphorus ylides of 2-substituted pyrrole in good yield.

Keywords: stable phosphorus ylides, acetylenic ester, 2-substituted pyrrole, triphenylphosphine, one geometrical rotamer

Nitrogen-containing heterocyclic compounds such as pyrrole and its derivatives are important in organic chemistry since their structures can be found in many natural or therapeutic compounds.[1] In recent years, the syntheses of organophosphorus compounds,[2-6] have been the subject of great interest.

This interest has resulted from the recognition of the value of such compounds in a variety of biological, industrial, and chemical synthetic systems.[7] A large number of methods have been introduced to describe the novel syntheses of organophosphorus compounds. In the relevant synthesis, the successful attack by nucleophilic trivalent phosphine on the carbon atom is facilitated when the latter is conjugated with a carbonyl group, or when it is the specified part of an unsaturated bond otherwise unactivated.[2-13]

There are many systematic investigations on the synthesis of the reactions between trivalent phosphorus nucleophiles and α,β-unsaturated carbonyl compounds in the presence of a proton source such as alcohols or CH-acids.[2,13-24]

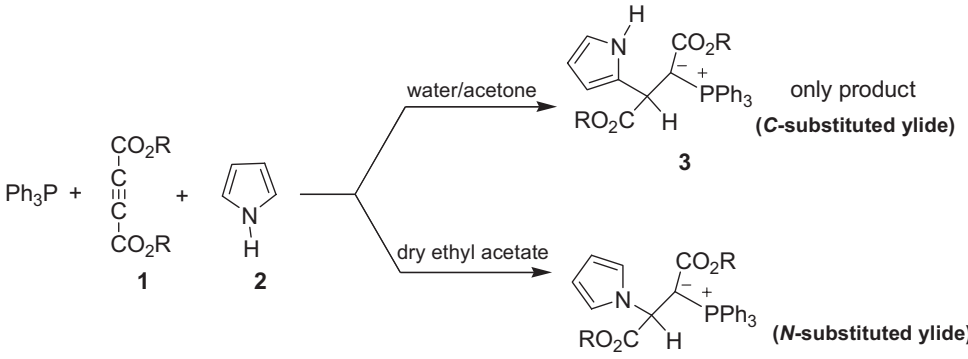
Results and discussion

Recently we have reported synthesis of *N*-substituted stable phosphorus ylides of pyrrole in ethyl acetate.[19] Herein we report a simple one-pot synthesis of 2-substituted stable crystalline phosphorus ylides **3** (*C*-substitued ylide).[15-21]

Because of the importance of pyrrole moiety and its derivatives in biological activity and organic polymers and also its antibiotic property,[25,26] for the present work generation of stable phosphorus ylides was undertaken in a mixture of water-acetone (50:50) in comparison with dry ethyl acetate solvent.[20] In an aqueous-organic solution the outlined reaction of triphenylphosphine with dialkyl acetylenedicarboxylates **1** in the presence of NH-acid **2** led to the corresponding ylides **3** in excellent yields (see Scheme 1).

On the basis of the well established chemistry of phosphorus nucleophiles[2-4] it is reasonable to assume that ylides **3** results from initial addition of triphenylphosphine to dialkyl acetylenedicarboxylates and concomitant protonation of the reactive 1 : 1 adduct, to generate an ion pair intermediate (see Scheme 2) which is subsequently attacked by the carbon of conjugated base the pyrrole.

The structure **3** was assigned to the isolated products on the basis of assessment of their IR, ^{1}H NMR, ^{13}C NMR spectra and mass spectral data. The NMR spectroscopy was used to distinguish the structure **3** and **5**. Compounds **3a**–**c** show one geometrical rotamer because of the intraction between



5

3, 5	R	% Yield of 3 and 5		(M	m)[a]
a	Me	93	95	68	32
b	Et	92	93	71	29
c	Bu^t	96	96	98	2

a: These Major and minor are only for compound **5**

Scheme 1

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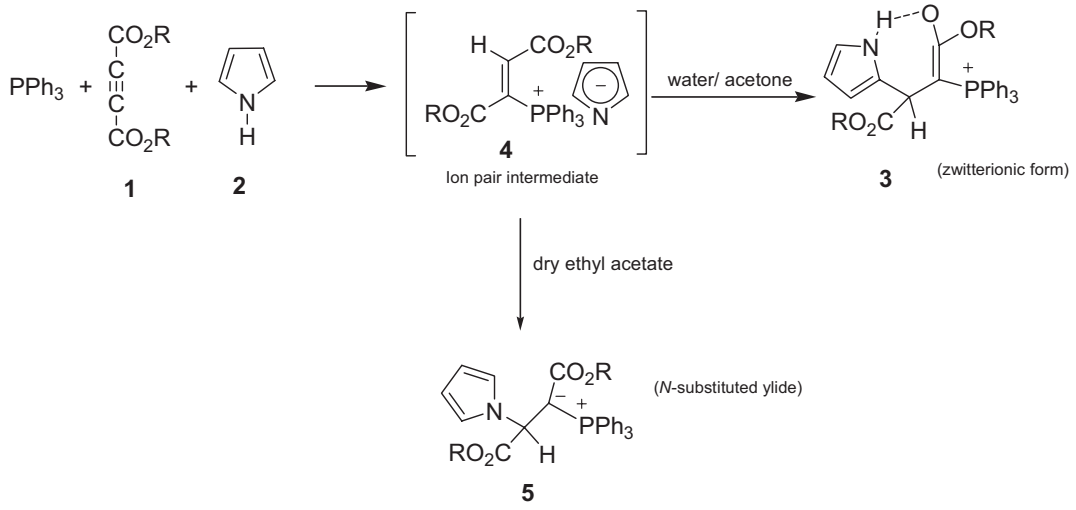
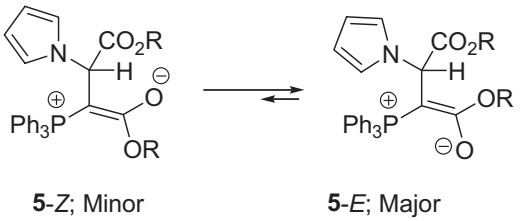


Scheme 2



Scheme 3

hydrogen of pyrrole and negative oxygen of ylide moiety (see Scheme 1 and 2); but in compounds **5a**–**c** two geometrical rotamer was observed in corresponding with these structures because the ylide moiety of these compounds is strongly conjugated with the adjacent carbonyl group and rotation about the partial double bond in (*E*)-**5** and (*Z*)-**5** geometrical isomers is slow on the NMR timescale at ambient temperature (Scheme 3). Any product other than **3** and **5** could not be detected by the NMR spectroscopy. The structures of compounds **3a–c** and **5a–c** were deduced from their IR, ^{1}H, ^{13}C, and ^{31}P NMR spectra. The mass spectra of these stable phosphorus ylides displayed molecular ion peaks at appropriate *m/z* values. Any initial fragmentation involves loss of the side chains.

In summary, we have prepared the novel pyrrole-containing phosphorus yield using a one-pot reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of NH-acid such as pyrrole in a mixture of aqueous-organic media (water–acetone, 50:50) (**3a–c**) and dry ethyl acetate (**5a–c**). The present method, carries the advantage that, not only the reaction is performed under the neutral conditions, but also the substances can be mixed without any activation or modification. Furthermore, pyrrole-containing stable phosphorus ylides **3a–c** and **5a–c** may be considered as the potentially useful synthetic intermediates. It seems that the procedure described here may be employed as an acceptable method for the preparation of 2-substituted and *N*-substituted pyrrole stable phosphorus ylides with variable functionalities.

Experimental

Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a Shimadzu IR-460 spectrometer respectively. Elemental analyses for C, H, and N were performed using a Heraeus CHN–O–Rapid analyser. The ^{1}H, ^{13}C, and ^{31}P NMR spectra were obtained from a Bruker DRX-500 Avence instrument with $CDCl_3$ as solvent at 500.1, 125.8, and 202.4 MHz respectively. The mass spectra were recorded on a Shimadzu GC/MS QP 1100 EX mass spectrometer operating at an ionisation potential of 70 eV. Triphenylphosphine, dialkyl acetylenedicarboxylates (**1a–c**) and pyrrole (**2**) were obtained from Fluka and used without further purification.

General procedures (exemplified by **3a***)*

Dimethyl 2-(1H-pyrrol-2-yl)-3-(triphenylphosphoranylidene)butanedioate (**3a**): To a magnetically stirred solution of triphenylphosphine (0.26 g, 1 mmol) and pyrrole (0.67 g, 1 mmol) in 8 ml of acetone/H_2O (50:50) was added dropwise a mixture of dimethyl acetylendicarboxylate (0.14 g, 1 mmol) in 3 ml of acetone at –5°C over 10 min. After 8 hours stirring at room temperature, the product was filtered and recrystallised from ethyl acetate solvent. The product **3a** was obtained as:

Light yellow needles, 0.45 g, yield 93%, m.p. 146–148°C; IR (KBr) ν_{max} 1621 and 1720 (C=O), 1600 (C=C) cm^{-1}. MS (*m/z*,%): 471 (M, 4), 412 (M–CO_2Me, 87), 288 (M–PPh_2, 3), 262 (PPh_3, 100), 183 (PPh_2, 19), 108 (PPh, 14), 77 (Ph, 4). Anal. Calcd for $C_{28}H_{26}NO_4P$ (471.50) C, 71.33; H, 5.56; N, 2.97%, Found: C, 70.85; H, 5.55; N, 2.95%. ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 3.14 and 3.67 (6H, 2 s, 2 OCH_3), 3.55 (1H, d, $^{3}J_{HP}$ = 17.5 Hz, P–C–C*H*), 5.25 (1H, br s, C_4H_4N), 5.95 (1H, dd, J_1 = 5.5, J_2 = 2.6 Hz, C_4H_4N), 6.70 (1H, d, J = 1.5 Hz, C_4H_4N), 7.43–7.72 (15H, m, 3 C_6H_5), 10.24 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 43.18 (d, $^{2}J_{CP}$ = 13.4 Hz, P–C–*C*H), 44.93 (d, $^{1}J_{CP}$ = 125.0 Hz, P–C), 49.28 and 52.28 (2 s, 2 OCH_3), 104.44, 105.94 and 116.76 (3C, C_4H_4N), 127.04 (d, $^{1}J_{CP}$ = 91.7 Hz, C_{ipso}), 128.67 (d, $^{3}J_{CP}$ = 11.9 Hz, C_{meta}), 131.98 (d, $^{4}J_{CP}$ = 2.5 Hz, C_{para}), 133.76 (d, $^{2}J_{CP}$ = 9.7 Hz, C_{ortho}), 134.05 (d, $^{3}J_{CP}$ = 3.5 Hz, Cα, C_4H_4N), 171.30 (d, $^{2}J_{CP}$ = 13.1 Hz, C=O), 175.31 (d, $^{3}J_{CP}$ = 9.0 Hz, C=O). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.52 (s, Ph_3P^{+}-C).

Diethyl 2-(1H pyrrol-2-yl)-3-(triphenylphosphoranylidene)butanedioate (**3b**): Light yellow crystal, 0.46 g, yield 92%, m.p. 135–137°C; IR (KBr) ν_{max} 1623 and 1718 (C=O), 1600 (C=C) cm^{-1}. MS (*m/z*,%): 499(M, 6), 426 (M–CO_2Et, 98), 316 (M–PPh_2, 2), 262 (PPh_3, 72), 237 (M–PPh_3, 16), 183 (PPh_2, 75), 108 (PPh, 9). Anal. Calcd for $C_{30}H_{30}NO_4P$ (499.54) C, 72.13; H, 6.05; N, 2.80%, Found: C, 71.85; H, 6.12; N, 2.65%. ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 0.43 and 1.22 (6H, 2t, 2 OCH_2CH_3), 3.54 (1H, d, $^{3}J_{HP}$ = 17.8 Hz, P–C–C*H*), 3.66 and 4.18 (4H, 2 ABX_3 system, 2 OCH_2CH_3), 5.27 (1H, br s, C_4H_4N), 5.94 (1H, dd, J_1 = 5.5, J_2 = 2.7 Hz, C_4H_4N), 6.69 (1H, d, J = 1.7 Hz, C_4H_4N), 7.42–7.70 (15H, m, 3 C_6H_5), 10.25 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 13.92 and 14.04 (2 s, 2 OCH_2CH_3), 43.05 (d, $^{2}J_{CP}$ = 12.9 Hz, P–C–CH), 44.50 (d, $^{1}J_{CP}$ = 124.8 Hz, P–C), 57.74 and 60.60 (2 s, 2 OCH_2CH_3), 104.33, 105.84 and 116.55 (3C, C_4H_4N), 127.17 (d, $^{1}J_{CP}$ = 91.7 Hz, C_{ipso}), 128.45 (d, $^{3}J_{CP}$ = 12.2 Hz, C_{meta}) 131.90 (d, $^{4}J_{CP}$ = 2.5 Hz, C_{para}), 131.99 (d, $^{2}J_{CP}$ = 9.8 Hz, C_{ortho}), 134.17 (d, $^{3}J_{CP}$ = 3.6 Hz, Cα, C_4H_4N), 170.74 (d, $^{2}J_{CP}$ = 13.2 Hz, C=O), 174.50 (d, $^{3}J_{CP}$ = 8.7 Hz, C=O), ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.42 (s, Ph_3P^{+}-C).

Di-tert-butyl 2-(1H pyrrole-2-yl)-3-(triphenylphosphoranylidene) butanedioate (**3c**): Pale yellow powder, 0.53 g, yield 96%, m.p. 163–165°C; IR (KBr) ν_{max} 1628 and 1718 (C=O), 1600 (C=C) cm^{-1}. MS (*m/z*,%): 555 (M, 7), 489 (M-heterocycle, 22), 454

(M-CO_2CMe_3, 42), 293 (M-PPh_3, 1), 262 (PPh_3, 44), 183 (PPh_2, 47), 108 (PPh, 8). Anal. Calcd for $C_{34}H_{38}NO_4P$ (555.64) C, 73.50; H, 6.89; N, 2.52%, Found: C, 73.45; H, 6.77; N, 2.58%. ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H. 0.94 and 1.49 (18H, 2 s, 2 $OCMe_3$), 3.67 (1H, d, $^3J_{HP}$ = 17.8 Hz, P–C–*C*H), 5.19 (1H, br s, C_4H_4N), 5.91 (1H, dd, J_1 = 5.6, J_2 = 2.7 Hz, C_4H_4N), 6.69 (1H, d, J = 2.1 Hz, C_4H_4N), 7.41–7.71 (15H, m, 3 C_6H_5), 10.28 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 28.24 and 28.38 (2 s, 2$OCMe_3$), 43.77 (d, $^2J_{CP}$ = 13.5 Hz, P-C-*C*H), 44.26 (d, $^1J_{CP}$ = 125.8 Hz, P–CH), 77.04 and 79.68 (2 s, 2 $OCMe_3$), 104.0, 105.6 and 116.4 (3C, C_4H_4N), 127.75 (d, $^1J_{CP}$ = 91.7 Hz, C_{ipso}), 128.47 (d, $^3J_{CP}$ = 11.8 Hz, C_{meta}), 131.90 (d, $^4J_{CP}$ = 2.4 Hz, C_{para}), 132.04 (d, $^2J_{CP}$ = 10.2 Hz, C_{ortho}), 135.03 (d, $^3J_{CP}$ = 3.0 Hz, Cα, C_4H_4N), 170.48 (d, $^2J_{CP}$ = 12.6 Hz, C=O), 173.72 (d, $^3J_{CP}$ = 9.6 Hz, C=O). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 22.83 (s, Ph_3P^+-C).

General procedures (exemplified by **5a***)*

To a magnetically stirred solution of triphenylphosphine (0.26 or 1 mmol) and pyrrole (0.66 g or 1 mmol) in 10 ml of dry ethyl acetate was added, dropwise, a mixture of dimethyl acetylendicarboxylate (0.14 g or 1 mmol) in 3 ml of ethyl acetate at –5°C over 10 min. After 8 hour stirring at room temperature, the product was filtered and recrystallised from ethyl acetate.

Dimethyl 2-(1H-pyrrole-1-yl)-3-(triphenylphosphoranylidene) butanedioate (**5a**): Colourless crystals, 0.45 g, yield 95%, m.p.147–149°C; IR (KBr) ν_{max} 1750 and 1715 (C=O), 1625 (C=C) cm^{-1}. MS (*m/z,*%): 471 (M, 7), 412 (45), 405 (100), 262 (56), 183 (100), 108(46), 77(4). Anal. Calcd for $C_{28}H_{26}NO_4P$ (471.50) C, 71.33; H, 5.56; N, 2.97%, Found: C, 71.08; H, 5.38; N, 2.74%.

Major isomer: (*E*)-**5a** (%68), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H. 3.20 and 3.78 (6H, 2 s, 2O*CH*$_3$), 4.53 (1H, d, $^3J_{HP}$ = 16.0 Hz, P–C–*CH*), 6.01–6.73 (4H_{arom}, C_4H_4N), 7.43–7.70 (15H, m, 3 C_6H_5); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 44.86 (d, $^1J_{CP}$ = 136.2 Hz, P =C), 49.25 and 52.67 (2 s, 2 O*CH*$_3$), 62.05 (d, $^2J_{CP}$ = 15.6 Hz, P–C–*CH*), 107.18 and 120.64 (2C, C_4H_4N), 126.08 (d, $^1J_{CP}$ = 92.5 Hz, C_{ipso}), 128.67 (1C, C_4H_4N), 128.86 (d, $^3J_{CP}$ = 12.3 Hz, C_{meta}), 132.05 (1C, C_4H_4N), 132.23 (d, $^4J_{CP}$ = 2.5 Hz, C_{para}), 133.72 (d, $^2J_{CP}$ = 9.8 Hz, C_{ortho}), 169.67 (d, $^3J_{CP}$ = 12.6 Hz, C=O), 173.15 (d, $^2J_{CP}$ = 14.7 Hz, P-C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 24.31 (Ph_3P^+-C).

Minor isomer: (*Z*)-**5a** (%32), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H. 3.64 and 3.75 (6H, 2 s, 2O*CH*$_3$), 4.60 (1H, d, $^3J_{HP}$ = 17.7 Hz, P–C–*CH*),6.03–6.75 (4H_{arom}, C_4H_4N), 7.43–7.70 (15H, m, 3C_6H_5); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 44.97 (d, $^1J_{CP}$ = 126.2 Hz, P =C), 50.29 and 52.49 (2 s, 2 O*CH*$_3$), 61.40 (d, $^3J_{CP}$ = 15.4 Hz, P-C-*CH*), 107.33 and 120.40 (2C, C_4H_4N), 127.11 (d, $^1J_{CP}$ = 92.2 Hz, C_{ipso}), 128.52 (1C, C_4H_4N), 128.91 (d, $^2J_{CP}$ = 12.2 Hz, C_{meta}), 132.10 (1C, C_4H_4N), 132.20 (d, $^4J_{CP}$ = 2.5 Hz, C_{para}), 133.78 (d, $^2J_{CP}$ = 8.3 Hz, C_{ortho}), 170.40 (d, $^3J_{CP}$ = 18.4 Hz, C=O), 175.31(d, $^2J_{CP}$ = 13.6 Hz, P-C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 25.17 (Ph_3P^+-C).

Diethyl 2-(1H-pyrrole-1-yl)-3-(triphenylphosphoranylidene) butanedioate (**5b**): White solid, 0.46 g, yield 93%, m.p. 129–131°C; IR (KBr) ν_{max} 1756 and 1725 (C=O), 1620 (C=C) cm^{-1}. MS (*m/z,*%): 499 (M, 3), 454 (18), 426 (87), 437 (63), 262 (37), 183 (66) 108(17). Anal. Calcd for $C_{30}H_{30}NO_4P$ (499.54) C, 72.13; H, 6.05; N, 2.80%, Found: C, 71.39; H, 5.93; N, 2.93%.

Major isomer: (*E*)-**5b** (%71), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 0.52 and 1.31 (6H, 2t, $^3J_{HH}$ = 6.0 Hz, 2 OCH_2CH_3), 3.79 and 4.19 (4H, 2 ABX_3 system, 2 O*CH*$_2$CH$_3$), 4.51 (1H, d, $^3J_{HP}$ = 16.4 Hz, P–C–*CH*), 5.98–6.72 (4H_{arom}, C_4H_4N), 7.41–7.70 (15H, m, 3C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.09 and 14.34 (2 s, 2 OCH_2CH_3), 44.09 (d, $^1J_{CP}$ = 127.5 Hz, P =C), 57.80 and 58.50 (2 s, 2 O*CH*$_2$CH$_3$), 61.31 (d, $^2J_{CP}$ = 15.8 Hz, P–C–*CH*), 107.06 and 120.68 (2C, C_4H_4N), 126.35 (d, $^1J_{CP}$ = 92.2 Hz, C_{ipso}) 128.33 (1C, C_4H_4N), 128.76 (d, $^3J_{CP}$ = 11.4 Hz, C_{meta}), 132.18 (d, $^4J_{CP}$ = 2.2 Hz, C_{para}), 133.78 (d, $^2J_{CP}$ = 9.8 Hz, C_{ortho}), 133.93 (1C, C_4H_4N), 169.17 (d, $^3J_{CP}$ = 12.7 Hz, C=O), 172.44 (d, $^2J_{CP}$ = 13.7 Hz, P–C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.43 (s, Ph_3P^+-C).

Minor isomer: (*Z*)-**5b** (%29), ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 1.25 and 1.38 (6H, 2t, $^3J_{HH}$ = 7.0 Hz, 2OCH_2CH_3), 3.78 and 4.27 (4H, 2 ABX_3 system, 2 O*CH*$_2$CH$_3$), 4.56 (1H, d, $^3J_{HP}$ = 18.2 Hz, P–C–*CH*), 5.99- 6.75 (4H_{arom}, C_4H_4N), 7.41–7.70 (15H, m, 3C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.33 and 15.01 (2 s, 2 OCH_2CH_3), 44.64 (d, $^1J_{CP}$ = 135.3 Hz, P =C), 57.83 and 58.52 (2 s, 2 O*CH*$_2$CH$_3$), 62.01 (d, $^2J_{CP}$ = 15.1 Hz, P–C–*CH*), 107.18 and 120.48 (2C, C_4H_4N), 127.33 (d, $^1J_{CP}$ = 92.0 Hz, C_{ipso}),128.24 (1C, C_4H_4N), 128.86 (d, $^3J_{CP}$ = 10.2 Hz, C_{meta}), 132.20 (d, $^4J_{CP}$ = 2.2 Hz, C_{para}), 133.80 (d, $^2J_{CP}$ = 10.0 Hz, C_{ortho}), 134.28 (1C, C_4H_4N), 170.10 (d, $^2J_{CP}$ = 17.5 Hz, C=O), 170.88 (d, $^3J_{CP}$ = 13.5 Hz, P–C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.87 (s, Ph_3P^+–C).

Di-tert-butyl 2-(1H-pyrrole-1-yl)-3-(triphenylphosphoranylidene) butanedioate (**5c**): White powder Yield 0.53 g yield 96%, m.p. 161–163°C; IR (KBr) ν_{max} 1737 and 1725 (C=O), 1620 (C=C) cm^{-1}. MS (*m/z,*%): 555 (M, 5), 489 (100), 454 (62), 262 (45), 183 (72), 108 (21), 77(19), 66 (49). Anal. Calcd for $C_{34}H_{38}NO_4P$ (555.64) C, 73.50; H, 6.89; N, 2.52%, Found: C, 73.66; H, 6.61; N, 2.36%.

Major isomer: (*E*)-**5c**, ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 1.03 and 1.59 (18 H, 2 s, 2C*CH*$_3$), 4.36 (1H, d, $^3J_{HP}$ = 19.8 Hz, P–C–*CH*), 5.96–6.80 (4H_{arom}, C_4H_4N), 7.49–7.71 (15H, m, 3C_6H_5). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 28.28 and 28.46 (2C*Me*$_3$), 43.62 (d, $^1J_{CP}$ = 128.3 Hz, P =C), 62.49 (d, $^2J_{CP}$ = 16.0 Hz, P–C–*CH*), 77.33 and 80.56 (2 s, 2 $OCMe_3$), 106.91 and 120.56 (2C, C_4H_4N), 127.54 (d, $^1J_{CP}$ = 91.8 Hz, C_{ipso}), 128.41 (1C, C_4H_4N), 128.62 (d, $^3J_{CP}$ = 11.7 Hz, C_{meta}), 134.00 (1C, C_4H_4N), 132.02 (d, $^4J_{CP}$ = 2.0 Hz, C_{para}), 133.84 (d, $^2J_{CP}$ = 9.3 Hz, C_{ortho}), 168.65 (d, $^2J_{CP}$ = 12.3 Hz, C=O), 171.33 (d, $^3J_{CP}$ = 13.6 Hz, P-C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.79 (Ph_3P^+-C).

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