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RESEARCH ARTICLE

Chemoselective synthesis of phosphorus ylides through the reaction of 2-mercaptobenzimidazole and 2-hydroxybenzimidazole with triphenylphosphine and acetylenic esters

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A one-step synthesis of dialkyl 2-(2-mercaptobenzimidazole-s-yl)-3-(triphenylphosphoranyliden) succinates and dialkyl 2-(2-hydroxybenzimidazole-n-yl)-3-(triphenylphosphoranyliden) succinates in fair yields are reported through the reaction of dialkyl acetylenedicarboxylates and triphenylphosphine in the presence of 2-mercaptobenzimidazole or 2-hydroxybenzimidazole.

Keywords: Chemoselective; Acetylenic esters; 2-Mercaptobenzimidazole; Triphenylphosphine; Stable phosphorus ylide; Geometrical isomers

1. Introduction

The synthesis of phosphorus ylides is important in organic chemistry because of the application of these compounds in the synthesis of organic products [1–14] especially the synthesis of naturally occurring products with biological and pharmacological activity [15]. Phosphorus ylides are usually prepared by deprotonation of phosphonium salts, which in turn, can be prepared most often by the reaction of triphenylphosphine and an alkyl halide [16]. In recent years, a three-component method has been developed [17–20] for the synthesis of organophosphorus compounds using a novel approach employing vinylphosphonium salts. This method is successful for the preparation of 1,4-diionic organophosphorus compounds [21, 22]. We wish to describe an efficient synthetic route of such derivatives from 2-mercaptobenzimidazole and 2-hydroxybenzimidazole stable phosphorus ylides. The benzimidazole moiety and its derivatives have the important pharmaceutical property and they have been used for medicinal

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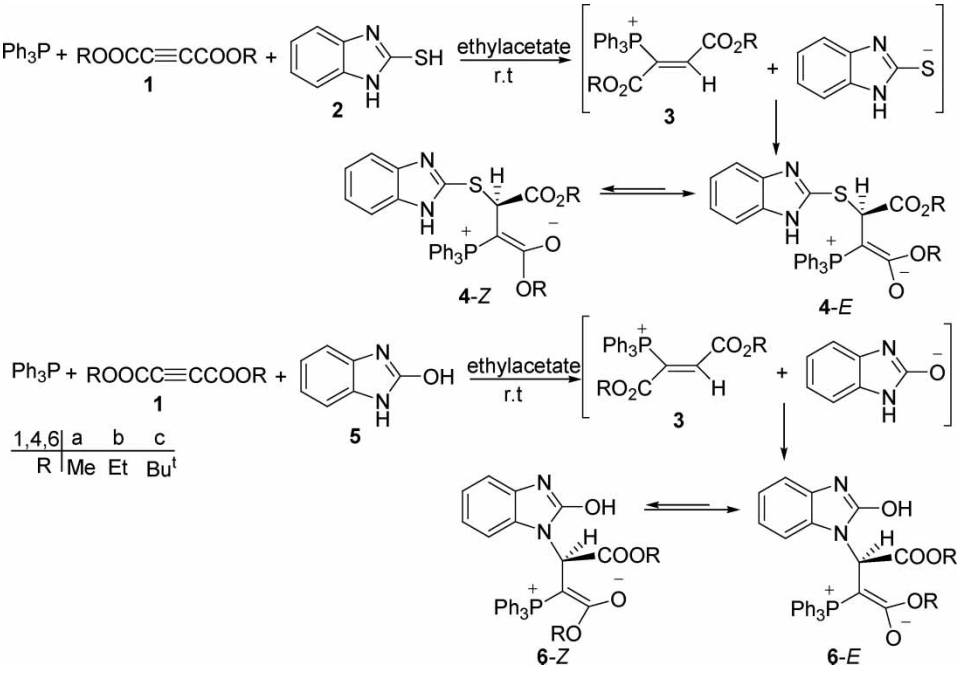
chemistry purposes [23]. Herein we outline the reaction of triphenylphosphine with dialkyl acetylenedicarboxylates (**1**) in the presence of (**2**) or (**5**) offering vinyl triphenylphosphonium salt (**3**), which in turn gives a series of phosphoranes after chemoselective attack of sulfur anion of the 2-mercaptobenzimidazole and nitrogen atom of the 2-hydroxybenzimidazole anion.

2. Results and discussion

The reactions of 2-mercaptobenzimidazole or 2-hydroxybenzimidazole with dialkyl acetylenedicarboxylates (**1**) in the presence of triphenylphosphine were proceeded in ethyl acetate solvent at room temperature and finished after approximately 3 hrs. The ^{1}H and ^{13}C NMR spectrum of the crude product clearly indicated the formation of phosphoranes **4** and **6** (scheme 1). Any product other than **4** and **6** could not be detected by NMR spectroscopy. The ^{1}H NMR and ^{13}C NMR spectra of compounds **4a** and **4b** exhibited two doublets at δ 6.05 ($J = 17.4$) and δ 6.11 ($J = 17.9$) for the S−C*H*−C−P and also a remarkable signal at δ 11.18 and δ 11.28 respectively could be observed for the N−H group in them. Furthermore in their IR spectra, a signal for S−H group was not observable. This evidence is indicative that 2-mercapto-benzimidazole has different behaviour with respect to 2-hydroxybenzimidazole. In addition, products **4a** and **4b** displayed ^{13}C NMR resonances at δ 165.40 ppm and δ 165.03 ppm, respectively for the N=*C*−S unit [13, 14, 24]. The IR and ^{1}H NMR spectra of compounds **6a–c** showed a signal at $\upsilon = 3200\,\mathrm{cm}^{-1}$ and δ 9.87 for the O−H group, respectively. This data confirms that it is the nitrogen anion of 2-hydroxybenzimidazole that has attacked the vinyl triphenylphosphonium cation. The structures of compounds **4a,b** and **6a–c** were deduced from their IR, ^{1}H, ^{13}C, and ^{31}P NMR spectra. The mass spectrum of them displayed molecular ion peaks at appropriate m/z values. Any fragmentations involve loss of the side chains. The ^{1}H, ^{13}C, and ^{31}P NMR spectra of ylides **4a,b** and **6a,b** are consistent with



SCHEME 1

the presence of two isomers but only one geometrical isomer was observed for the di-tert-butyl derivative of **6a**, presumably, because of the bulky tert-butyl group. The ylide moiety of these compounds is strongly conjugated with the adjacent carbonyl group and rotation around the partial double bond in **4**-*E*, **4**-*Z*, **6**-*E*, and **6**-*Z* is slow on the NMR timescale at ambient temperature. ^{31}P NMR chemical shifts and coupling constants in the major (M) and minor (m) geometrical isomers of compounds **4a,b** and **6a–c** are reported in the Experimental section.

In conclusion, we have prepared novel stable phosphorus ylides using a one-pot reaction between triphenylphosphine and acetylenic compounds in the presence of such related heterocycles as 2-mercaptobenzimidazole and 2-hydroxybenzimidazole. The present method carries the advantage that not only is the reaction performed under neutral conditions, but also the substances can be mixed without any activation or modification.

3. 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 spectrum were obtained from a BRUKER DRX-500 AVANCE instrument with $CDCl_3$ 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 analyzer. In addition, the mass spectrum were recorded on a Shimadzu QP 1100 EX mass spectrometer operating at an ionization potential of 70 eV. Triphenylphosphine, dialkyl acetylenedicarboxylates (**1a–c**), 2-mercaptobenzimidazole (**2**) and 2-hydroxybenzimidazole (**5**) were purchased from Fluka (Buchs, Switzerland) and used without further purification.

3.1 *Preparation of dimethyl-2-(2-mercaptobenzimidazole-s-yl)-3-(triphenylphosphanylidene)succinate (4a)*

3.1.1 General procedure. To a stirred solution of 2-mercaptobenzimidazole (0.15 g, 1 mmol) and triphenylphosphine (0.26 g, 1 mmol) in 8 mL of ethyl acetate was added, dropwise, a mixture of dimethyl acetylenedicarboxylate (0.14 g, 1 mmol) in 4 mL of ethyl acetate at −5 °C over 10 min. After approximately 3 hrs stirring at room temperature, the reaction mixture was filtered and solid phase was separated from liquid phase. The solid phase was then washed with cold diethyl ether (3 × 5 mL, three times) in order to obtain the product as white powder. mp 196–198 °C, 0.53 g, yield 95%, IR (υ_{max}, cm^{-1}) 1750 and 1608 (C=O). MS (*m*/*z*, %): 493 (M−2OMe, 9), 406 (M−$C_7H_5N_2$ and OMe, 16), 405 (M−$C_7H_5N_2S$, 18), 262 (PPh_3, 72), 183 (PPh_2, 76), 108 (PPh, 40). Anal. calcd. for $C_{31}H_{27}N_2O_4PS$ (554): C, 66.87; H, 4.91; N, 4.92%. Found: C, 67.15; H, 4.87; N, 5.05%.

Major isomer (*E*)-**4a** (69%): ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 3.21 and 3.79 (6H, 2s, 2OCH_3), 6.05 (1H, d, $^{3}J_{PH}$ = 17.4 Hz, P−C−C*H*), 7.33–7.71 (19H_{arom}, m, 3C_6H_5 and $C_7H_4N_2$), 11.18 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 40.82 (d, $^{1}J_{PC}$ = 123.1 Hz, P=C), 49.54 and 52.34 (2s, 2OCH_3), 60.48 (d, $^{2}J_{PC}$ = 18.2 Hz, P−C−C*H*), 109.37, 109.61, 110.10, 112.96, 113.54 and 122.53 (6C, $C_7H_5N_2$), 126.09 (d, $^{1}J_{PC}$ = 91.0 Hz, C_{ipso}), 128.53 (d, $^{3}J_{PC}$ = 12.2 Hz, C_{meta}), 132.25 (C_{para}), 133.48 (d, $^{2}J_{PC}$ = 11.3 Hz, C_{ortho}), 165.40 (1C, N=C−S), 169.91 (d, $^{3}J_{PC}$ = 12.4 Hz, C=O), 171.05 (d, $^{2}J_{PC}$ = 14.2 Hz, P−C=*C*). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P23.8 (Ph_3P^+−C).

Minor isomer (*Z*)-**4a** (31%): ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 3.71 and 3.79 (6H, 2s, 2OCH_3), 5.92 (1H, d, $^{3}J_{PH}$ = 19.4 Hz, P−C−C*H*), 7.33–7.71 (19H_{arom}, m, 3C_6H_5 and

$C_7H_4N_2$), 11.29 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 41.74 (d, $^1J_{PC} = 129.4$ Hz, P=C), 50.66 and 52.54 (2s, 2OCH_3), 60.83 (d, $^2J_{PC} = 18.8$ Hz, P−C−CH), 109.39, 109.64, 110.14, 112.98, 113.60 and 122.78 (6C, $C_7H_5N_2$), 125.37 (d, $^1J_{PC} = 91.4$ Hz, C_{ipso}), 128.94 (d, $^3J_{PC} = 12.2$ Hz, C_{meta}), 132.23 (C_{para}), 133.56 (d, $^2J_{PC} = 10.0$ Hz, C_{ortho}), 165.03 (1C, N=C−S), 168.41 (d, $^3J_{PC} = 13.3$ Hz, C=O), 170.60 (d, $^2J_{PC} = 13.8$ Hz, P−C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 24.8 (Ph_3P^+−C).

3.2 *Diethyl-2-(2-mercaptobenzimidazole-s-yl)-3-(triphenylphosphanylidene) succinate (4b)*

White powder, mp 179–181 °C, 0.54 g, yield 93%, IR (υ_{max}, cm^{-1}) 1738 and 1603 (C=O). MS (m/z, %): 433 (M−$C_7H_5N_2S$, 22), 326 (M−PPh_2 and CO_2Et, 10), 320 (M−PPh_3, 9), 275 (M−PPh_3 and OEt, 12), 262 (PPh_3, 78), 247 (M−PPh_3 and CO_2Et, 38), 183 (PPh_2, 79), 108 (PPh, 33). Anal. calcd. for $C_{33}H_{31}N_2O_4PS$ (582): C, 68.22; H, 5.45; N, 4.71%. Found: C, 68.04; H, 5.33; N, 4.81%.

Major isomer (E)-**4b** (69%): ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 0.49 and 1.29 (6H, 2t, $^3J_{HH} = 7.2$ Hz, 2OCH$_2$CH_3), 3.80 and 4.25 (4H, 2m, 2ABX$_3$ system, 2OCH_2CH$_3$), 6.11 (1H, d, $^3J_{PH} = 17.9$ Hz, P−C−CH), 7.37–8.05 (19H$_{arom}$, m, 3C_6H_5 and $C_7H_4N_2$), 11.28 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.06 and 14.31 (2s, 2OCH$_2$CH_3), 40.54 (d, $^1J_{PC} = 123.2$ Hz, P=C), 58.24 and 60.57 (2S, 2OCH$_2$CH$_3$), 61.38 (d, $^2J_{PC} = 15.8$ Hz, P−C−CH), 109.45, 109.70, 110.16, 113.09, 113.60 and 122.42 (6C, $C_7H_5N_2$), 125.63 (d, $^1J_{PC} = 91.9$ Hz, C_{ipso}), 128.86 (d, $^3J_{pc} = 12.2$ Hz, C_{meta}), 132.23 (C_{para}), 133.54 (d, $^2J_{pc} = 9.4$ Hz, C_{ortho}), 165.40 (1C, N=C−S), 169.53 (d, $^3J_{PC} = 12.6$ Hz, C=O), 170.38 (d, $^2J_{PC} = 14.2$ Hz, P−C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 23.8 (Ph_3P^+−C).

Minor isomer (Z)-**4b** (31%): ^{1}H NMR (500.1 MHz, $CDCl_3$): δ_H 1.22 and 1.34 (6H, 2t, $^3J_{HH} = 7.1$ Hz 2OCH$_2$CH_3), 4.13 and 4.32 (4H, 2m, 2ABX$_3$ system, 2OCH_2CH$_3$), 5.87(1H, d, $^3J_{PH} = 20.0$ Hz, P−C−CH), 7.37–8.05 (19H$_{arom}$, m, 3C_6H_5 and $C_7H_4N_2$), 11.36 (1H, s, NH). ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.13 and 14.25 (2s, 2OCH$_2$CH_3), 40.55 (d, $^1J_{PC} = 135.7$ Hz, P=C), 58.24 and 60.42 (2s, 2OCH$_2$CH$_3$), 60.87 (d, $^2J_{PC} = 16.2$ Hz, P−C−CH), 109.48, 109.72, 110.19, 113.12, 113.63 and 122.34 (6C, $C_7H_4N_2$), 126.29 (d, $^1J_{PC} = 91.5$ Hz, C_{ipso}), 128.90 (d, $^3J_{pc} = 12.1$ Hz, C_{meta}), 132.26 (C_{para}), 133.60 (d, $^2J_{pc} = 9.4$ Hz, C_{ortho}), 165.03 (1C, N=C−S), 170.32 (d, $^3J_{PC} = 13.7$ Hz, C=O), 170.48 (d, $^2J_{PC} = 14.1$ Hz, P−C=C). ^{31}P NMR (202.4 MHz, $CDCl_3$): δ_P 25.1 (Ph_3P^+−C).

3.3 *Dimethyl-2-(2-hydroxybenzimidazole-n-yl)-3-(triphenylphosphanylidene) succinate (6a)*

Colorless crystals, mp 178–180 °C, 0.52 g, yield 96%; IR (υ_{max}, cm^{-1}): 1724 and 1630 (C=O); MS, (m/z, %): 538 (M^+, 1), 405 (M-heterocycle), 420 (M-2CO_2Me, 1), 183 (PPh_2, 100), 276 (M−PPh_3, 23), 262 (PPh_3, 91), 108 (PPh, 51).

Major rotamer (E)-**6a** (63%): ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 3.16 and 3.79 (6H, 2s, 2OCH$_3$), 5.32 (1H, d, $^3J_{PH} = 16.4$ Hz, P−C−CH), 6.96–7.78 (19H$_{arom}$, m, 3C_6H_5 and $C_7H_4N_2$), 9.86 (1H, s, OH); ^{13}C NMR (125.8 MHz, $CDCl_3$), δ_C 40.47 (d, $^1J_{PC} = 124.3$ Hz, P=C), 49.32 and 52.63 (2OCH$_3$), 55.57 (d, $^2J_{PC} = 16.1$ Hz, P−C−CH), 126.27 (d, $^1J_{PC} = 91.6$ Hz, C_{ipso}), 108.92, 108.96, 112.66, 120.93, 121.21 and 125.19 (6C, $C_7H_4N_2$), 128.78 (d, $^3J_{PC} = 12.3$ Hz, C_{meta}), 132.16 (C_{para}), 133.50 (d, $^2J_{PC} = 9.9$ Hz, C_{ortho}), 155.03 (1C, $C_7H_4N_2$), 169.62 (d, $^3J_{PC} = 12.6$ Hz, C=O), 171.71 (d, $^2J_{PC} = 15.2$ Hz, P−C=C); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 23.4 (Ph_3P^+−C).

Minor rotamer (*Z*)-**6a** (37%): ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 3.70 and 3.77 (6H, 2s, 2OC*H*$_3$), 5.24 (1H, d, $^{3}J_{PH}$ = 18.6 Hz, P−C−C*H*), 6.96–7.78 (19*H*$_{arom}$, m, $3C_6H_5$ and $C_7H_4N_2$), 9.90 (1H, s, OH); ^{13}C NMR (125.76 MHz, $CDCl_3$), δ_C 40.85 (d, $^{1}J_{PC}$ = 132.7 Hz, P=C), 50.38 and 52.39 (2O*C*H$_3$), 56.12 (d, $^{2}J_{PC}$ = 16.7 Hz, P−C−C*H*), 126.41 (d, $^{1}J_{PC}$ = 92.0 Hz, C_{ipso}), 108.89, 108.93, 111.80, 120.88, 121.26 and 125.23 (6C, $C_7H_4N_2$), 128.84 (d, $^{3}J_{PC}$ = 12.3 Hz, C_{meta}), 132.18 (C_{para}), 133.58 (d, $^{2}J_{PC}$ = 10.2 Hz, C_{ortho}), 155.24 (1C, $C_7H_4N_2$), 170.23 (d, $^{3}J_{PC}$ = 15.8 Hz, C=O), 171.91 (d, $^{2}J_{PC}$ = 15.2 Hz, P−C=*C*); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 24.1 (Ph_3P^+−C).

3.4 *Diethyl-2-(2-hydroxybenzimidazole-n-yl)-3-(triphenylphosphanylidene) succinate (6b)*

Colorless crystals, mp 141–143 °C, 0.52 g, yield 92%; IR (υ_{max}, cm^{-1}): 1733 and 1620 (C=O). MS, (*m*/*z*, %): 476 (M−2OEt, 61), 433 (M−C_7H_5ON, 24), 304 (M−PPh_3, 7), 262 (PPh_3, 74), 183 (PPh_2, 76), 108 (PPh, 35).

Major rotamer (*E*)-**6b** (71%): ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 0.47 and 1.29 (6H, 2t, $^{3}J_{HH}$ = 6.8 Hz 2OCH_2C*H*$_3$), 3.76 and 4.21 (4H, m, 2ABX$_3$ systhem 2O*C*H$_2$CH$_3$), 5.29 (1H, d, $^{3}J_{PH}$ = 17.0 Hz, P−C−C*H*), 6.96–7.83 (19H$_{arom}$, m, $3C_6H_5$ and $C_7H_4N_2$), 9.82 (1H, s, OH); ^{13}C NMR (125.8 MHz, $CDCl_3$), δ_C 13.21 and 13.64 (2s, 2O−C−*C*H$_3$), 40.86 (d, $^{1}J_{PC}$ = 123.6 Hz, P=C), 59.13 and 60.57 (2S, 2O*C*H$_2$CH$_3$), 61.54 (d, $^{2}J_{PC}$ = 14.3 Hz, P−C−*C*H), 108.83, 108.98, 112.32, 121.13, 121.39 and 124.81 (6C, $C_7H_4N_2$), 126.31 (d, $^{1}J_{PC}$ = 91.8 Hz, C_{ipso}), 128.57 (d, $^{3}J_{PC}$ = 11.6 Hz, C_{meta}), 132.19 (C_{para}), 133.59 (d, $^{2}J_{PC}$ = 9.8 Hz, C_{ortho}), 156.12 (1C, $C_7H_4N_2$), 169.93 (d, $^{3}J_{PC}$ = 13.6 Hz, C=O), 170.29 (d, $^{2}J_{PC}$ = 12.3 Hz, P−C=*C*); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 23.4 (Ph_3P^+−C).

Minor rotamer (*Z*)-**6b** (29%): ^{1}H NMR (500.1 MHz, $CDCl_3$), δ_H 1.21 and 1.33 (6H, 2t, $^{3}J_{HH}$ = 7.1 Hz, 2OCH_2C*H*$_3$), 4.15 and 4.28 (4H, m, 2ABX$_3$systhem 2O*C*H$_2$CH$_3$), 5.19 (1H, d, $^{3}J_{PH}$ = 19.3 Hz, P−C−C*H*), 6.96–7.83 (19H$_{arom}$, m, $3C_6H_5$ and $C_7H_4N_2$), 9.85 (1H, s, OH); ^{13}C NMR (125.8 MHz, $CDCl_3$), δ_C 13.76 and 13.84 (2s, 2O−C−*C*H$_3$), 41.09 (d, $^{1}J_{PC}$ = 134.5 Hz, P=C), 59.25 and 60.61 (2s, 2O*C*H$_2$CH$_3$), 61.98 (d, $^{2}J_{PC}$ = 15.8 Hz, P−C−*C*H), 108.54, 109.13, 112.24, 120.16, 121.76 and 125.69 (6C, $C_7H_4N_2$), 126.46 (d, $^{1}J_{PC}$ = 92.1 Hz, C_{ipso}), 128.61 (d, $^{3}J_{PC}$ = 11.2 Hz, C_{meta}), 132.16 (C_{para}), 133.63 (d, $^{2}J_{PC}$ = 9.8 Hz, C_{ortho}), 156.39 (1C, $C_7H_4N_2$), 168.14 (d, $^{3}J_{PC}$ = 12.3 Hz, C=O), 171.82 (d, $^{2}J_{PC}$ = 14.3 Hz, P−C=C), ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 24.3 (Ph_3P^+−C).

3.5 *Di-tert-buthyl-2-(2-hydroxybenzimidazole-n-yl)-3-(triphenylphosphanylidene) succinate (6c)*

Colorless crystals, mp 151–153 °C, 0.59 g, yield 95%; IR (υ_{max}, cm^{-1}): 1720 and 1618 (C=O).

Major rotamer: 1H NMR (500.1 MHz, $CDCl_3$), δ_H 0.99 and 1.57 (18H, 2s, 2OC*Me*$_3$), 5.09 (1H, d, $^{3}J_{PH}$ = 18.1 Hz, P−C−C*H*), 6.92–7.96 (19H$_{arom}$, m, $3C_6H_5$ and $C_7H_4N_2$), 9.92 (1H, s, OH); ^{13}C NMR (125.8 MHz, $CDCl_3$), δ_C 28.25 and 28.42 (2OC*Me*$_3$), 41.33 (d, $^{1}J_{PC}$ = 132.8 Hz, P=C), 59.32 (d, $^{2}J_{PC}$ = 18.2 Hz, P−C−C*H*), 79.28 and 81.68 (2s, 2O*C*Me$_3$), 108.67, 109.24, 113.11, 120.51, 122.08 and 125.86 (6C, $C_7H_4N_2$), 127.01 (d, $^{1}J_{PC}$ = 92.2 Hz, C_{ipso}), 128.51 (d, $^{3}J_{PC}$ = 12.3 Hz, C_{meta}), 132.08 (C_{para}), 133.67 (d, $^{2}J_{PC}$ = 9.7 Hz, C_{ortho}), 156.72 (1C, $C_7H_4N_2$), 169.11(d, $^{3}J_{PC}$ = 13.5 Hz, C=O), 170.29 (d, $^{2}J_{PC}$ = 12.3 Hz, P−C=C); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 23.2 (Ph_3P^+−C).

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