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Synthesis of Precursors of 4-Phosphino-1*H*-pyrrole-3-carbaldehyde Derivatives

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ABSTRACT: *In this work, possible approaches to the synthesis of 1,2,5-substituted 4-phosphoryl-3-formylpyrroles have been considered. As a result, two methods for the synthesis of 4-(diphenylphosphoryl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1*H*-pyrrole-3-carbaldehyde were proposed; the highest yields gives formylation of 3-(diphenylphosphorothioyl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1*H*-pyrrole. The formyl fragment was successfully converted into a Schiff base with phenethylamine, and the phosphoryl group has been reduced to phosphine with silicochloroform, which suggests a promising approach to the synthesis of chiral bidentate phosphine ligands.* © 2013 Wiley Periodicals, Inc. Heteroatom Chem 24:146–151, 2013; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21069

INTRODUCTION

Ligands containing a phosphine donor and a carbonyl fragment at the adjacent atoms show exceptional applicability in metal complex catalysis; an example of this type of ligands is represented by the well-known Trost ligand [1, 2].

This approach for the synthesis of new chiral phosphine ligands is straightforward and convenient. A chiral inductor is a derivative of carboxylic acids—amides, amidines, or derivatives of ketones (aldehydes)—various azomethines. Scaffolds on which these groups are usually placed are the phenyl ring, but there are examples of heterocyclic compounds and metallocenes. These ligands, which can be depicted by a general formula **I**, work with various metals, for example, palladium [1–6], rhodium [7, 8], copper [9], and iridium [10, 11] (see Fig. 1). The literature analysis shows that these ligands are poorly represented by the pyrrole derivatives, whereas the well-known property of pyrroles is easy direct—phosphorylation by phosphorus halides and good reactivity in other reactions of electrophilic substitution [12, 13]. This work presents an attempt to reach the precursors of the mentioned type of ligands by formylation of substituted phosphorylated pyrroles.

RESULTS AND DISCUSSION

As the pyrrole ring represents electron-rich heterocycles for which double electrophilic reactions are common, our strategy anticipated consecutive introduction of phosphino and carbonyl groups at 2,5-dimethyl-*N*-aryl pyrroles by electrophilic substitution reactions. Phosphine **2** was easily prepared by phosphorylation of 2,5-dimethyl-*N*-arylpyrrole **1** (Scheme 1) [12]. Phosphine **2** is a colorless crystalline solid capable of prolonged storage in an inert atmosphere. The straightforward method to the target compounds,

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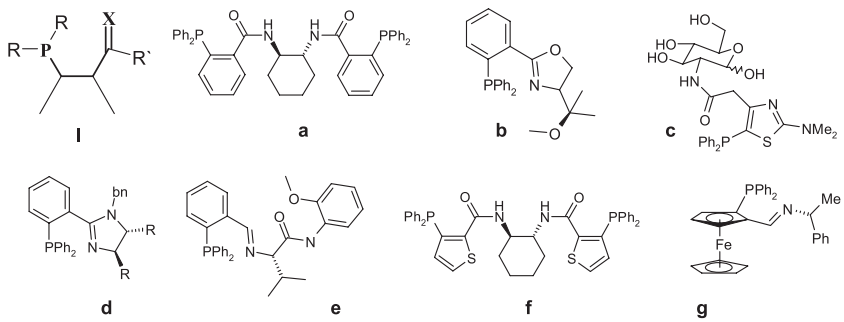
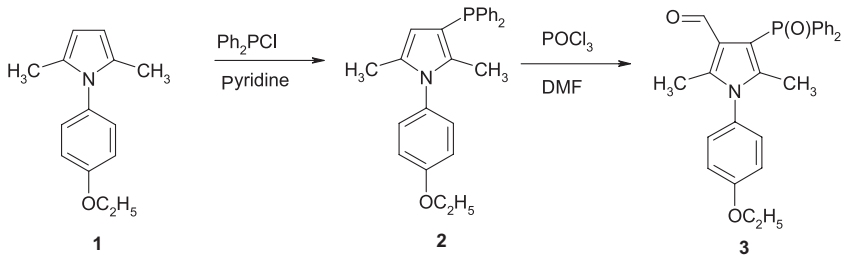


FIGURE 1 Examples of phosphines of type **I**: **a** [1,2], **b** [11], **c** [3], **d** [10], **e** [8], **f** [5], and **g** [6].



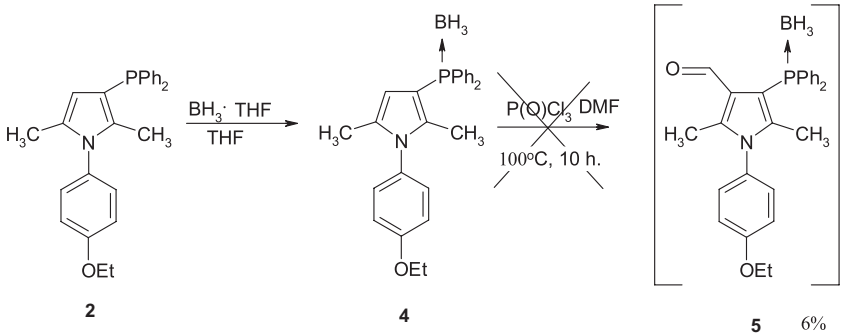
SCHEME 1

3-formyl-4-diphenylphosphinopyrroles, seems to be direct formylation of phosphine **2**. Although phosphine **2** contains two nucleophilic centers—3-position of the pyrrole ring and phosphorus atom, we expected that by manipulating the reaction conditions we would be able to get direct formylation exclusively at the pyrrole ring.

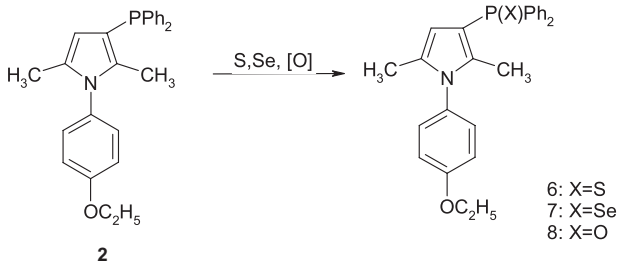
We have found that Vilsmeier formylation of phosphine **2** led to the formation of complex mixtures, in which judging by ^{31}P NMR complete oxidation of trivalent phosphorus took place (Scheme 1). Therefore, this approach produced compound **3** only in 35% yield. It should be mentioned that deoxygenation of solvents and saturation with argon had no effect on the yield of compound **3**. This may prove that the oxidation of trivalent phosphorus occurs by formylation reagents.

To protect the phosphino group, compound **2** was converted into phosphine-borane **4** a colorless solid by a standard procedure (Scheme 2) [14]. Unfortunately, formylation under Vilsmeier conditions failed to produce the target aldehyde. Thus, at 100°C in 8–10 h conversion of phosphine-borane **4** was only 50%, accompanied by marked resinification of the reaction mixture. Varying the reaction conditions had a little effect so that this approach did not work and we failed to prepare **5**.

To avoid the formation of large amounts of byproducts, we decided to use pentavalent phosphorus derivatives **6–8** in formylation with further reduction to trivalent phosphorus derivatives. For this purpose, phosphine **2** was converted into sulfide **6**, selenide **7**, and oxide **8** (Scheme 3). It was found that formylation of pyrroles **6–8** proceeded under



SCHEME 2



2

SCHEME 3

rather harsh conditions—100°C, a double excess of phosphorus oxychloride and 10 h, which is probably due to electron withdrawing and steric effects of the phosphoryl groups. In all three cases, formylation led to the sole aldehyde **3** (Scheme 4). It is obvious that the thio(seleno)phosphoryl groups are not tolerant to the Vilsmeier reagent. It is likely that the electrophilic Vilsmeier reagent attacks sulfur (selenium), affording an intermediate that upon hydrolysis gave phosphoryl derivatives. Similar transformation of thiophosphoryl and selenophosphoryl groups upon treatment with trifluoroacetic acids anhydride was described in the literature [15]. As in the case of the first approach (Scheme 1), this approach also does not allow completely avoiding appreciable resinification of the reaction mixture, although the products yields considerably increased. Thus, the lowest yield was observed for oxide **8** (51%), whereas the sulfide **6** gave the highest yield (75%), which makes it the most appropriate starting material in this reaction. Since in the present study the target compounds are the phosphines with a chiral center at the adjacent atom, our further research was focused on the synthesis of the corresponding derivatives of the formyl group, as well as the reduction of the phosphineoxide fragment to the trivalent phosphine.

We have obtained the Schiff base using the aldehyde **3** and racemic 1-phenylethanamine. We have attempted the synthesis of azomethine **9** by boiling the reaction mixture in methanol and in toluene with removal of water. The former procedure gave an 80% isolated yield of azomethine **9** when aldehyde **3** was boiling in methanol with a sesquialteral excess of the amine. For the reduction of phosphineoxide **9**, two reagents, diphenylsilane and trichlorosilane, were tested. Diphenylsilane was not very useful for this reaction; it began to work only upon prolonged (16 h) heating at 160°C, which leads to a considerable resinification mixtures, purification of which additionally complicated by the presence of phenylsiloxanes. The reaction with trichlorosilane proceeds readily in boiling toluene and completes the reaction in 6 h, affording a target product **10** in 80% isolated yield.

CONCLUSIONS

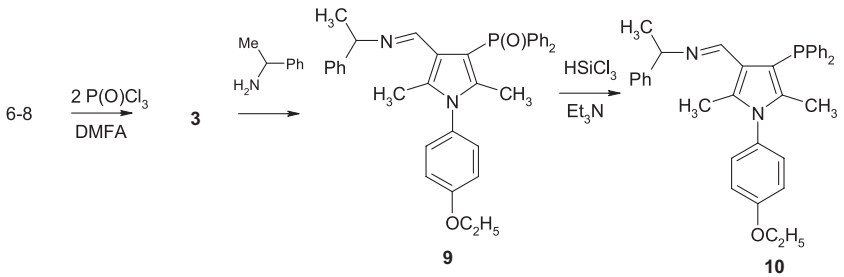
As a result of the current study, the approaches to the synthesis of 4-(diphenylphosphoryl)-1-aryl-2,5-dimethyl-1*H*-pyrrole-3-carbaldehydes were found. A consecutive transformation of the formyl moiety into an azomethine one and the reduction of phosphineoxide to the phosphine center with satisfactory yields have been shown. The obtained results look promising for further synthesis of chiral ligands based on the pyrrole ring.

EXPERIMENTAL

NMR spectra were recorded on a Varian VXR-300 (Paula Alta, USA) spectrometer or Bruker Avance drx 500: ^{1}H (300/500 MHz) and ^{13}C (125 MHz) with TMS as an internal standard and ^{31}P (121 MHz) with 85% H_3PO_4 as an external standard. APT experiments were used to assign ^{13}C chemical shifts. Mass spectra were recorded using an Agilent 1100 series LC/MSD system. All reactions were carried out under dry argon in Schlenk-type glassware. Solvents including deuterated ones used for NMR spectroscopy were dried and distilled prior to use.

*3-(Diphenylphosphino)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole **2***

To a stirred solution of pyrrole **1**, 2.15 g (0.01 mol) in pyridine (60 mL) and diphenylphosphinous



9 **10**

SCHEME 4

chloride 2.21 g (1.87 mL, 0.01 mol) was added. The reaction mixture was stirred for 5 days at room temperature. Hexane (100 mL) was added to the reaction mixture. After 30 min, the precipitated pyridine hydrochloride was filtered off and the filtrate was evaporated in vacuo. The product was recrystallized from acetonitrile as colorless solid. mp 146–147°C, yield 82%. ^{1}H NMR ($CDCl_3$), δ (ppm): 1.45 (t, 3H, $J = 7.2$ Hz, OCH_2CH_3), 1.95 (s, 3H, CH_3), 2.14 (s, 3H, CH_3), 4.08 (q, 2H, $J = 7.2$ Hz, OCH_2CH_3), 5.65 (s, 1H, CH, Pyr), 6.95 (d, 2H, $J = 8.8$ Hz, Ar), 7.11 (d, 2H, $J = 8.8$ Hz, Ar), 7.25–7.40 (m, 6H, Ar), 7.39–44 (m, 4H, Ar). ^{13}C{^{1}H}NMR ($CDCl_3$), δ (ppm): 12.16; 12.66; 14.76; 63.76; 108.72, 107.69 (d, $J_{CP} = 129.5$ Hz); 109.48; 114.87; 127,80; 128.15 (d, $J_{CP} = 6.3$ Hz); 129.20; 129.27; 130.18; 131.83 (d, $J_{CP} = 8.6$ Hz); 133.02; 133,17; 139.80; 158.49. ^{31}P NMR ($CDCl_3$), δ (ppm): –29.9. Anal. Calcd for $C_{26}H_{26}NOP$ (399.46): C 78.17; H 6.56; P 7.75. Found: C 77.94, H 6.44, P 7.69.

4-(Diphenylphosphoryl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole-3-carbaldehyde **3**

Method A. To a stirred solution of phosphine **2** (4.0 g, 0.01 mol) in DMF (10 mL) $P(O)Cl_3$ (1.68 g, 1.00 mL, 0.011 mol) was added at room temperature. The reaction mixture was stirred 2 h and poured into a saturated solution of $NaHCO_3$ (50 mL). The product was extracted with toluene, dried over Na_2SO_4, and evaporated in vacuo. The residue was recrystallized from acetonitrile as colorless solid. mp 195–196°C, yield 35%.

Method B (General for Compounds **6–8***).* To a stirred solution of the appropriate compound **6–8** (0.01 mol) in dry DMF (10 mL), $P(O)Cl_3$ (3.07 g, 1.83 mL, 0.02 mol) was added dropwise at 90°C. The reaction mixture was heated for 10 h at 100°C, and after cooling it was poured into 1 M aqueous solution of NaOH (30 mL) at 0°C. The resulting precipitate was filtered and recrystallized from acetonitrile. Yield 75% (for **6**); 65% (for **7**); 51% (for **8**).

^{1}H NMR (DMSO-d_6), δ (ppm): 1.35 (t, 3H, $J = 7.2$ Hz, OCH_2CH_3), 1.81 (s, 3H, CH_3), 2.23 (s, 3H, CH_3), 4.10 (q, 2H, $J = 7.2$ Hz, OCH_2CH_3), 7.09 (d, 2H, $J = 8.0$ Hz, Ar), 7.31 (d, 2H, $J = 8.0$ Hz, Ar), 7.53–7.60 (m, 6H, Ar), 7.66–7.70 (m, 4H, Ar), 9.68 (s, 1H, C(O)H). ^{13}C{^{1}H}NMR (DMSO-d_6), δ (ppm): 11.93; 12.14; 14.54; 63.46; 108.31; 108.27 (d, $J_{CP} = 120.7$ Hz); 115.24; 121.96 (d, $J_{CP} = 8.8$ Hz); 127.76; 128.46; 128.55; 129.23; 131.04; 131.12; 131.54; 134.53; 135.38; 138.90; 139.03; 140.07 (d, $J_{CP} = 8.8$ Hz); 158.89; 185.68. ^{31}P NMR (DMSO-d_6), δ (ppm): 22.97. *m/z* 444 $[M + 1]^+$. Anal. Calcd for $C_{27}H_{26}NO_3P$ (443.47): C 73.12; H 5.91; P 6.98. Found: C 72.88, H 5.67, P 6.69.

Borane Complex of 3-(Diphenylphosphino)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole **4**

To a stirred solution of phosphine **3** (0.55 g, 1.38 mmol) in dry THF (30 mL), 2.06 mL (4.13 mmol) of the BH_3·THF complex in THF was added through a rubber septum. After overnight, the solution was evaporated to its half volume under reduced pressure and methyltert-butyl ether (40 mL) and aqueous solution of hydrochloric acid (30 mL) were added. The organic layer was separated. The product was extracted three times from the aqueous solution with methyltertbutyl ether. All organic phases were combined and washed twice with water, dried over Na_2SO_4, and evaporated under reduced pressure. The product **4** is a colorless solid, mp 155–156°C. Yield: 98%.

^{1}H NMR (DMSO-d_6), δ (ppm): 1.03 br s (3H, BH_3), 1.36 (t, 3H, $J = 7.2$ Hz, OCH_2CH_3), 1.87 (s, 3H, CH_3), 1.91 (s, 3H, CH_3), 4.08 (q, 2H, $J = 7.2$ Hz, OCH_2CH_3), 5.71 br s (1H, CH, Pyr), 7.06 (d, 2H, $J = 8.0$ Hz, Ar), 7.34 (d, 2H, $J = 8.0$ Hz, Ar), 7.54 (m, 10H, Ar). ^{13}C{^{1}H}($CDCl_3$), δ (ppm): 12.48; 12.72; 14.61; 63.36; 100.31, 100.92 (d, $J_{CP} = 77.9$ Hz); 110.36; 114.97; 128,64; 128.72; 129.01; 129.51; 129.88 (d, $J_{CP} = 8.8$ Hz); 130.69, 131.16 (d, $J_{CP} = 59.2$ Hz); 130.75; 132.03; 132,11; 135.56 (d, $J_{CP} = 18.9$ Hz); 158.29. ^{31}P NMR (DMSO-d_6), δ (ppm): 7.26. Anal. Calcd for $C_{26}H_{29}BNOP$ (413): C 75.56, H 7.07, P 7.49. Found: C 75.15, H 7.24, P 7.21.

General Procedure for Synthesis of Compounds **6** *and* **7**

To a stirred solution of phosphine **2** (3.99 g, 0.01 mol) in dry benzene, sulfur or selenium (0.01 mol) was added at ambient temperature. The reaction mixture was refluxed for 0.5 h. Benzene was evaporated under reduced pressure, and the product was recrystallized from acetonitrile.

3-(Diphenylphosphorothioyl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole **6**. mp 177°C. Yield: 78%. ^{1}H NMR (DMSO-d_6), δ (ppm): 1.36 (t, 3H, $J = 7.2$ Hz, OCH_2CH_3), 1.89 (s, 3H, CH_3), 1.92 (s, 3H, CH_3), 4.08 (q, 2H, $J = 7.2$ Hz, OCH_2CH_3), 5.50 (d, 1H, $J = 3.6$ Hz, CH, Pyr), 7.06 (d, 2H, $J = 8.8$ Hz, Ar), 7.24 (d, 2H, $J = 8.8$ Hz, Ar), 7.45–7.60 (m, 6H, Ar), 7.75–7.80 (m, 4H, Ar). ^{13}C{^{1}H}($CDCl_3$), δ (ppm): 12.30; 12.58; 14.57; 63.35; 106.07, 106.96 (d, $J_{CP} = 111.9$ Hz); 109.93 (d, $J_{CP} = 10.1$ Hz); 115.00; 128.46 (d, $J_{CP} = 12.6$ Hz); 128.91 (d, $J_{CP} = 12.6$ Hz); 129.06; 129.32; 131.16 (d, $J_{CP} = 3.8$ Hz); 131.32 (d, $J_{CP} =$

10.1 Hz); 133.98; 134.66; 135.43 (d, J_{CP} = 17.6 Hz); 158.34. ^{31}P NMR (DMSO-d_6), δ (ppm): 32.8. *m/z* 432 $[M + 1]^+$. Anal. Calcd for $C_{26}H_{26}NOPS$ (431.15): C 72.37, H 6.07, P 7.18. Found: C 72.15, H 5.84, P 7.01.

3-(Diphenylphosphoroselenoyl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole **7**. mp 204°C. Yield: 82% ^{1}H NMR (DMSO-d_6), δ (ppm): 1.34 (t, 3H, J = 7.2 Hz, OCH_2CH_3), 1.87 (s, 3H, CH_3), 1.89 (s, 3H, CH_3), 4.07 (q, 2H, J = 7.2 Hz, OCH_2CH_3), 5.47 (d, 1H, J = 3.9 Hz, CH, Pyr), 7.05 (d, 2H, J = 8.4 Hz, Ar), 7.30 (d, 2H, J = 8.4 Hz, Ar), 7.45–7.55 (m, 6H, Ar), 7.71–7.79 (m, 4H, Ar). ^{13}C{^{1}H}($CDCl_3$), δ (ppm): 12.24; 12.64; 14.54; 63.33; 104.52, 105.32 (d, J_{CP} = 100.6 Hz); 110.01 (d, J_{CP} = 12.6 Hz); 114.99; 128.45 (d, J_{CP} = 12.6 Hz); 128.94 (d, J_{CP} = 12.6 Hz); 129.03; 129.25; 131.23 (d, J_{CP} = 2.51 Hz); 131.73 (d, J_{CP} = 3.77 Hz); 132.58, 133.20 (d, J_{CP} = 78.0 Hz); 135.42 (d, J_{CP} = 18.9 Hz); 158.36. ^{31}P NMR (DMSO-d_6), δ (ppm): 21.0. *m/z* 480 $[M + 1]^+$. Anal. Calcd for $C_{26}H_{26}NOPSe$ (478.42): C 65.27, H 5.48, P 6.47. Found: C 65.11, H 5.25, P 6.29.

3-(Diphenylphosphoryl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrole **8**

To a solution of phosphine **2** (3.99 g, 0.01 mol) in dry benzene (10 mL), hexachloroethane (2.37 g, 0.01 mol) was added. The reaction mixture was stirred for 0.5 h. The benzene layer was decanted, and the oil was dissolved in dichloromethane (20 mL). The solution was treated with 5% aqueous $NaHCO_3$ solution (10 mL). The organic layer was separated, dried over anhydrous sodium sulfate, and evaporated in vacuo. The product was recrystallized from hexane as a colorless solid. mp 135°C, yield 83%. ^{1}H NMR (CDCl3), δ (ppm): 1.41 (t, 3H, J = 7.2 Hz, OCH_2CH_3), 1.89 (s, 3H, CH_3), 2.03 (s, 3H, CH_3), 4.04 (q, 2H, J = 7.2 Hz, OCH_2CH_3), 5.60 (s, 1H, CH, Pyr), 6.93 (d, 2H, J = 8.8 Hz, Ar), 7.06 (d, 2H, J = 8.8 Hz, Ar), 7.41–7.45 (m, 6H, Ar), 7.71–76 (m, 4H, Ar). ^{13}C{^{1}H}($CDCl_3$), δ (ppm): 12.71; 12.85; 14.81; 63.79; 108.29, 107.26 (d, J_{CP} = 129.5); 110.35d (d, J_{CP} = 12.6 Hz); 114.99; 128.23d (d, J_{CP} = 12.6 Hz); 129.05; 129.61d (d, J_{CP} = 12.6 Hz); 130.30; 131.16; 131.74d (d, J_{CP} = 10.1 Hz); 134.59; 135.43; 137.41d (d, J_{CP} = 17.6 Hz); 158.87. ^{31}P NMR ($CDCl_3$), δ (ppm): 24.9. *m/z* 415 $[M]^+$. Anal. Calcd for $C_{26}H_{26}NO_2P$ (415.46): C 75.16; H 6.31; P 7.46. Found: C 75.13, H 6.29, P 7.26.

N-((4-(Diphenylphosphoryl)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrol-3-yl)methylene)-1-phenylethanamine **9**

A mixture of aldehyde **3** (4.43 g, 0.01 mol) and 1-phenylethanamine (2.42 g, 2.58 mL, 0.02 mol) in methanol (50 mL) was refluxed for 4 h. The solvent was evaporated under reduced pressure. The excess of 1-phenylethanamine was removed in vacuo by heating the residue on boiling water bath. The product is brown oil. Yield: 95%. ^{1}H NMR ($CDCl_3$), δ (ppm): 1,12 (d, 3H, J = 6.8 Hz, CH_3); 1.36 (t, 3H, J = 6.8 Hz, CH_3); 1.74 (s, 3H, CH_3); 2.21 (s, 3H, CH_3); 3.80 (q, 1H, J = 6.8 Hz); 4.09 (q, 2H, J = 6.8 Hz); 7.08–7.65 (m, 19H, Ar); 7.98 (s, 1H, CH). ^{13}C{^{1}H}($CDCl_3$), δ (ppm): 12.67; 12.73; 14.78; 25.72; 51.28; 63.83; 106.74, 107.72 (d, J_{CP} = 123.2 Hz); 115.44; 125.70; 126.46; 128.20; 128.49 (d, J_{CP} = 2.5 Hz); 128.57; 129.21 (d, J_{CP} = 6.3 Hz); 131.39; 131.90 (d, J_{CP} = 11.3 Hz); 147.83; 159.13. ^{31}P NMR ($CDCl_3$), δ (ppm): 22.0. *m/z* 546 $[M]^+$. Anal. Calcd for $C_{35}H_{35}N_2O_2P$ (546.64): C 76.90; H 6.45; P 5.67. Found: C 76.79, H 6.31, P 5.58.

N-((4-(Diphenylphosphino)-1-(4-ethoxyphenyl)-2,5-dimethyl-1H-pyrrol-3-yl)methylene)-1-phenylethanamine **10**

A mixture of phosphinoxide **9** (5.47g, 0.01 mol), triethylamine (0.2 mol), and trichlorosilane (0.05 mol) in toluene (40 mL) was heated in a sealed tube at 120°C for 6 h. After cooling to room temperature, the reaction mixture was treated with saturated aqueous $NaHCO_3$ solution. The organic layer was separated and filtered through a layer of alkaline aluminum oxide. The filtrate was evaporated in vacuo. Yield 57%. mp 123–125°C. ^{1}H NMR ($CDCl_3$), δ (ppm): 1.25 (d, 3H, J = 6.8 Hz, CH_3); 1.44 (t, 3H, J = 6.8 Hz, CH_3); 1.81 (s, 3H, CH_3); 1.91 (s, 3H, CH_3); 2.35 (s, 3H, CH_3); 3.22 (m, 1H, CH); 4.07 (q, 2H, J = 6.8 Hz, CH_2); 6.94–7.44 (m, 19H, Ar); 7.61 (br. s, 1H, CH). ^{31}P NMR ($CDCl_3$), δ (ppm): –32.9. Anal. Calcd for $C_{35}H_{35}N_2O_2P$ (530.64): C 79.22; H 6.65; P, 5.84. Found: C 79.15, H 6.43, P 5.79.

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