Supporting Information

Straightforward Synthesis of Bifunctional Phosphorus Phenols via Phosphination of in situ Generated o-Quinone Methides

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**General Information:** All compounds were fully characterized by spectroscopic data. The NMR spectra were recorded on a Bruker Avance III 400 M NMR at room temperature (1H: 400 MHz, 13C: 100 MHz, 31P NMR: 162 MHz external standard 85% H3PO4, 19F: 376 MHz), chemical shifts (δ) are expressed in parts per million (ppm), coupling constants (J) values are given in Hz, and CDCl3 or DMSO-d6 was used as the solvent. The high resolution mass spectra (HR-MS) data were recorded on Agilent 1290 Infinity LC & 6540 UHD Q-TOF mass spectrometer. IR spectra were recorded on a Thermo Scientific Nicolet 6700 Fourier IR spectrometer (AT-IR) with KBr pellet. Melting points were measured on a digital Electrothermal 9100 apparatus. Commercially available reagents were used without further purification. Solvents were treated prior to use according to the standard methods. The reactions were monitored by thin layer chromatography (TLC) using silica gel GF254. Column chromatography was performed on silica gel (200–300 mesh).

**Experimental Section**

![Reaction Scheme](image)

**General Procedure:** A reaction mixture of 2-(1-tosylalkyl)phenols 1 (0.50 mmol), potassium carbonate (0.6 mmol, 82.9 mg) and diphenylphosphine oxide 2a (0.6 mmol) in toluene (5 mL) was stirred at 110 °C for 4 h. Then water (20 mL) was added to the mixture. The organic layer was separated and the aqueous layer was extracted with dichloromethane (30 mL×3). The combined organic layer was dried by anhydrous sodium sulfate, concentrated in vacuo. The crude product was purified through column chromatography using dichloromethane and ethyl acetate to give the corresponding product 3.

**A Large-Scale Reaction:** A reaction mixture of 2-(phenyl(tosyl)methyl)phenol 1a (2.96 mmol, 1.002 g), potassium carbonate (3.55 mmol, 0.491 g) and diphenylphosphine oxide (3.55 mmol, 0.718 g) in toluene (25 mL) was stirred at 110 °C for 4 h. Then water (50 mL) was added to the mixture. The organic layer was separated and the aqueous layer was extracted with dichloromethane (50 mL×3). The combined organic layer was dried by anhydrous sodium sulfate, concentrated in vacuo. The crude product was purified by column chromatography (dichloromethane : ethyl acetate = 60:1 to 55:1, gradient) to afford 3a (0.933 g, 82% yield).
Synthesis of 2-((diphenylphosphoryl)(phenyl)methyl)phenyl acrylate 4: The mixture of 3a (0.50 mmol, 192.2 mg) and Et,N (0.75 mmol, 75.9 mg) in dry CH₂Cl₂ (10 mL) was cooled to 0 °C in an ice-water bath and acryloyl chloride (0.75 mmol, 67.9 mg) was added dropwise. The mixture was warmed to room temperature and stirred for 4 h. Following quenching with 2N HCl (10 mL), the mixture was extracted with dichloromethane (30 mLx3). The combined organic phases were sequentially washed with 2N HCl (20 mL), saturated aqueous potassium carbonate solution (20 mL) and brine (20 mL), and then dried over anhydrous sodium sulfate concentrated in vacuo. The crude product was purified by column chromatography (dichloromethane : ethyl acetate = 50:1) to afford 4 (154.4 mg, 70% yield).

(2-Hydroxyphenyl)(phenyl)methyl diphenylphosphine oxide (3a): 176.8 mg, 92% yield, unknown compound, pale white solid, mp: 238-240 °C, Rf = 0.45 (DCM/EA = 50/1); ¹H NMR (400 MHz, DMSO-d₆) δ 9.85 (s, 1H), 8.01 (d, J = 7.6 Hz, 1H), 7.83–7.77 (m, 2H), 7.75–7.65 (m, 2H), 7.48–7.34 (m, 8H), 7.17–7.13 (m, 2H), 7.12–7.03 (m, 1H), 6.96–6.92 (m, 1H), 6.72–6.68 (m, 2H), 5.62 (d, J_H,P = 9.2 Hz, 1H); ³¹P NMR (100 MHz, DMSO-d₆) δ 154.9 (d, J_C,P = 7.9 Hz), 137.8 (d, J_C,P = 4.4 Hz), 134.1 (d, J_C,P = 12.2 Hz), 133.1 (d, J_C,P = 12.4 Hz), 131.9 (d, J_C,P = 2.3 Hz), 131.8 (d, J_C,P = 2.3 Hz), 131.2 (d, J_C,P = 8.7 Hz), 130.9 (d, J_C,P = 8.7 Hz), 130.7 (d, J_C,P = 5.7 Hz), 130.3 (d, J_C,P = 6.3 Hz), 128.8 (d, J_C,P = 18.5 Hz), 128.8 (d, J_C,P = 4.1 Hz), 128.5, 128.4, 126.9, 125.0 (d, J_C,P = 3.1 Hz), 119.5, 115.7, 43.2 (d, J_C,P = 68.2 Hz); ³¹P NMR (162 MHz, DMSO-d₆) δ 31.3; IR (KBr): 3413, 3058, 1576, 1485, 1437, 1275, 1248, 1144, 1119, 811, 750, 691, 560, 530; HRMS (ESI) calcd for C₁₇H₁₄O₂P [(M+H)⁺]: 385.1352. found: 385.1352.

(2-Hydroxyphenyl)(o-toly)methyl diphenylphosphine oxide (3b): 175.3 mg, 88% yield, unknown compound, pale white solid, mp: 232-235 °C, Rf = 0.42 (DCM/EA = 50/1); ¹H NMR (400 MHz, DMSO-d₆) δ 9.75 (s, 1H), 8.07 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 7.7 Hz, 1H), 7.78–7.68 (m, 2H), 7.55–7.38 (m, 6H), 7.36–7.31 (m, 2H), 7.12 (t, J = 7.5 Hz, 1H), 7.02 (t, J = 7.4 Hz, 1H), 6.98–6.89 (m, 2H), 6.74–6.63 (m, 2H), 5.66 (d, J_H,P = 9.5 Hz, 1H), 2.16 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆) δ 154.8 (d, J_C,P = 7.4 Hz), 137.0 (d, J_C,P = 7.8 Hz), 136.5 (d, J_C,P = 3.6 Hz), 133.9 (d, J_C,P = 95.5 Hz), 133.2 (d, J_C,P = 96.6 Hz), 131.9 (d, J_C,P = 2.8 Hz), 131.8 (d, J_C,P = 2.7 Hz), 131.3 (d, J_C,P = 8.7 Hz), 131.1 (d, J_C,P = 5.4 Hz), 131.0 (d, J_C,P = 8.8 Hz), 130.6 (d, J_C,P = 5.1 Hz), 130.3, 128.8 (d, J_C,P = 2.3 Hz), 128.6 (d, J_C,P = 2.3 Hz), 128.2, 127.0, 126.1, 124.4 (d, J_C,P = 3.9 Hz), 119.4, 115.2, 38.5 (d, J_C,P = 68.4 Hz), 19.9; ³¹P NMR (162 MHz, DMSO-d₆) δ 31.4; IR (KBr): 3435, 3039, 2955, 2733, 1596, 1488, 1457, 1438, 1383, 1277, 1157, 1112, 849, 785, 697, 560, 527; HRMS (ESI) calcd for C₂₉H₂₅O₃P [(M+H)⁺]: 399.1508. found: 399.1508.

(2-Hydroxyphenyl)(m-toly)methyl diphenylphosphine oxide (3c): 179.3 mg, 90% yield, unknown compound, pale white solid, mp: 238-240 °C, Rf = 0.42 (DCM/EA = 50/1); ¹H NMR (400 MHz, DMSO-d₆) δ 9.85 (s, 1H), 7.99 (d, J = 7.6 Hz, 1H), 7.86–7.75 (m, 2H), 7.75–7.63 (m, 2H), 7.45–7.35 (m, 6H), 7.29 (d, J = 7.8 Hz, 1H), 7.24 (s, 1H), 7.04 (t, J = 7.6 Hz, 1H), 6.99–6.84 (m, 2H), 6.72–6.68
(2-Hydroxyphenyl)(p-tolyl)methyl)diphenylphosphine oxide (3d): 169.3 mg, 85% yield, unknown compound, pale white solid, mp: 241-245 °C, Rf = 0.42 (DCM/EA = 50/1); 1H NMR (400 MHz, CDCl₃) δ 10.54 (s, 1H), 7.76–7.65 (m, 2H), 7.65–7.54 (m, 2H), 7.51–7.40 (m, 2H), 7.38–7.33 (m, 4H), 7.27–7.24 (m, 2H), 7.07–7.03 (m, 2H), 6.96–6.88 (m, 3H), 6.67 (t, J = 7.4 Hz, 1H), 4.80 (d, J_H,P = 12.4 Hz, 1H), 2.22 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 155.8 (d, J_C,P = 4.6 Hz), 136.8 (d, J_C,P = 1.9 Hz), 132.6 (d, J_C,P = 4.0 Hz), 132.3 (d, J_C,P = 8.6 Hz), 132.1 (d, J_C,P = 1.8 Hz), 132.1 (d, J_C,P = 7.7 Hz), 131.4 (d, J_C,P = 9.2 Hz), 131.2 (d, J_C,P = 8.8 Hz), 130.7 (d, J_C,P = 98.1 Hz), 130.4 (d, J_C,P = 99.1 Hz), 129.6 (d, J_C,P = 6.2 Hz), 129.2 (d, J_C,P = 1.0 Hz), 128.5 (d, J_C,P = 3.5 Hz), 128.5 (d, J_C,P = 20.2 Hz), 123.7 (d, J_C,P = 4.7 Hz), 120.2, 119.8 (d, J_C,P = 1.2 Hz), 53.1 (d, J_C,P = 65.3 Hz), 21.0; 31P NMR (162 MHz, CDCl₃) δ 38.3; IR (KBr): 3421, 3062, 2925, 2739, 1596, 1511, 1456, 1438, 1386, 1154, 852, 792, 753, 723, 560, 530, 494; HRMS (ESI) calcd for C₁₈H₁₅O₂P [(M+H)+]: 399.1508, found: 399.1506.

(2-Hydroxyphenyl)(4-methoxyphenyl)methyl)diphenylphosphine oxide (3e): 178.2 mg, 86% yield, unknown compound, pale white solid, mp: 204–205 °C, Rf = 0.32 (DCM/EA = 50/1); 1H NMR (400 MHz, CDCl₃) δ 10.54 (s, 1H), 7.75–7.67 (m, 2H), 7.62–7.54 (m, 2H), 7.49–7.41 (m, 2H), 7.39–7.33 (m, 4H), 7.30–7.26 (m, 2H), 7.08–7.03 (m, 2H), 6.92–6.88 (m, 1H), 6.72–6.64 (m, 3H), 4.78 (d, J_H,P = 12.4 Hz, 1H), 3.70 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 158.6 (d, J_C,P = 1.7 Hz), 155.7 (d, J_C,P = 4.7 Hz), 132.2 (d, J_C,P = 8.8 Hz), 132.1 (d, J_C,P = 7.0 Hz), 132.1 (d, J_C,P = 1.2 Hz), 131.4 (d, J_C,P = 9.2 Hz), 131.2 (d, J_C,P = 8.9 Hz), 130.8 (d, J_C,P = 6.0 Hz), 130.6 (d, J_C,P = 97.7 Hz), 130.3 (d, J_C,P = 99.1 Hz), 129.2, 128.5 (d, J_C,P = 3.6 Hz), 128.5 (d, J_C,P = 20.1 Hz), 127.6 (d, J_C,P = 4.0 Hz), 123.8 (d, J_C,P = 4.4 Hz), 120.2, 119.9 (d, J_C,P = 1.0 Hz), 113.9, 55.2, 52.8 (d, J_C,P = 65.6 Hz); 31P NMR (162 MHz, CDCl₃) δ 38.4; IR (KBr): 3425, 3060, 2958, 1608, 1510, 1455, 1348, 1384, 1249, 1154, 1118, 1031, 831, 784, 754, 561, 529; HRMS (ESI) calcd for C₁₆H₁₄O₂P [(M+H)+]: 415.1458, found: 415.1457.

(2-Hydroxyphenyl)(4-(trifluoromethyl)phenyl)methyl)diphenylphosphine oxide (3f): 180.8 mg, 80% yield, unknown compound, pale white solid, mp: 187–189 °C, Rf = 0.46 (DCM/EA = 50/1); 1H NMR (400 MHz, CDCl₃) δ 10.02 (s, 1H), 7.76–7.70 (m, 2H), 7.62–7.50 (m, 4H), 7.49–7.28 (m, 9H), 7.03 (t, J = 7.8 Hz, 1H), 6.88 (d, J = 8.1 Hz, 1H), 6.70 (t, J = 7.5 Hz, 1H), 5.12 (d, J_H,P = 10.9 Hz, 1H); 13C NMR (100 MHz, CDCl₃) δ 155.2 (d, J_C,P = 5.8 Hz), 140.2 (d, J_C,P = 2.9 Hz), 132.3 (d, J_C,P = 2.8 Hz), 132.2 (d, J_C,P = 2.9 Hz), 131.8 (d, J_C,P = 7.6 Hz), 131.2 (d, J_C,P = 19.5 Hz), 131.2, 130.9 (d, J_C,P = 5.4 Hz), 130.7, 130.2 (d, J_C,P = 6.1 Hz), 129.9 (d, J_C,P = 4.6 Hz), 129.3, 129.0 (d, J_C,P = 13.1 Hz).
Hz), 128.7 (d, $J_{CP} = 1.7$ Hz), 128.6 (d, $J_{CP} = 1.8$ Hz), 125.4, 125.2 (q, $J_{CP} = 4.0$ Hz), 123.0 (d, $J_{CP} = 4.4$ Hz), 122.7, 120.4, 118.6, 50.3 (d, $J_{CP} = 66.1$ Hz); $^{31}$P NMR (162 MHz, CDCl$_3$) δ 37.1; $^{19}$F NMR (376 MHz, CDCl$_3$) δ -62.6; IR (KBr): 3428, 3061, 1619, 1455, 1439, 1325, 1167, 1121, 1068, 761, 726, 699, 557; HRMS (ESI) calcd for C$_{28}$H$_3$F$_3$O$_7$P [(M+H)]: 453.1226, found: 453.1223.

((4-Bromo-2-hydroxyphenyl)(phenyl)methyl)diphenylphosphine oxide (3g): 189.9 mg, 82% yield, unknown compound, pale white solid, mp: 270-271 °C, $R_t = 0.46$ (DCM/EA = 50/1); $^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.35 (s, 1H), 7.99-7.96 (m, 1H), 7.83-7.78 (m, 2H), 7.74-7.68 (m, 2H), 7.52-7.34 (m, 8H), 7.18-7.07 (m, 2H), 7.12-7.05 (m, 1H), 6.94-6.91 (m, 1H), 6.89-6.87 (m, 1H), 5.56 (d, $J_{HP} = 9.1$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 156.2 (d, $J_{CP} = 7.7$ Hz), 137.2 (d, $J_{CP} = 4.4$ Hz), 133.8 (d, $J_{CP} = 23.3$ Hz), 132.8 (d, $J_{CP} = 24.3$ Hz), 132.3 (d, $J_{CP} = 5.5$ Hz), 132.1 (d, $J_{CP} = 2.9$ Hz), 131.9 (d, $J_{CP} = 2.6$ Hz), 131.2 (d, $J_{CP} = 8.7$ Hz), 130.9 (d, $J_{CP} = 8.8$ Hz), 130.2 (d, $J_{CP} = 6.2$ Hz), 128.9 (d, $J_{CP} = 3.2$ Hz), 128.9 (d, $J_{CP} = 26.0$ Hz), 128.6, 127.1, 124.7 (d, $J_{CP} = 3.2$ Hz), 122.3, 120.7, 118.3, 42.7 (d, $J_{CP} = 67.5$ Hz); $^{31}$P NMR (162 MHz, DMSO-$d_6$) δ 31.0; IR (KBr): 3422, 3025, 2901, 2718, 1589, 1491, 1419, 1260, 1145, 1116, 1095, 1072, 887, 859, 837, 726, 537, 501; HRMS (ESI) calcd for C$_{28}$H$_3$BrO$_7$P [(M+H)]: 463.0457, found: 463.0453.

((2-Hydroxy-4-methoxyphenyl)(3-toly)methyl)diphenylphosphine oxide (3h): 186.4 mg, 87% yield, unknown compound, pale white solid, mp: 240-241 °C, $R_t = 0.34$ (DCM/EA = 50/1); $^1$H NMR (400 MHz, CDCl$_3$) δ 10.80 (s, 1H), 7.76-7.67 (m, 2H), 7.60-7.55 (m, 2H), 7.48-7.41 (m, 2H), 7.39-7.32 (m, 4H), 7.23-7.20 (m, 2H), 6.95-6.90 (m, 3H), 6.47 (d, $J = 2.6$ Hz, 1H), 6.27-6.24 (m, 1H), 4.73 (d, $J_{HP} = 12.7$ Hz, 1H), 3.67 (s, 3H), 2.21 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 160.4 (d, $J_{CP} = 1.4$ Hz), 157.0 (d, $J_{CP} = 4.6$ Hz), 136.7 (d, $J_{CP} = 2.0$ Hz), 132.9 (d, $J_{CP} = 8.9$ Hz), 132.9 (d, $J_{CP} = 3.6$ Hz), 132.1 (d, $J_{CP} = 5.4$ Hz), 132.1, 131.4 (d, $J_{CP} = 9.2$ Hz), 131.2 (d, $J_{CP} = 8.8$ Hz), 130.8 (d, $J_{CP} = 97.8$ Hz), 130.4 (d, $J_{CP} = 98.5$ Hz), 129.5 (d, $J_{CP} = 6.1$ Hz), 129.2 (d, $J_{CP} = 1.3$ Hz), 128.5 (d, $J_{CP} = 2.2$ Hz), 128.5 (d, $J_{CP} = 25.8$ Hz), 116.0 (d, $J_{CP} = 4.6$ Hz), 107.0, 104.4 (d, $J_{CP} = 1.5$ Hz), 55.1, 52.5 (d, $J_{CP} = 65.6$ Hz), 47.9, 21.0; $^{31}$P NMR (162 MHz, CDCl$_3$) δ 38.7; IR (KBr): 3412, 3058, 3007, 2940, 2898, 1736, 1615, 1524, 1437, 1153, 1036, 854, 802, 720, 696, 536; HRMS (ESI) calcd for C$_{28}$H$_3$O$_7$P [(M+H)]: 429.1614, found: 429.1617.

(1-(2-Hydroxyphenyl)ethyl)diphenylphosphine oxide (3i): 135.4 mg, 84% yield, unknown compound, pale white solid, mp: 198-200 °C, $R_t = 0.45$ (DCM/EA = 50/1); $^1$H NMR (400 MHz, CDCl$_3$) δ 10.20 (s, 1H), 7.94-7.80 (m, 2H), 7.70-7.48 (m, 5H), 7.47-7.28 (m, 3H), 7.13-7.04 (m, 1H), 6.96-6.93 (m, 1H), 6.84-6.81 (m, 1H), 6.67 (t, $J = 7.4$ Hz, 1H), 3.64 (dt, $J = 9.4$, 7.4 Hz, 1H), 1.59 (dd, $J = 15.9$, 7.5 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 156.3 (d, $J_{CP} = 4.1$ Hz), 132.3 (d, $J_{CP} = 2.9$ Hz), 132.0 (d, $J_{CP} = 2.8$ Hz), 131.3 (d, $J_{CP} = 7.3$ Hz), 131.2 (d, $J_{CP} = 8.9$ Hz), 130.9 (d, $J_{CP} = 9.1$ Hz), 130.2 (d, $J_{CP} = 6.5$ Hz), 129.2, 128.9 (d, $J_{CP} = 2.1$ Hz), 128.9 (d, $J_{CP} = 11.5$ Hz), 128.5 (d, $J_{CP} = 11.7$ Hz), 124.5 (d, $J_{CP} = 5.8$ Hz), 120.2, 119.9 (d, $J_{CP} = 2.1$ Hz), 40.7 (d, $J_{CP} = 67.2$ Hz), 13.0 (d, $J_{CP} = 2.2$ Hz); $^{31}$P NMR (162 MHz, CDCl$_3$) δ 41.6; IR (KBr): 3426, 3060, 2961, 1592, 1451, 1437, 1390, 1158, 1119, 1091, 1020, 778, 751, 721, 696, 605, 556; HRMS (ESI) calcd for C$_{23}$H$_2$O$_7$P [(M+H)]: 323.1195, found: 323.1196.
(6-Hydroxybenzo[d][1,3]dioxol-5-yl)(phenyl)methyl)diphenylphosphine oxide (3j): 177.8 mg, 83% yield, unknown compound, pale white solid, mp: 253-255 °C, Rf = 0.42 (DCM/EA = 40/1); 1H NMR (400 MHz, DMSO-d6) δ 9.64 (s, 1H), 7.84–7.78 (m, 2H), 7.73–7.64 (m, 2H), 7.58–7.56 (m, 1H), 7.52–7.31 (m, 8H), 7.17–7.13 (m, 2H), 7.12–7.04 (m, 1H), 6.35–6.34 (m, 1H), 5.85–5.80 (m, 2H), 5.54 (d, ḟJHP = 9.6 Hz, 1H); 13C NMR (100 MHz, DMSO-d6) δ 149.3 (d, fJC= 8.2 Hz), 146.2, 139.5, 137.5 (d, fJC= 4.1 Hz), 133.6 (d, fJC= 15.8 Hz), 132.6 (d, fJC= 15.7 Hz), 131.5 (d, fJC= 2.6 Hz), 131.3 (d, fJC = 2.4 Hz), 131.4 (d, fJC = 14.7 Hz), 130.5 (d, fJC = 32.2 Hz), 130.5 (d, fJC = 14.8 Hz), 129.6 (d, fJC = 6.2 Hz), 128.4 (d, fJC = 4.5 Hz), 128.4 (d, fJC = 27.0 Hz), 128.0, 126.4, 115.7 (d, fJC = 3.3 Hz), 109.2 (d, fJC = 5.5 Hz), 100.6, 97.5, 42.3 (d, fJC = 68.2 Hz); 31P NMR (162 MHz, DMSO-d6) δ 31.7; HRMS (ESI) calcd for C16H22O5P [(M+H)]+: 429.1256, found: 429.1251.

(6-Hydroxybenzo[d][1,3]dioxol-5-yl)(ρ-tolyl)methyl)diphenylphosphine oxide (3k): 183.6 mg, 83% yield, unknown compound, pale white solid, mp: 236-237 °C, Rf = 0.42 (DCM/EA = 40/1); 1H NMR (400 MHz, CDCl3) δ 10.40 (s, 1H), 7.74–7.65 (m, 2H), 7.62–7.52 (m, 2H), 7.51–7.44 (m, 2H), 7.41–7.33 (m, 4H), 7.24–7.16 (m, 2H), 6.97–6.93 (m, 2H), 6.49–6.45 (m, 2H), 5.86–5.74 (m, 2H), 4.66 (d, ḟJHP = 13.5 Hz, 1H), 2.23 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 151.2 (d, ḟJC= 4.9 Hz), 147.9 (d, fJC = 1.2 Hz), 141.0, 136.9 (d, fJC = 1.8 Hz), 132.7 (d, fJC = 3.3 Hz), 132.1 (d, fJC = 4.9 Hz), 132.1, 131.4 (d, fJC = 9.2 Hz), 131.2, 131.2 (d, fJC = 8.8 Hz), 130.7 (d, fJC = 98.2 Hz), 130.4 (d, fJC = 98.1 Hz), 129.6 (d, fJC = 6.4 Hz), 129.2, 128.7, 128.5, 128.4, 115.2 (d, fJC = 4.8 Hz), 110.5 (d, fJC = 8.8 Hz), 101.5 (d, fJC = 1.5 Hz), 101.1, 52.4 (d, fJC = 65.8 Hz), 11.0; 31P NMR (162 MHz, CDCl3) δ 38.6; IR (KBr): 3422, 3055, 2923, 1623, 1504, 1438, 1158, 1039, 938, 975, 721, 696, 538; HRMS (ESI) calcd for C25H25O5P [(M+H)]+: 443.1407, found: 443.1406.

(6-Hydroxybenzo[d][1,3]dioxol-5-yl)(4-methoxyphenyl)methyl)diphenylphosphine oxide (3l): 192.5 mg, 84% yield, unknown compound, pale white solid, mp: 231-233 °C, Rf = 0.42 (DCM/EA = 40/1); 1H NMR (400 MHz, DMSO-d6) δ 9.58 (s, 1H), 7.80–7.75 (m, 2H), 7.70–7.65 (m, 2H), 7.53–7.32 (m, 9H), 6.75–6.68 (m, 2H), 6.31 (s, 1H), 5.82 (m, 2H), 5.46 (d, ḟJHP = 9.8 Hz, 1H), 3.63 (s, 3H); 13C NMR (100 MHz, DMSO-d6) δ 158.2, 149.7 (d, ḟJC = 7.8 Hz), 146.6, 140.0, 134.2 (d, fJC = 15.5 Hz), 133.3 (d, fJC = 16.4 Hz), 131.8 (d, fJC = 12.2 Hz), 131.1 (d, fJC = 4.5 Hz), 131.1, 130.9 (d, fJC = 8.6 Hz), 129.9 (d, fJC = 4.0 Hz), 128.9 (d, fJC = 1.3 Hz), 128.9 (d, fJC = 20.2 Hz), 116.7 (d, fJC = 2.9 Hz), 114.0, 109.6 (d, fJC = 3.8 Hz), 101.1, 98.0, 55.4, 41.8 (d, fJC = 69.2 Hz); 31P NMR (162 MHz, DMSO-d6) δ 31.9; IR (KBr): 3425, 3061, 2960, 2903, 1607, 1509, 1439, 1245, 1174, 1154, 1113, 1048, 942, 877, 833, 727, 700, 593, 542, 523; HRMS (ESI) calcd for C25H24O5P [(M+H)]+: 459.1356, found: 459.1354.

2-((Diphenylphosphoryl)(phenyl)methyl)phenyl acrylate (4): 154.3 mg, 70% yield, unknown compound, pale white solid, mp: 78-79 °C, Rf = 0.38 (DCM/EA = 40/1); 1H NMR (400 MHz, CDCl3) δ 8.35–8.25 (m, 1H), 7.74–7.62 (m, 2H), 7.53–7.44 (m, 2H), 7.44–7.31 (m, 4H), 7.30–7.24 (m, 4H), 7.21–7.15 (m, 2H), 7.14–7.10 (m, 3H), 7.01–6.92 (m, 1H), 6.53 (dd, ḟJ = 17.3, 1.3 Hz, 1H), 6.29 (dd, ḟJ
= 17.3, 10.4 Hz, 1H), 6.03 (dd, J = 10.4, 1.3 Hz, 1H), 4.93 (d, J_H,P = 9.2 Hz, 1H);
^{13}C NMR (100 MHz, CDCl_3) δ 163.9, 148.2 (d, J_C,P = 8.8 Hz), 135.6 (d, J_C,P = 5.2 Hz), 133.1, 132.5 (d, J_C,P = 99.7 Hz), 132.1 (d, J_C,P = 97.2 Hz), 131.6 (d, J_C,P = 3.0 Hz), 131.5 (d, J_C,P = 2.6 Hz), 131.3, 131.3 (d, J_C,P = 2.2 Hz), 131.2 (d, J_C,P = 2.3 Hz), 130.2 (d, J_C,P = 6.1 Hz), 129.9 (d, J_C,P = 2.9 Hz), 128.4 (d, J_C,P = 11.6 Hz), 128.3 (d, J_C,P = 1.3 Hz), 128.2, 128.1, 127.9 (d, J_C,P = 62.5 Hz), 127.0 (d, J_C,P = 2.1 Hz), 126.4, 45.70 (d, J_C,P = 67.4 Hz); ^{31}P NMR (162 MHz, CDCl_3) δ 31.6; IR (KBr): 3052, 3019, 2925, 1739, 1486, 1436, 1402, 1139, 982, 800, 724, 693, 553, 515; HRMS (ESI) calcd for C_{28}H_{24}O_{3}P [(M+H)^+]: 439.1458, found: 439.1455.
$^1$H NMR (400 MHz, DMSO-$d_6$)
$^{13}$C NMR (100 MHz, DMSO-$d_6$)
$^{31}$P NMR (162 MHz, DMSO-$d_6$)

$\text{Ph}_2\text{P}$

$\text{Ph}_2\text{P}$

3a OH
$^{31}$P NMR (162 MHz, DMSO-$d_6$)
$^{13}$C NMR (100 MHz, DMSO-$d_6$)
$^{31}$P NMR (162 MHz, DMSO-$d_6$)
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{31}\text{P NMR (162 MHz, CDCl}_3\text{)}$

[Chemical structure diagram]
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{31}$P NMR (162 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{31}P$ NMR (162 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, DMSO-$d_6$)
$^{13}$C NMR (100 MHz, DMSO-$d_6$)
$^{31}$P NMR (162 MHz, DMSO-$d_6$)
$^{31}$P NMR (162 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, DMSO-$d_6$)
$^{31}$P NMR (162 MHz, DMSO-$d_6$)
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{31}$P NMR (162 MHz, CDCl$_3$)