

# Benzene Activation at a Charge Neutral Zwitterionic Platinum(II) Complex

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## Experimental Section

Unless otherwise noted, all syntheses were carried out in the absence of water and dioxygen, using standard Schlenk and glovebox techniques. Tetrahydrofuran, diethyl ether, petroleum ether, dichloromethane, acetonitrile, toluene, and benzene were deoxygenated and dried by thorough sparging with N<sub>2</sub> gas followed by passage through an activated alumina column and were stored over 3-Å molecular sieves. Pentane, hexanes, and ethanol were deoxygenated by repeated evacuation under reduced pressure followed by introduction of dinitrogen and were dried by storing over 3-Å molecular sieves. Nonhalogenated solvents were typically tested with a standard solution of sodium benzophenone ketyl in tetrahydrofuran in order to confirm effective oxygen and moisture removal. Deuterated chloroform, benzene, dichloromethane, acetonitrile, and acetone were purchased from Cambridge Isotope Laboratories, Inc. and were degassed by repeated freeze-pump-thaw cycles and dried over activated 3-Å molecular sieves prior to use. B(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub> was recrystallized from pentane at -35 °C prior to use. (COD)Pt(Cl)<sub>2</sub>,<sup>1</sup> (COD)Pt(Me)(Cl),<sup>1</sup> (COD)Pt(Me)<sub>2</sub>,<sup>2</sup> Ph<sub>2</sub>BCl,<sup>3</sup> Ph<sub>2</sub>PMe,<sup>4</sup> Ph<sub>2</sub>PCH<sub>2</sub>Li(TMEDA),<sup>5</sup> and 5-azonia-spiro[4.4]nonane bromide (ASNBr)<sup>6</sup> were prepared by literature methods. [Pr<sub>2</sub>EtNH][BPh<sub>4</sub>] was prepared by acidifying an aqueous solution of <sup>1</sup>Pr<sub>2</sub>EtN and NaBPh<sub>4</sub> with aqueous HCl. (COD)Pt(Me)(Ph)<sup>7</sup> was prepared by addition of PhMgBr to a cold (-35 °C) dichloromethane solution of (COD)Pt(Me)(Cl). All other chemicals were purchased from Aldrich, Strem, Alfa Aesar, or Pressure Chemicals and used without further purification. NMR spectra were recorded at ambient temperature on Varian Mercury 300 MHz and Inova 500 MHz, and Joel 400 MHz spectrometers, unless otherwise noted. <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts were referenced to residual solvent. <sup>31</sup>P NMR and <sup>11</sup>B NMR chemical shifts are reported relative to an external standard of 85% H<sub>3</sub>PO<sub>4</sub> or neat BF<sub>3</sub>·Et<sub>2</sub>O respectively. IR spectra were recorded on a Bio-Rad Excalibur FTS 3000 spectrometer controlled by Win-IR Pro software. Elemental Analyses were performed by Desert Analytics, Tucson, AZ. X-ray diffraction experiments were carried out by the Beckmann Institute Crystallographic Facility on a Bruker Smart 1000 CCD diffractometer.

**[Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][ASN] (1).** Solid yellow Ph<sub>2</sub>PCH<sub>2</sub>Li(TMEDA) (4.82 g, 15.0 mmol) was dissolved in diethyl ether (180 mL) in a Schlenk flask with a stir bar and sealed with a septum. The reaction vessel was cooled to -78 °C in a dry ice/acetone bath. Ph<sub>2</sub>BCl (1.514 g, 7.553 mmol), dissolved in toluene (10 mL), was introduced dropwise via syringe to the cooled reaction flask. The reaction was stirred and warmed gradually to r.t. over 14 h, providing a pale yellow precipitate. Volatiles were removed in vacuo, and the resulting solids were isolated in a drybox on a sintered glass frit and washed with diethyl ether [5 x 10 mL]. Drying in vacuo provided pale yellow solid [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][Li(TMEDA)<sub>2</sub>] (5.67 g). Crystals suitable for an X-ray diffraction experiment were grown by slowly cooling a hot toluene solution of [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][Li(TMEDA)<sub>2</sub>].

Solid [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][Li(TMEDA)<sub>2</sub>] was dissolved in ethanol (40 mL). ASNBr (1.8 g, 8.7 mmol) was dissolved in ethanol (8 mL) and added to stirring **1**. A white precipitate formed immediately. The mixture was stirred for 10 min, and white solids were subsequently collected by filtration. The solids were washed with ethanol [2 x 10 mL] and diethyl ether [3 x 10 mL] and dried in vacuo for 24 h, providing **1** as a pure, white solid (4.30 g, 6.23 mmol, 83.1 %).

<sup>1</sup>H NMR (300 MHz, d<sub>6</sub>-acetone): δ = 7.29 (b, 4H, *ortho* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 7.17 (m, 8H, *ortho* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 7.00 (m, 12H, *meta* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub> and P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 6.74 (m, 4H, *para* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 6.62 (m, 2H, *para* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 3.65 (m, 8H, ((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N), δ = 2.23 (m, 8H, ((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N), δ = 1.64 (b, 4H, Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (125.7 MHz, d<sub>6</sub>-acetone): δ = 165 (b, *ipso* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 147.4 (d, *ipso* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>, <sup>1</sup>J<sub>P-C</sub> = 22 Hz), δ = 134.7 (s, *ortho* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 133.6 (d, *ortho* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>, <sup>2</sup>J<sub>P-C</sub> = 19 Hz), δ = 127.1 (s, *meta* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>, <sup>3</sup>J<sub>P-C</sub> = 6 Hz), δ = 126.0 (s, *para* P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 125.3 (s, *meta* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 121.5 (s, *para* B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 63.1 (((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N), δ = 25.7 (b, [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]), δ = 22.1 (((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N). <sup>31</sup>P{<sup>1</sup>H} NMR (121.4 MHz, d<sub>6</sub>-acetone): δ = -8.78 (<sup>2</sup>J<sub>P-B</sub> = 10.0 Hz). <sup>11</sup>B{<sup>1</sup>H} NMR (128.3 MHz, d<sub>6</sub>-acetone): δ = -12.6. Anal. Calcd. for C<sub>46</sub>H<sub>50</sub>BNP<sub>2</sub>: C, 80.11; H, 7.31; N, 2.03. Found: C, 79.89; H, 7.45; N, 2.15.

**[Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>Pt(CH<sub>3</sub>)<sub>2</sub>][ASN] (2).** Solid **1** (391.8 mg, 0.5680 mmol) was suspended in THF (6 mL). A solution of (COD)Pt(Me)<sub>2</sub> (189.3 mg, 0.5679 mmol) in THF (2 mL) was added to the suspension, and the reaction homogenized as it stirred. A white precipitate formed after 1 h. The resulting mixture was concentrated in vacuo and triturated with pentane [2 x 2 mL]. The off white solids were dried in vacuo, providing **2** as an off-white solid (511.2 mg, 98.4 %). Crystals suitable for X-ray diffraction were grown from slow evaporation of an acetonitrile solution of **2**.

<sup>1</sup>H NMR (300 MHz, d<sub>6</sub>-acetone): δ = 7.40 (m, 8H, P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 7.07 (m, 12H, B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>, (P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>)<sub>2</sub>), δ = 6.88 (m, 4H, B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 6.64 (m, 4H, *para*-(P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>)<sub>2</sub>), δ = 6.58 (m, 2H, *para*-B(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>), δ = 3.71 (m, 8H, ((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N), δ = 2.26 (m, 8H, ((CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>N), δ = 1.98 (b, 4H, Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>), δ = 0.08 (t, 6H, Pt(CH<sub>3</sub>)<sub>2</sub>, <sup>3</sup>J<sub>P-H</sub> = 12 Hz, <sup>2</sup>J<sub>P-H</sub> = 68 Hz).

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$^{13}\text{C}\{^1\text{H}\}$  NMR (125.7 MHz,  $d_6$ -acetone):  $\delta = 167$  (b, *ipso*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 140.1$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 39.0$  Hz),  $\delta = 134.4$  (m, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 133.5$  (s, *ortho*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 128.2$  (s, *meta*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 127.3$  (m, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 126.3$  (m, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 122.0$  (s, *para*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 63.7$  ( $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 22.9$  (b,  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2]$ ),  $\delta = 22.8$  ( $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 5.5$  (dd,  $\text{Pt}(\text{CH}_3)_2$ ,  $^1J_{\text{Pt-C}} = 600$  Hz,  $^2J_{\text{P-C}} = 103$  Hz,  $^2J_{\text{P-C}} = 9.1$  Hz).  $^{31}\text{P}\{^1\text{H}\}$  NMR (121.4 MHz,  $d_6$ -acetone):  $\delta = 20.60$  ( $^1J_{\text{Pt-P}} = 1892$  Hz).  $^{11}\text{B}\{^1\text{H}\}$  NMR (128.3 MHz,  $d_6$ -acetone):  $\delta = -13.7$ . Anal. Calcd. for  $\text{C}_{48}\text{H}_{56}\text{BNP}_2\text{Pt}$ : C, 63.02; H, 6.17; N, 1.53. Found: C, 62.97; H, 5.90; N, 1.81.

**$[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2\text{Pt}(\text{CH}_3)(\text{C}_6\text{H}_5)][\text{ASN}]$  (3).** A THF solution (1 mL) of  $(\text{COD})\text{Pt}(\text{Me})(\text{Ph})$  (70.6 mg, 0.179 mmol) was added to a stirring suspension of **1** (123.1 mg, 0.1785 mmol) in THF (2 mL). The reaction was stirred for 30 min and became homogeneous. The solution was concentrated in vacuo, and off-white solids were precipitated with diethyl ether (2 mL). The supernatant was removed, and the solids were washed with ethanol [2 x 2 mL] and diethyl ether [2 x 2 mL] and dried in vacuo, producing off-white **3** (122.4 mg, 70.2%).

$^1\text{H}$  NMR (300 MHz,  $d_6$ -acetone):  $\delta = 7.47$  (m, 4H),  $\delta = 7.24$  (m, 4H),  $\delta = 7.12$  (m, 2H),  $\delta = 7.09$  (m, 4H),  $\delta = 6.98$  (m, 4H),  $\delta = 6.87$  (m, 8H),  $\delta = 6.62$  (m, 4H),  $\delta = 6.57$  (m, 2H),  $\delta = 6.43$  (m, 2H),  $\delta = 6.29$  (m, 1H, *para*  $\text{Pt-C}_6\text{H}_5$ ),  $\delta = 3.69$  (m, 8H,  $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 2.26$  (m, 8H,  $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 2.10$  (b,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ),  $\delta = 2.02$  (b,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ),  $\delta = 0.08$  (dd, 3H,  $\text{Pt}(\text{CH}_3)_2$ ,  $^3J_{\text{P-H(cis)}} = 6.9$  Hz,  $^3J_{\text{P-H(trans)}} = 7.8$  Hz,  $^2J_{\text{Pt-H}} = 69$  Hz).  $^{13}\text{C}\{^1\text{H}\}$  NMR (125.7 MHz,  $d_6$ -acetone):  $\delta = 166$  (b, *ipso*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 144$  (b, *ipso*  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 140.5$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 39.5$  Hz),  $\delta = 139.8$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 22.5$  Hz),  $\delta = 138.7$  (s, *ortho*  $\text{Pt}(\text{C}_6\text{H}_5)$ ,  $^1J_{\text{Pt-C}} = 32$  Hz),  $\delta = 135.0$  (m, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 134.7$  (m, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 133.8$  (s, *ortho*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 128.9$  (s, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 128.4$  (s, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 127.9$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 8.5$  Hz),  $\delta = 127.5$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 8.5$  Hz),  $\delta = 127.0$  (s, *meta*  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 126.8$  (s, *meta*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 122.4$  (s, *para*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 120.0$  (s, *para*  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 64.3$  ( $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 23.3$  ( $(\text{CH}_2\text{CH}_2)_2\text{N}$ ),  $\delta = 23$  (b,  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2]$ ),  $\delta = 22$  (b,  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2]$ ),  $\delta = 5.5$  (dd,  $\text{Pt-CH}_3$ ,  $^2J_{\text{P-C(trans)}} = 93$  Hz).  $^{31}\text{P}\{^1\text{H}\}$  NMR (121.4 MHz,  $d_6$ -acetone):  $\delta = 18.3$  (d,  $^1J_{\text{Pt-P}} = 1775$  Hz,  $^2J_{\text{P-P}} = 19$  Hz),  $\delta = 17.29$  (d,  $^1J_{\text{Pt-P}} = 1868$  Hz,  $^2J_{\text{P-P}} = 19$  Hz).  $^{11}\text{B}\{^1\text{H}\}$  NMR (128.3 MHz,  $d_6$ -acetone):  $\delta = -13.8$ . Anal. Calcd. for  $\text{C}_{53}\text{H}_{57}\text{BNP}_2\text{Pt}$ : C, 65.16; H, 5.98; N, 1.43. Found: C, 64.90; H, 6.05; N, 1.54.

**$\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2\text{Pt}(\text{CH}_3)(\text{THF})$  (4).** Solid **2** (49.3 mg, 53.9  $\mu\text{mol}$ ) was dissolved in THF (2 mL). A THF solution (1 mL) of  $[\text{Pr}_2\text{EtNH}][\text{BPh}_4]$  (24.3 mg, 54.1  $\mu\text{mol}$ ) was added to the stirring solution of **2**. The clear, colorless reaction rapidly produced a white precipitate. The mixture was stirred for 15 min, and the white solids ( $\text{ASNBPh}_4$ ) were filtered away. The solution was concentrated in vacuo, and pentane (2 mL) was added, precipitating solid **4** as a spectroscopically pure solid. The solid was collected by filtration. Due to the lability of the coordinated THF molecule, obtaining satisfactory combustion analysis was problematic.

$^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta = 7.64$  (m, 4H, aryl protons),  $\delta = 7.48$  (m, 4H, aryl protons),  $\delta = 7.24$  (m, 4H, aryl protons),  $\delta = 7.00$  (m, 18H, aryl protons),  $\delta = 2.90$  (b, 4H,  $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ),  $\delta = 2.51$  (d, 2H,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ,  $^2J_{\text{P-H}} = 18$  Hz),  $\delta = 2.37$  (d, 2H,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ,  $^2J_{\text{P-H}} = 14$  Hz),  $\delta = 0.71$  (b, 4H,  $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ),  $\delta = 0.35$  (bd, 3H,  $\text{Pt-CH}_3$ ,  $^3J_{\text{P-H}} = 6$  Hz,  $^2J_{\text{Pt-H}} = 40$  Hz).  $^{13}\text{C}\{^1\text{H}\}$  NMR (125.7 MHz, THF,  $-5^\circ\text{C}$ ):  $\delta = 160.3$  (b, *ipso*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 134.1$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 42.1$  Hz),  $\delta = 130.9$  (d, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^2J_{\text{P-C}} = 11.2$  Hz),  $\delta = 130.8$  (d, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^2J_{\text{P-C}} = 10.7$  Hz),  $\delta = 130.1$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 32.6$  Hz),  $\delta = 129.7$  (s, *ortho*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 127.3$  (s, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 126.9$  (s, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 125.5$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 9.1$  Hz),  $\delta = 125.1$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 11.2$  Hz),  $\delta = 123.5$  (s, *meta*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 119.2$  (s, *para*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 64.9$  ( $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ),  $\delta = 22.6$  ( $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ),  $\delta = 21.2$  (b,  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2]$ ),  $\delta = 15.3$  (b,  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2]$ ),  $\delta = 8.2$  (dd,  $\text{Pt-CH}_3$ ,  $^2J_{\text{P-C(trans)}} = 85.5$  Hz,  $^2J_{\text{P-C(cis)}} = 4.8$  Hz).  $^{31}\text{P}\{^1\text{H}\}$  NMR (121.4 MHz, THF):  $\delta = 33.44$  ( $^1J_{\text{Pt-P}} = 1820$  Hz,  $^2J_{\text{P-P}} = 22$  Hz),  $\delta = 15.96$  ( $^1J_{\text{Pt-P}} = 4478$  Hz,  $^2J_{\text{P-P}} = 22$  Hz).  $^{11}\text{B}\{^1\text{H}\}$  NMR (128.3 MHz, THF):  $\delta = -14.5$ .

**Generation of  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2\text{Pt}(\text{C}_6\text{H}_5)(\text{THF})$  (5).** (a) Solid **3** (23.6 mg, 24.2  $\mu\text{mol}$ ) was dissolved in THF (2 mL). Solid  $\text{B}(\text{C}_6\text{F}_5)_3$  (12.5 mg, 24.4  $\mu\text{mol}$ ) was added to the stirring solution of **3**. After 10 min,  $^{31}\text{P}$  NMR analysis showed the formation of one major product, consistent with the formulation of **5** (see (b)).

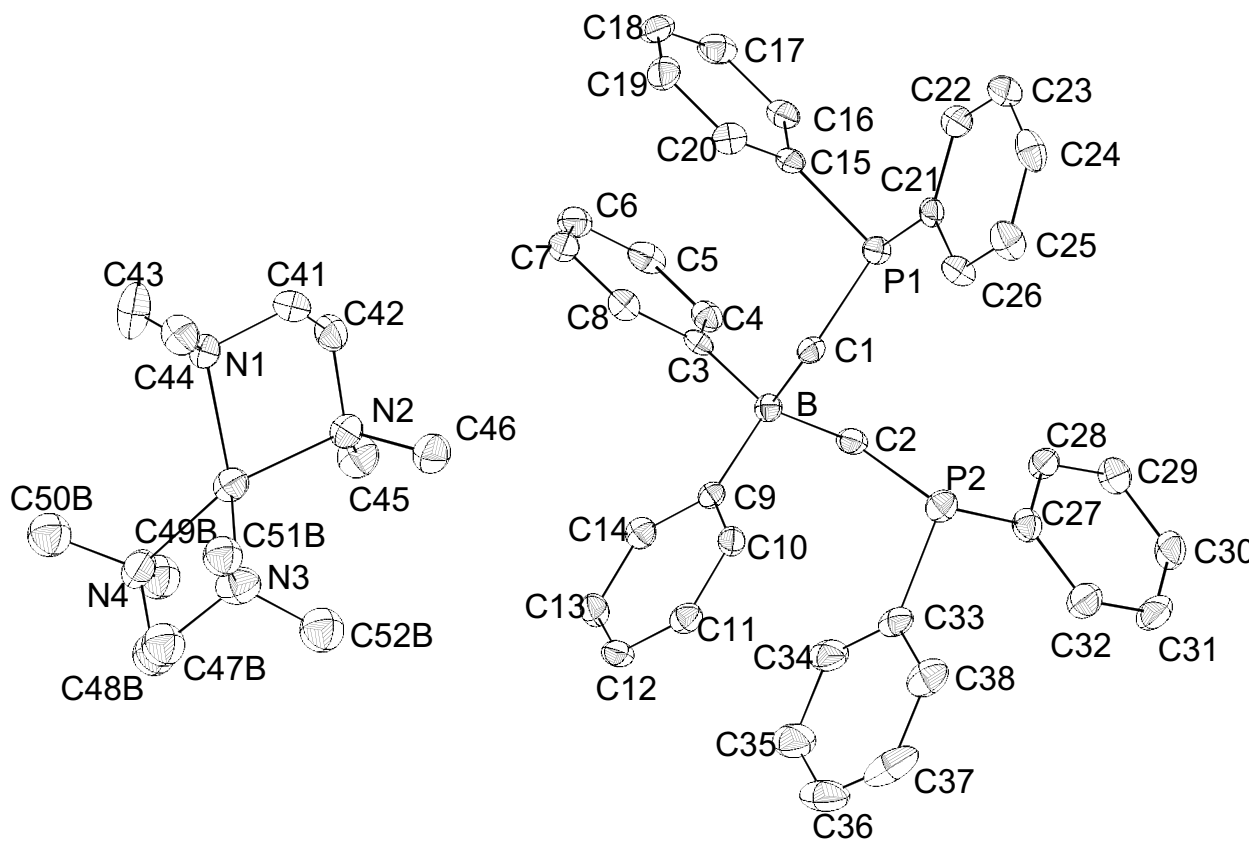
(b) Solid **3** (51.4 mg, 52.6  $\mu\text{mol}$ ) was dissolved in THF (2 mL). A THF solution (2 mL) of  $[\text{Pr}_2\text{EtNH}][\text{BPh}_4]$  (23.5 mg, 52.3  $\mu\text{mol}$ ) was added to the stirring solution of **3**. The clear, colorless reaction slowly produced a white precipitate. The mixture was stirred for 1 h, and the white solids were filtered away. The solution was concentrated in vacuo, and pentane (2 mL) was added, precipitating white solids. The solids were collected by filtration. NMR analysis of the solids was consistent with the formulation of **5** as the major product (~80%). Due to the lability of the coordinated THF molecule, obtaining satisfactory combustion analysis was problematic.

$^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta = 7.56$  (m, 4H, aryl protons),  $\delta = 7.46$  (m, 4H, aryl protons),  $\delta = 7.21$  (m, 4H, aryl protons),  $\delta = 6.95$  (m, 18H, aryl protons),  $\delta = 6.88$  (m, 2H,  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 6.78$  (m, 2H,  $\text{Pt-C}_6\text{H}_5$ ),  $\delta = 6.73$  (m, 1H, *para*  $\text{Pt-C}_6\text{H}_5$ ),  $\delta = 2.87$  (b, 4H,  $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ),  $\delta = 2.64$  (d, 2H,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ,  $^2J_{\text{P-H}} = 17$  Hz),  $\delta = 2.42$  (d, 2H,  $\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2$ ,  $^2J_{\text{P-H}} = 14$  Hz),  $\delta = 0.46$  (b, 4H,  $\text{Pt-O}(\text{CH}_2\text{CH}_2)_2$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR (125.7 MHz, THF):  $\delta = 161$  (b, *ipso*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 136.3$  (s, *ortho*  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 135.4$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 43.8$  Hz),  $\delta = 133.0$  (d, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^2J_{\text{P-C}} = 10.7$  Hz),  $\delta = 132.9$  (d, *ortho*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^2J_{\text{P-C}} = 11.2$  Hz),  $\delta = 132.1$  (d, *ipso*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^1J_{\text{P-C}} = 57.7$  Hz),  $\delta = 131.5$  (s, *ortho*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 129.2$  (s, *meta*

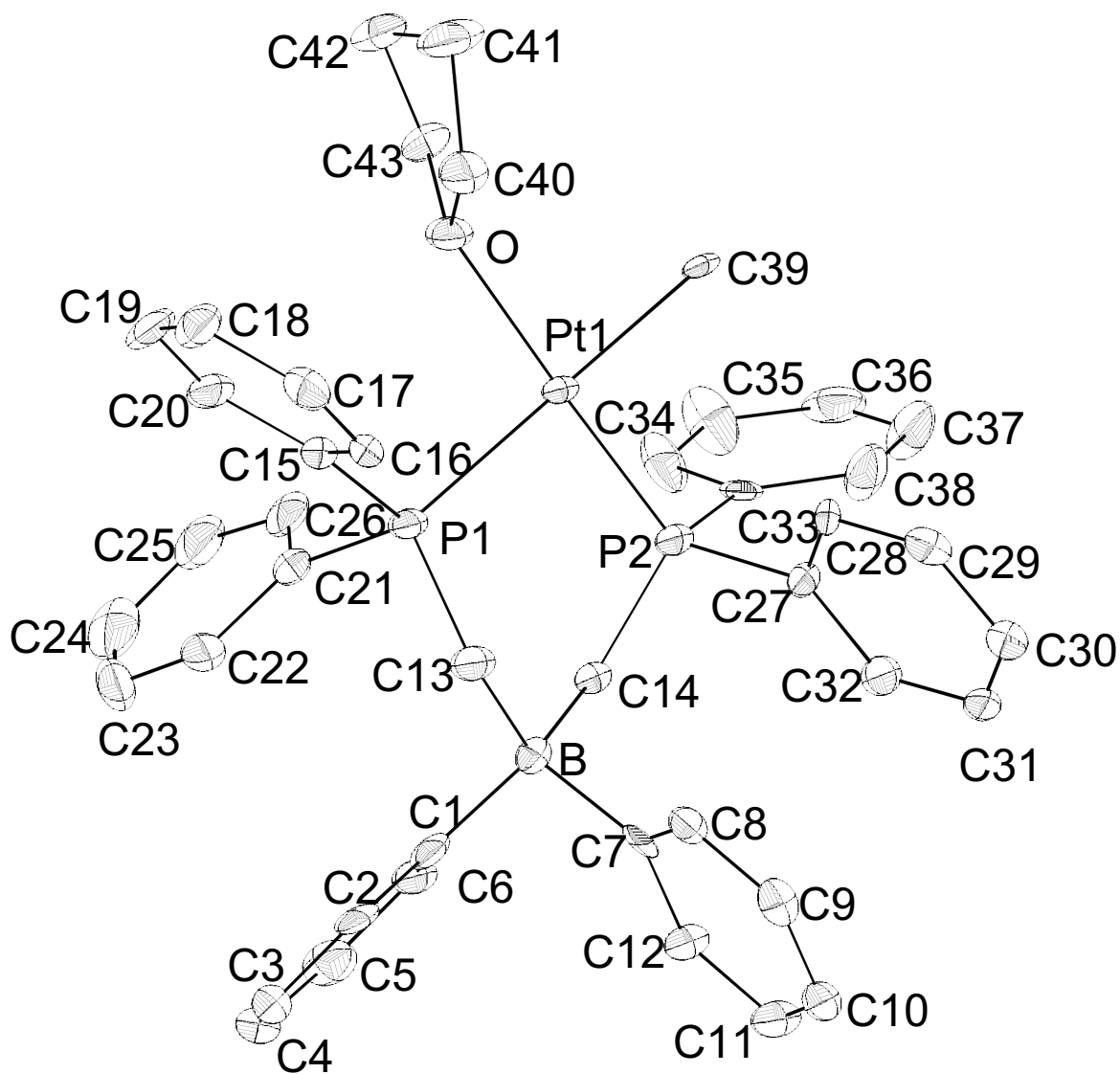
$\text{P}(\text{C}_6\text{H}_5)_2$ ,  $\delta = 129.1$  (s, *meta*  $\text{P}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 127.6$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 9.1$  Hz),  $\delta = 126.9$  (d, *para*  $\text{P}(\text{C}_6\text{H}_5)_2$ ,  $^4J_{\text{P-C}} = 11.2$  Hz),  $\delta = 126.4$  (d, *meta*  $\text{Pt}(\text{C}_6\text{H}_5)$ ,  $^4J_{\text{P-C}} = 9.6$  Hz),  $\delta = 125.5$  (s, *meta*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 122.6$  (s, *para*  $\text{Pt}(\text{C}_6\text{H}_5)$ ),  $\delta = 121.2$  (s, *para*  $\text{B}(\text{C}_6\text{H}_5)_2$ ),  $\delta = 67$  ( $\text{Pt-O}(\underline{\text{C}}\text{H}_2\text{C}\underline{\text{H}}_2)_2$ ),  $\delta = 26$  ( $\text{Pt-O}(\underline{\text{C}}\text{H}_2\text{C}\underline{\text{H}}_2)_2$ ),  $\delta = 21$  (b,  $[\text{Ph}_2\text{B}(\underline{\text{C}}\text{H}_2\text{PPh}_2)_2]$ ),  $\delta = 17$  (b,  $[\text{Ph}_2\text{B}(\underline{\text{C}}\text{H}_2\text{PPh}_2)_2]$ ).  $^{31}\text{P}\{^1\text{H}\}$  NMR (121.4 MHz, THF):  $\delta = 28.60$  ( $^1J_{\text{Pt-P}} = 1740$  Hz,  $^2J_{\text{P-P}} = 23$  Hz),  $\delta = 11.20$  ( $^1J_{\text{Pt-P}} = 4393$  Hz,  $^2J_{\text{P-P}} = 23$  Hz).  $^{11}\text{B}\{^1\text{H}\}$  NMR (128.3 MHz, THF):  $\delta = -14.9$ .

**Reaction of 4 with benzene.** Solid **4** was generated as above and dissolved in benzene (2 mL). The solution was concentrated *in vacuo* (vol  $\approx 0.5$  mL). Benzene was added (1 mL), and the solution was again concentrated *in vacuo* (vol  $\approx 0.7$  mL). Concentration of the benzene solution serves to ensure complete removal of residual pentane. The solution was placed in an NMR tube, and an initial  $^{31}\text{P}$  NMR spectrum showed the presence of one species (**4**). The NMR tube was heated to 50 °C and monitored by  $^{31}\text{P}$  NMR spectroscopy. After 4 h,  $^{31}\text{P}$  NMR spectroscopy showed the presence of 4 products, where **5** was the major product (ca. 80%). The identities of the three minor byproducts have yet to be confirmed. They may result from (i) ligand activation, (ii) THF activation, or (iii) bimolecular reaction pathways.

**Figure 1.** Fully labeled drawing of  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2][\text{Li}(\text{TMEDA})_2]$ . Hydrogens have been omitted for clarity. One TMEDA molecule coordinated to lithium was found to be disordered over two positions: only one position is shown in the figure below.



**Figure 2.** Fully labeled drawing of **4**. Hydrogens and solvent THF molecules have been omitted for clarity.



**Table 1. Crystal data and structure refinement for [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][Li(TMEDA)<sub>2</sub>].**

Empirical formula	C <sub>50</sub> H <sub>65</sub> BLiN <sub>4</sub> P <sub>2</sub>	
Formula weight	801.75	
Crystal Habit	Prisms	
Crystal size	0.41 x 0.34 x 0.16 mm <sup>3</sup>	
Crystal color	Colorless	
<b>Data Collection</b>		
Type of diffractometer	CCD area detector	
Wavelength	0.71073 Å MoK $\alpha$	
Data Collection Temperature	98 K	
Unit cell dimensions	a = 11.7922(6) Å b = 11.7081(6) Å c = 33.1336(18) Å	$\alpha$ = 90° $\beta$ = 94.0620(10)° $\gamma$ = 90°
Volume	4563.1(4) Å <sup>3</sup>	
Z	4	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Density (calculated)	1.167 Mg/m <sup>3</sup>	
F(000)	1724	
$\theta$ range for data collection	1.73 to 28.39°	
Completeness to $\theta$ = 28.39°	93.3 %	
Index ranges	-15 ≤ h ≤ 15, -15 ≤ k ≤ 15, -44 ≤ l ≤ 43	
Data collection scan type	omega scans at 5 $\phi$ settings	
Reflections collected	65068	
Independent reflections	10687 [R <sub>int</sub> = 0.0648]	
Absorption coefficient	0.133 mm <sup>-1</sup>	
Absorption correction	None	
Max. and min. transmission	0.9786 and 0.9477	

**Structure solution and Refinement**

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	10687 / 0 / 714

Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on $F^2$	1.952
Final R indices [ $I > 2\sigma(I)$ ]	R1 = 0.0608, wR2 = 0.0862
R indices (all data)	R1 = 0.0882, wR2 = 0.0879
Largest diff. peak and hole	1.130 and -0.580 e. $\text{\AA}^{-3}$



**Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2][\text{Li}(\text{TMEDA})_2]$ .  $U(\text{eq})$  is defined as the trace of the orthogonalized  $U^{ij}$  tensor.**

	x	y	z		$U_{\text{eq}}$
P(1)	11110(1)	6033(1)	1606(1)	19(1)	1
P(2)	9278(1)	7541(1)	688(1)	23(1)	1
B	8947(2)	5458(2)	1165(1)	17(1)	1
C(1)	10337(2)	5271(2)	1191(1)	19(1)	1
C(2)	8661(2)	6842(2)	1114(1)	22(1)	1
C(3)	8455(2)	4973(2)	1581(1)	18(1)	1
C(4)	7890(2)	5610(2)	1862(1)	22(1)	1
C(5)	7531(2)	5144(2)	2217(1)	26(1)	1
C(6)	7727(2)	4012(2)	2308(1)	27(1)	1
C(7)	8281(2)	3345(2)	2037(1)	26(1)	1
C(8)	8622(2)	3820(2)	1687(1)	21(1)	1
C(9)	8348(2)	4741(2)	782(1)	17(1)	1
C(10)	8873(2)	4503(2)	425(1)	20(1)	1
C(11)	8327(2)	3958(2)	95(1)	23(1)	1
C(12)	7216(2)	3613(2)	107(1)	25(1)	1
C(13)	6661(2)	3818(2)	455(1)	25(1)	1
C(14)	7225(2)	4363(2)	781(1)	22(1)	1
C(15)	11059(2)	5044(2)	2038(1)	19(1)	1
C(16)	10612(2)	5422(2)	2389(1)	23(1)	1
C(17)	10512(2)	4700(2)	2716(1)	28(1)	1
C(18)	10860(2)	3584(2)	2696(1)	29(1)	1
C(19)	11325(2)	3191(2)	2351(1)	27(1)	1
C(20)	11421(2)	3913(2)	2026(1)	23(1)	1
C(21)	12617(2)	5878(2)	1506(1)	18(1)	1
C(22)	13449(2)	5947(2)	1827(1)	25(1)	1
C(23)	14593(2)	5932(2)	1764(1)	29(1)	1
C(24)	14949(2)	5867(2)	1378(1)	28(1)	1
C(25)	14147(2)	5812(2)	1056(1)	30(1)	1
C(26)	12996(2)	5817(2)	1120(1)	25(1)	1
C(27)	8910(2)	9065(2)	739(1)	23(1)	1
C(28)	8823(2)	9592(2)	1107(1)	23(1)	1
C(29)	8657(2)	10761(2)	1136(1)	26(1)	1
C(30)	8563(2)	11433(2)	795(1)	29(1)	1
C(31)	8640(2)	10926(2)	424(1)	37(1)	1
C(32)	8821(2)	9763(2)	393(1)	33(1)	1
C(33)	8301(2)	7182(2)	247(1)	24(1)	1
C(34)	7173(2)	6883(2)	276(1)	31(1)	1
C(35)	6493(2)	6575(2)	-59(1)	41(1)	1
C(36)	6909(3)	6550(2)	-431(1)	46(1)	1
C(37)	8021(3)	6839(2)	-476(1)	46(1)	1
C(38)	8729(2)	7165(2)	-131(1)	34(1)	1
Li	4435(3)	316(3)	1368(1)	28(1)	1
N(1)	5540(1)	-468(1)	1820(1)	23(1)	1
N(2)	4904(1)	1916(1)	1644(1)	25(1)	1
N(3)	4686(2)	-98(2)	767(1)	37(1)	1
N(4)	2685(2)	-76(2)	1245(1)	37(1)	1
C(41)	6222(2)	515(2)	1976(1)	28(1)	1
C(42)	5505(2)	1557(2)	2029(1)	30(1)	1

C(43)	4877(2)	-927(2)	2140(1)	43(1)	1
C(44)	6322(2)	-1372(2)	1707(1)	33(1)	1
C(45)	3979(2)	2696(2)	1730(1)	38(1)	1
C(46)	5692(2)	2560(2)	1405(1)	36(1)	1
C(47A)	3547(5)	-59(7)	559(2)	51(2)	0.445(6)
C(48A)	2787(5)	-741(6)	809(2)	40(2)	0.445(6)
C(49A)	1975(5)	842(5)	1180(2)	47(2)	0.445(6)
C(50A)	2113(5)	-936(5)	1511(2)	30(2)	0.445(6)
C(51A)	5197(6)	-1183(5)	711(2)	37(2)	0.445(6)
C(52A)	5387(6)	844(4)	554(2)	33(2)	0.445(6)
C(47B)	3576(4)	-616(5)	643(2)	42(1)	0.555(6)
C(48B)	2562(4)	-33(5)	821(1)	42(2)	0.555(6)
C(49B)	1850(4)	817(4)	1410(2)	44(1)	0.555(6)
C(50B)	2453(5)	-1161(4)	1412(2)	43(1)	0.555(6)
C(51B)	5624(4)	-936(4)	695(1)	36(1)	0.555(6)
C(52B)	4837(5)	924(4)	555(1)	45(1)	0.555(6)

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**Table 3. Bond lengths [Å] and angles [°] for [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>][Li(TMEDA)<sub>2</sub>].**

P(1)-C(1)	1.829(2)	C(23)-H(23)	0.956(18)
P(1)-C(21)	1.8401(19)	C(24)-C(25)	1.376(3)
P(1)-C(15)	1.8438(19)	C(24)-H(24)	0.941(18)
P(2)-C(2)	1.825(2)	C(25)-C(26)	1.389(3)
P(2)-C(33)	1.844(2)	C(25)-H(25)	0.907(18)
P(2)-C(27)	1.847(2)	C(26)-H(26)	0.901(16)
B-C(3)	1.636(3)	C(27)-C(28)	1.379(3)
B-C(9)	1.639(3)	C(27)-C(32)	1.404(3)
B-C(1)	1.650(3)	C(28)-C(29)	1.386(3)
B-C(2)	1.662(3)	C(28)-H(28)	0.921(17)
C(1)-H(1A)	0.924(16)	C(29)-C(30)	1.374(3)
C(1)-H(1B)	0.999(17)	C(29)-H(29)	0.927(17)
C(2)-H(2A)	1.14(2)	C(30)-C(31)	1.373(3)
C(2)-H(2B)	0.947(17)	C(30)-H(30)	0.925(17)
C(3)-C(4)	1.397(3)	C(31)-C(32)	1.383(3)
C(3)-C(8)	1.405(3)	C(31)-H(31)	0.986(19)
C(4)-C(5)	1.389(3)	C(32)-H(32)	0.932(19)
C(4)-H(4)	0.925(15)	C(33)-C(38)	1.384(3)
C(5)-C(6)	1.375(3)	C(33)-C(34)	1.385(3)
C(5)-H(5)	0.964(17)	C(34)-C(35)	1.369(3)
C(6)-C(7)	1.385(3)	C(34)-H(34)	1.001(19)
C(6)-H(6)	0.960(18)	C(35)-C(36)	1.360(3)
C(7)-C(8)	1.375(3)	C(35)-H(35)	1.00(2)
C(7)-H(7)	0.981(18)	C(36)-C(37)	1.373(4)
C(8)-H(8)	0.914(16)	C(36)-H(36)	0.97(2)
C(9)-C(14)	1.396(3)	C(37)-C(38)	1.420(3)
C(9)-C(10)	1.400(3)	C(37)-H(37)	0.85(2)
C(10)-C(11)	1.384(3)	C(38)-H(38)	0.913(17)
C(10)-H(10)	0.894(15)	Li-N(3)	2.091(4)
C(11)-C(12)	1.374(3)	Li-N(1)	2.122(4)
C(11)-H(11)	0.922(16)	Li-N(4)	2.126(4)
C(12)-C(13)	1.385(3)	Li-N(2)	2.140(4)
C(12)-H(12)	0.940(17)	Li-C(47B)	2.766(6)
C(13)-C(14)	1.384(3)	Li-C(48B)	2.787(6)
C(13)-H(13)	0.952(17)	N(1)-C(43)	1.463(3)
C(14)-H(14)	0.962(15)	N(1)-C(44)	1.470(3)
C(15)-C(16)	1.384(3)	N(1)-C(41)	1.477(2)
C(15)-C(20)	1.393(3)	N(2)-C(45)	1.466(3)
C(16)-C(17)	1.384(3)	N(2)-C(46)	1.471(3)
C(16)-H(16)	0.942(16)	N(2)-C(42)	1.474(3)
C(17)-C(18)	1.372(3)	N(3)-C(52B)	1.404(5)
C(17)-H(17)	0.951(16)	N(3)-C(51A)	1.424(6)
C(18)-C(19)	1.380(3)	N(3)-C(47A)	1.467(6)
C(18)-H(18)	0.908(17)	N(3)-C(47B)	1.475(5)
C(19)-C(20)	1.381(3)	N(3)-C(51B)	1.510(5)
C(19)-H(19)	0.938(19)	N(3)-C(52A)	1.574(5)
C(20)-H(20)	0.924(16)	N(4)-C(49A)	1.370(5)
C(21)-C(26)	1.386(3)	N(4)-C(48B)	1.404(4)
C(21)-C(22)	1.396(3)	N(4)-C(50B)	1.420(5)
C(22)-C(23)	1.379(3)	N(4)-C(50A)	1.526(5)
C(22)-H(22)	0.941(17)	N(4)-C(49B)	1.561(5)
C(23)-C(24)	1.375(3)	N(4)-C(48A)	1.651(6)

C(41)-C(42)	1.502(3)	C(2)-P(2)-C(33)	104.45(10)
C(41)-H(41A)	0.993(18)	C(2)-P(2)-C(27)	104.73(9)
C(41)-H(41B)	0.982(19)	C(33)-P(2)-C(27)	98.88(9)
C(42)-H(42A)	1.026(18)	C(3)-B-C(9)	108.43(15)
C(42)-H(42B)	0.988(18)	C(3)-B-C(1)	108.70(16)
C(43)-H(43A)	0.834(18)	C(9)-B-C(1)	110.14(15)
C(43)-H(43B)	0.907(17)	C(3)-B-C(2)	110.01(16)
C(44)-H(44A)	1.01(2)	C(9)-B-C(2)	110.34(16)
C(44)-H(44B)	0.961(18)	C(1)-B-C(2)	109.18(16)
C(44)-H(44C)	1.00(2)	B-C(1)-P(1)	114.46(13)
C(45)-H(45A)	1.01(2)	B-C(1)-H(1A)	109.4(11)
C(45)-H(45B)	1.02(2)	P(1)-C(1)-H(1A)	104.4(11)
C(45)-H(45C)	0.98(2)	B-C(1)-H(1B)	111.8(10)
C(46)-H(46A)	0.981(19)	P(1)-C(1)-H(1B)	108.9(10)
C(46)-H(46B)	0.96(2)	H(1A)-C(1)-H(1B)	107.4(14)
C(46)-H(46C)	1.05(2)	B-C(2)-P(2)	115.25(14)
C(47A)-C(48A)	1.496(9)	B-C(2)-H(2A)	123.9(10)
C(47A)-H(47A)	0.9900	P(2)-C(2)-H(2A)	91.4(10)
C(47A)-H(47B)	0.9900	B-C(2)-H(2B)	107.3(10)
C(48A)-H(48A)	0.9900	P(2)-C(2)-H(2B)	101.8(10)
C(48A)-H(48B)	0.9900	H(2A)-C(2)-H(2B)	114.4(15)
C(49A)-H(49A)	0.9800	C(4)-C(3)-C(8)	114.38(18)
C(49A)-H(49B)	0.9800	C(4)-C(3)-B	126.21(17)
C(49A)-H(49C)	0.9800	C(8)-C(3)-B	119.38(17)
C(50A)-H(50A)	0.9800	C(5)-C(4)-C(3)	122.8(2)
C(50A)-H(50B)	0.9800	C(5)-C(4)-H(4)	118.6(10)
C(50A)-H(50C)	0.9800	C(3)-C(4)-H(4)	118.6(10)
C(51A)-H(51A)	0.9800	C(6)-C(5)-C(4)	120.5(2)
C(51A)-H(51B)	0.9800	C(6)-C(5)-H(5)	120.1(10)
C(51A)-H(51C)	0.9800	C(4)-C(5)-H(5)	119.4(11)
C(52A)-H(52A)	0.9800	C(5)-C(6)-C(7)	118.7(2)
C(52A)-H(52B)	0.9800	C(5)-C(6)-H(6)	120.6(11)
C(52A)-H(52C)	0.9800	C(7)-C(6)-H(6)	120.6(11)
C(47B)-C(48B)	1.532(7)	C(8)-C(7)-C(6)	119.9(2)
C(47B)-H(47C)	0.9900	C(8)-C(7)-H(7)	121.0(11)
C(47B)-H(47D)	0.9900	C(6)-C(7)-H(7)	119.1(11)
C(48B)-H(48C)	0.9900	C(7)-C(8)-C(3)	123.7(2)
C(48B)-H(48D)	0.9900	C(7)-C(8)-H(8)	119.8(10)
C(49B)-H(49D)	0.9800	C(3)-C(8)-H(8)	116.6(10)
C(49B)-H(49E)	0.9800	C(14)-C(9)-C(10)	114.32(18)
C(49B)-H(49F)	0.9800	C(14)-C(9)-B	121.37(17)
C(50B)-H(50D)	0.9800	C(10)-C(9)-B	124.24(17)
C(50B)-H(50E)	0.9800	C(11)-C(10)-C(9)	123.2(2)
C(50B)-H(50F)	0.9800	C(11)-C(10)-H(10)	118.1(10)
C(51B)-H(51D)	0.9800	C(9)-C(10)-H(10)	118.6(10)
C(51B)-H(51E)	0.9800	C(12)-C(11)-C(10)	120.3(2)
C(51B)-H(51F)	0.9800	C(12)-C(11)-H(11)	119.7(10)
C(52B)-H(52D)	0.9800	C(10)-C(11)-H(11)	120.0(11)
C(52B)-H(52E)	0.9800	C(11)-C(12)-C(13)	118.7(2)
C(52B)-H(52F)	0.9800	C(11)-C(12)-H(12)	119.8(11)
		C(13)-C(12)-H(12)	121.4(11)
C(1)-P(1)-C(21)	104.59(9)	C(14)-C(13)-C(12)	119.9(2)
C(1)-P(1)-C(15)	103.67(9)	C(14)-C(13)-H(13)	120.7(10)
C(21)-P(1)-C(15)	99.35(8)	C(12)-C(13)-H(13)	119.3(10)

C(13)-C(14)-C(9)	123.47(19)	C(31)-C(32)-C(27)	121.0(2)
C(13)-C(14)-H(14)	117.9(10)	C(31)-C(32)-H(32)	118.9(12)
C(9)-C(14)-H(14)	118.6(10)	C(27)-C(32)-H(32)	120.0(12)
C(16)-C(15)-C(20)	117.60(18)	C(38)-C(33)-C(34)	118.1(2)
C(16)-C(15)-P(1)	119.14(15)	C(38)-C(33)-P(2)	118.29(18)
C(20)-C(15)-P(1)	123.23(15)	C(34)-C(33)-P(2)	123.54(16)
C(17)-C(16)-C(15)	121.4(2)	C(35)-C(34)-C(33)	121.4(2)
C(17)-C(16)-H(16)	120.5(10)	C(35)-C(34)-H(34)	118.2(12)
C(15)-C(16)-H(16)	118.2(10)	C(33)-C(34)-H(34)	120.4(12)
C(18)-C(17)-C(16)	120.2(2)	C(36)-C(35)-C(34)	120.8(3)
C(18)-C(17)-H(17)	118.5(10)	C(36)-C(35)-H(35)	123.8(14)
C(16)-C(17)-H(17)	121.3(10)	C(34)-C(35)-H(35)	115.4(14)
C(17)-C(18)-C(19)	119.6(2)	C(35)-C(36)-C(37)	120.2(3)
C(17)-C(18)-H(18)	122.4(12)	C(35)-C(36)-H(36)	120.9(14)
C(19)-C(18)-H(18)	118.0(12)	C(37)-C(36)-H(36)	118.8(14)
C(18)-C(19)-C(20)	120.1(2)	C(36)-C(37)-C(38)	119.3(3)
C(18)-C(19)-H(19)	121.0(12)	C(36)-C(37)-H(37)	125.4(16)
C(20)-C(19)-H(19)	118.9(12)	C(38)-C(37)-H(37)	115.3(16)
C(19)-C(20)-C(15)	121.2(2)	C(33)-C(38)-C(37)	120.2(3)
C(19)-C(20)-H(20)	118.9(11)	C(33)-C(38)-H(38)	117.5(12)
C(15)-C(20)-H(20)	119.9(11)	C(37)-C(38)-H(38)	122.3(12)
C(26)-C(21)-C(22)	116.73(19)	N(3)-Li-N(1)	116.79(17)
C(26)-C(21)-P(1)	123.35(15)	N(3)-Li-N(4)	88.19(15)
C(22)-C(21)-P(1)	119.55(15)	N(1)-Li-N(4)	125.50(17)
C(23)-C(22)-C(21)	121.8(2)	N(3)-Li-N(2)	124.06(17)
C(23)-C(22)-H(22)	120.8(11)	N(1)-Li-N(2)	87.14(14)
C(21)-C(22)-H(22)	117.3(11)	N(4)-Li-N(2)	119.27(17)
C(24)-C(23)-C(22)	120.5(2)	N(3)-Li-C(47B)	31.65(12)
C(24)-C(23)-H(23)	118.4(11)	N(1)-Li-C(47B)	127.66(19)
C(22)-C(23)-H(23)	121.1(11)	N(4)-Li-C(47B)	57.13(14)
C(25)-C(24)-C(23)	119.0(2)	N(2)-Li-C(47B)	141.7(2)
C(25)-C(24)-H(24)	122.0(11)	N(3)-Li-C(48B)	60.36(13)
C(23)-C(24)-H(24)	118.8(11)	N(1)-Li-C(48B)	144.50(19)
C(24)-C(25)-C(26)	120.4(2)	N(4)-Li-C(48B)	29.48(10)
C(24)-C(25)-H(25)	118.5(13)	N(2)-Li-C(48B)	125.11(19)
C(26)-C(25)-H(25)	121.1(13)	C(47B)-Li-C(48B)	32.02(15)
C(21)-C(26)-C(25)	121.6(2)	C(43)-N(1)-C(44)	107.32(18)
C(21)-C(26)-H(26)	121.5(11)	C(43)-N(1)-C(41)	109.73(18)
C(25)-C(26)-H(26)	116.9(11)	C(44)-N(1)-C(41)	108.29(17)
C(28)-C(27)-C(32)	117.11(19)	C(43)-N(1)-Li	109.75(17)
C(28)-C(27)-P(2)	123.04(16)	C(44)-N(1)-Li	119.77(17)
C(32)-C(27)-P(2)	119.54(16)	C(41)-N(1)-Li	101.65(15)
C(27)-C(28)-C(29)	121.4(2)	C(45)-N(2)-C(46)	107.02(19)
C(27)-C(28)-H(28)	120.2(11)	C(45)-N(2)-C(42)	109.16(17)
C(29)-C(28)-H(28)	118.4(11)	C(46)-N(2)-C(42)	109.18(18)
C(30)-C(29)-C(28)	120.9(2)	C(45)-N(2)-Li	117.09(17)
C(30)-C(29)-H(29)	120.5(11)	C(46)-N(2)-Li	111.83(17)
C(28)-C(29)-H(29)	118.6(11)	C(42)-N(2)-Li	102.33(15)
C(31)-C(30)-C(29)	118.7(2)	C(52B)-N(3)-C(51A)	128.7(3)
C(31)-C(30)-H(30)	120.9(11)	C(52B)-N(3)-C(47A)	83.3(3)
C(29)-C(30)-H(30)	120.4(11)	C(51A)-N(3)-C(47A)	110.4(4)
C(30)-C(31)-C(32)	120.8(2)	C(52B)-N(3)-C(47B)	110.7(3)
C(30)-C(31)-H(31)	121.9(11)	C(51A)-N(3)-C(47B)	88.3(3)
C(32)-C(31)-H(31)	117.3(11)	C(47A)-N(3)-C(47B)	27.9(3)

C(52B)-N(3)-C(51B)	110.8(3)	N(1)-C(44)-H(44C)	110.9(11)
C(51A)-N(3)-C(51B)	22.8(3)	H(44A)-C(44)-H(44C)	113.3(16)
C(47A)-N(3)-C(51B)	127.1(3)	H(44B)-C(44)-H(44C)	107.4(16)
C(47B)-N(3)-C(51B)	109.5(3)	N(2)-C(45)-H(45A)	108.2(11)
C(52B)-N(3)-C(52A)	24.6(3)	N(2)-C(45)-H(45B)	110.1(11)
C(51A)-N(3)-C(52A)	109.0(3)	H(45A)-C(45)-H(45B)	109.2(16)
C(47A)-N(3)-C(52A)	105.1(3)	N(2)-C(45)-H(45C)	111.2(12)
C(47B)-N(3)-C(52A)	130.4(3)	H(45A)-C(45)-H(45C)	109.6(16)
C(51B)-N(3)-C(52A)	88.3(3)	H(45B)-C(45)-H(45C)	108.6(16)
C(52B)-N(3)-Li	108.1(2)	N(2)-C(46)-H(46A)	109.2(11)
C(51A)-N(3)-Li	114.9(3)	N(2)-C(46)-H(46B)	110.6(12)
C(47A)-N(3)-Li	104.7(3)	H(46A)-C(46)-H(46B)	101.6(16)
C(47B)-N(3)-Li	100.3(2)	N(2)-C(46)-H(46C)	111.2(12)
C(51B)-N(3)-Li	117.1(2)	H(46A)-C(46)-H(46C)	113.6(16)
C(52A)-N(3)-Li	112.2(2)	H(46B)-C(46)-H(46C)	110.3(17)
C(49A)-N(4)-C(48B)	78.2(3)	N(3)-C(47A)-C(48A)	106.7(5)
C(49A)-N(4)-C(50B)	129.1(3)	N(3)-C(47A)-H(47A)	110.4
C(48B)-N(4)-C(50B)	114.5(3)	C(48A)-C(47A)-H(47A)	110.4
C(49A)-N(4)-C(50A)	108.6(3)	N(3)-C(47A)-H(47B)	110.4
C(48B)-N(4)-C(50A)	125.7(3)	C(48A)-C(47A)-H(47B)	110.4
C(50B)-N(4)-C(50A)	23.0(3)	H(47A)-C(47A)-H(47B)	108.6
C(49A)-N(4)-C(49B)	30.3(3)	C(47A)-C(48A)-N(4)	108.5(5)
C(48B)-N(4)-C(49B)	107.7(3)	C(47A)-C(48A)-H(48A)	110.0
C(50B)-N(4)-C(49B)	108.6(3)	N(4)-C(48A)-H(48A)	110.0
C(50A)-N(4)-C(49B)	85.7(3)	C(47A)-C(48A)-H(48B)	110.0
C(49A)-N(4)-C(48A)	108.3(3)	N(4)-C(48A)-H(48B)	110.0
C(48B)-N(4)-C(48A)	31.9(2)	H(48A)-C(48A)-H(48B)	108.4
C(50B)-N(4)-C(48A)	87.0(3)	N(4)-C(49A)-H(49A)	109.5
C(50A)-N(4)-C(48A)	104.8(3)	N(4)-C(49A)-H(49B)	109.5
C(49B)-N(4)-C(48A)	135.1(3)	H(49A)-C(49A)-H(49B)	109.5
C(49A)-N(4)-Li	115.8(3)	N(4)-C(49A)-H(49C)	109.5
C(48B)-N(4)-Li	102.4(2)	H(49A)-C(49A)-H(49C)	109.5
C(50B)-N(4)-Li	109.2(2)	H(49B)-C(49A)-H(49C)	109.5
C(50A)-N(4)-Li	119.7(2)	N(4)-C(50A)-H(50A)	109.5
C(49B)-N(4)-Li	114.6(2)	N(4)-C(50A)-H(50B)	109.5
C(48A)-N(4)-Li	98.0(2)	H(50A)-C(50A)-H(50B)	109.5
N(1)-C(41)-C(42)	112.13(18)	N(4)-C(50A)-H(50C)	109.5
N(1)-C(41)-H(41A)	111.2(10)	H(50A)-C(50A)-H(50C)	109.5
C(42)-C(41)-H(41A)	110.0(10)	H(50B)-C(50A)-H(50C)	109.5
N(1)-C(41)-H(41B)	108.3(11)	N(3)-C(51A)-H(51A)	109.5
C(42)-C(41)-H(41B)	107.2(11)	N(3)-C(51A)-H(51B)	109.5
H(41A)-C(41)-H(41B)	107.7(15)	H(51A)-C(51A)-H(51B)	109.5
N(2)-C(42)-C(41)	111.82(18)	N(3)-C(51A)-H(51C)	109.5
N(2)-C(42)-H(42A)	108.6(10)	H(51A)-C(51A)-H(51C)	109.5
C(41)-C(42)-H(42A)	108.4(10)	H(51B)-C(51A)-H(51C)	109.5
N(2)-C(42)-H(42B)	111.4(11)	N(3)-C(52A)-H(52A)	109.5
C(41)-C(42)-H(42B)	107.8(10)	N(3)-C(52A)-H(52B)	109.5
H(42A)-C(42)-H(42B)	108.7(15)	H(52A)-C(52A)-H(52B)	109.5
N(1)-C(43)-H(43A)	109.1(13)	N(3)-C(52A)-H(52C)	109.5
N(1)-C(43)-H(43B)	106.8(11)	H(52A)-C(52A)-H(52C)	109.5
H(43A)-C(43)-H(43B)	110.7(18)	H(52B)-C(52A)-H(52C)	109.5
N(1)-C(44)-H(44A)	109.0(11)	N(3)-C(47B)-C(48B)	114.2(4)
N(1)-C(44)-H(44B)	109.7(11)	N(3)-C(47B)-Li	48.05(17)
H(44A)-C(44)-H(44B)	106.4(16)	C(48B)-C(47B)-Li	74.8(2)

N(3)-C(47B)-H(47C)	108.7	H(49D)-C(49B)-H(49F)	109.5
C(48B)-C(47B)-H(47C)	108.7	H(49E)-C(49B)-H(49F)	109.5
Li-C(47B)-H(47C)	98.0	N(4)-C(50B)-H(50D)	109.5
N(3)-C(47B)-H(47D)	108.7	N(4)-C(50B)-H(50E)	109.5
C(48B)-C(47B)-H(47D)	108.7	H(50D)-C(50B)-H(50E)	109.5
Li-C(47B)-H(47D)	150.8	N(4)-C(50B)-H(50F)	109.5
H(47C)-C(47B)-H(47D)	107.6	H(50D)-C(50B)-H(50F)	109.5
N(4)-C(48B)-C(47B)	110.0(4)	H(50E)-C(50B)-H(50F)	109.5
N(4)-C(48B)-Li	48.16(17)	N(3)-C(51B)-H(51D)	109.5
C(47B)-C(48B)-Li	73.2(2)	N(3)-C(51B)-H(51E)	109.5
N(4)-C(48B)-H(48C)	109.7	H(51D)-C(51B)-H(51E)	109.5
C(47B)-C(48B)-H(48C)	109.7	N(3)-C(51B)-H(51F)	109.5
Li-C(48B)-H(48C)	94.7	H(51D)-C(51B)-H(51F)	109.5
N(4)-C(48B)-H(48D)	109.7	H(51E)-C(51B)-H(51F)	109.5
C(47B)-C(48B)-H(48D)	109.7	N(3)-C(52B)-H(52D)	109.5
Li-C(48B)-H(48D)	153.6	N(3)-C(52B)-H(52E)	109.5
H(48C)-C(48B)-H(48D)	108.2	H(52D)-C(52B)-H(52E)	109.5
N(4)-C(49B)-H(49D)	109.5	N(3)-C(52B)-H(52F)	109.5
N(4)-C(49B)-H(49E)	109.5	H(52D)-C(52B)-H(52F)	109.5
H(49D)-C(49B)-H(49E)	109.5	H(52E)-C(52B)-H(52F)	109.5
N(4)-C(49B)-H(49F)	109.5		

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**Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ ) for  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2][\text{Li}(\text{TMEDA})_2]$ . The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$**

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
P(1)	200(3)	181(3)	194(3)	3(2)	-10(2)	-4(2)
P(2)	254(3)	209(3)	237(3)	17(2)	19(2)	19(3)
B	186(13)	148(12)	180(13)	-4(10)	10(10)	15(10)
C(1)	228(12)	186(12)	148(12)	21(9)	13(9)	-14(10)
C(2)	272(13)	183(11)	198(12)	-10(9)	-68(10)	-4(10)
C(3)	117(10)	221(11)	192(11)	-26(9)	-36(8)	-16(9)
C(4)	174(12)	208(12)	269(13)	-19(10)	-23(9)	13(10)
C(5)	179(12)	380(14)	213(13)	-71(11)	24(10)	-9(10)
C(6)	220(12)	389(14)	207(12)	32(11)	5(10)	-70(11)
C(7)	217(12)	257(13)	288(13)	58(11)	-43(10)	-33(10)
C(8)	188(12)	235(12)	217(12)	-39(10)	27(9)	1(10)
C(9)	176(11)	127(10)	201(11)	31(8)	-17(9)	30(8)
C(10)	181(12)	175(11)	249(12)	31(9)	-15(10)	-14(10)
C(11)	296(13)	197(11)	186(12)	0(10)	19(10)	30(10)
C(12)	322(14)	210(12)	211(13)	-10(10)	-93(11)	-22(10)
C(13)	203(13)	239(12)	308(13)	31(10)	-15(10)	-49(10)
C(14)	236(12)	193(11)	219(12)	-2(9)	25(10)	20(9)
C(15)	140(11)	228(12)	188(11)	13(9)	-33(9)	-13(9)
C(16)	200(12)	240(13)	225(12)	-26(10)	-40(9)	5(10)
C(17)	232(13)	402(15)	192(13)	-14(11)	11(10)	-5(11)
C(18)	230(13)	382(15)	237(13)	131(11)	-32(10)	-25(11)
C(19)	229(13)	240(13)	332(14)	64(11)	-24(10)	27(10)
C(20)	226(12)	254(12)	216(12)	-8(10)	27(10)	37(10)
C(21)	197(11)	123(10)	229(12)	6(9)	-13(9)	-14(8)
C(22)	253(13)	279(12)	218(13)	25(10)	12(10)	-46(10)
C(23)	226(13)	308(13)	313(14)	27(11)	-76(11)	-28(10)
C(24)	164(12)	228(12)	449(16)	-64(10)	46(11)	-11(10)
C(25)	281(14)	335(14)	291(14)	-93(11)	77(11)	-58(11)
C(26)	222(13)	282(13)	240(13)	-32(10)	-35(10)	-29(10)
C(27)	210(12)	187(11)	287(13)	2(10)	7(9)	0(9)
C(28)	248(12)	218(12)	223(13)	40(10)	13(10)	0(10)
C(29)	259(13)	236(13)	281(14)	-47(11)	41(10)	-1(10)
C(30)	317(14)	159(12)	390(15)	10(11)	-7(11)	6(10)
C(31)	582(17)	220(13)	286(14)	67(11)	-54(12)	-4(12)
C(32)	503(16)	233(13)	240(14)	-5(11)	-1(12)	-25(11)
C(33)	380(14)	142(11)	198(12)	-1(9)	-23(10)	73(10)
C(34)	389(15)	243(13)	294(14)	21(11)	-82(12)	76(11)
C(35)	519(19)	302(14)	394(17)	22(12)	-160(14)	50(13)
C(36)	680(20)	283(15)	385(18)	-61(12)	-254(16)	138(14)
C(37)	900(30)	298(15)	194(15)	13(12)	91(16)	292(15)
C(38)	423(17)	249(13)	335(15)	44(10)	35(13)	126(12)
Li	330(20)	260(20)	240(20)	8(16)	27(16)	19(17)
N(1)	235(10)	208(9)	247(10)	0(8)	44(8)	13(8)
N(2)	260(10)	233(10)	264(11)	12(8)	43(8)	22(8)
N(3)	470(13)	365(12)	287(11)	-40(9)	14(9)	78(10)
N(4)	351(11)	353(12)	413(13)	151(9)	-8(9)	-51(9)
C(41)	263(13)	319(14)	245(14)	-14(11)	-30(11)	5(11)
C(42)	339(15)	258(13)	292(14)	-39(11)	7(12)	-53(11)



C(43)	538(19)	255(15)	529(18)	-32(13)	292(15)	-51(15)
C(44)	305(15)	289(14)	389(17)	-44(12)	19(13)	27(12)
C(45)	414(17)	306(15)	416(17)	-23(13)	45(14)	61(13)
C(46)	348(16)	392(16)	354(16)	53(13)	38(13)	-37(13)

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**Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for  $[\text{Ph}_2\text{B}(\text{CH}_2\text{PPh}_2)_2][\text{Li}(\text{TMEDA})_2]$ .**

	x	y	z	$U_{\text{iso}}$
H(47A)	3558	-390	284	61
H(47B)	3275	740	534	61
H(48A)	3106	-1515	857	48
H(48B)	2025	-818	666	48
H(49A)	2334	1404	1011	71
H(49B)	1826	1191	1440	71
H(49C)	1256	587	1042	71
H(50A)	2599	-1610	1552	46
H(50B)	1378	-1164	1378	46
H(50C)	1992	-586	1773	46
H(51A)	5949	-1203	857	56
H(51B)	5275	-1310	422	56
H(51C)	4717	-1782	815	56
H(52A)	5034	1592	589	49
H(52B)	5396	669	265	49
H(52C)	6168	857	677	49
H(47C)	3591	-1429	725	51
H(47D)	3464	-593	344	51
H(48C)	2516	773	730	50
H(48D)	1849	-421	723	50
H(49D)	1925	813	1706	66
H(49E)	1068	619	1316	66
H(49F)	2033	1579	1311	66
H(50D)	2564	-1122	1708	65
H(50E)	2969	-1732	1311	65
H(50F)	1665	-1378	1334	65
H(51D)	5503	-1642	845	54
H(51E)	6359	-603	788	54
H(51F)	5620	-1106	405	54
H(52D)	5572	1263	646	67
H(52E)	4225	1458	608	67
H(52F)	4821	764	265	67
H(1A)	10627(14)	5563(14)	960(5)	13(5)
H(1B)	10546(14)	4444(15)	1207(5)	17(5)
H(2A)	7784(19)	7196(17)	1017(6)	60(7)
H(2B)	9037(14)	7222(14)	1337(5)	18(5)
H(4)	7748(13)	6374(13)	1809(5)	4(4)
H(5)	7133(14)	5619(14)	2397(5)	21(5)
H(6)	7493(15)	3692(15)	2555(6)	27(6)
H(7)	8426(15)	2538(16)	2101(5)	29(6)
H(8)	8998(13)	3382(13)	1511(5)	9(5)
H(10)	9592(14)	4729(13)	406(5)	6(5)
H(11)	8713(14)	3809(13)	-131(5)	11(5)
H(12)	6848(14)	3249(14)	-118(5)	17(5)
H(13)	5895(15)	3570(14)	468(5)	19(5)
H(14)	6811(13)	4509(13)	1015(5)	6(4)
H(16)	10383(14)	6191(14)	2403(5)	12(5)

H(17)	10166(14)	4948(13)	2951(5)	15(5)
H(18)	10793(15)	3084(15)	2903(5)	25(6)
H(19)	11565(16)	2431(16)	2331(5)	34(6)
H(20)	11706(14)	3627(14)	1794(5)	14(5)
H(22)	13201(15)	6040(14)	2088(5)	20(5)
H(23)	15156(16)	5968(15)	1986(5)	27(6)
H(24)	15732(16)	5819(14)	1344(5)	23(5)
H(25)	14389(16)	5767(16)	803(6)	32(6)
H(26)	12507(14)	5760(13)	900(5)	12(5)
H(28)	8865(14)	9168(14)	1342(5)	20(5)
H(29)	8597(15)	11079(14)	1389(5)	20(5)
H(30)	8459(14)	12212(15)	816(5)	17(5)
H(31)	8554(16)	11366(16)	170(6)	35(6)
H(32)	8880(16)	9444(16)	138(6)	38(7)
H(34)	6832(17)	6892(17)	544(6)	44(7)
H(35)	5700(20)	6360(20)	-6(7)	73(9)
H(36)	6445(19)	6278(18)	-666(7)	60(8)
H(37)	8339(18)	6843(18)	-699(7)	45(8)
H(38)	9473(15)	7365(15)	-147(5)	23(6)
H(41A)	6655(15)	324(14)	2235(6)	23(5)
H(41B)	6773(16)	707(15)	1778(6)	32(6)
H(42A)	4913(16)	1368(15)	2231(5)	27(6)
H(42B)	6007(15)	2168(15)	2143(5)	26(6)
H(43A)	4392(16)	-447(16)	2197(6)	22(6)
H(43B)	4538(15)	-1574(15)	2041(5)	16(5)
H(44A)	6805(17)	-1070(16)	1489(6)	37(6)
H(44B)	5895(15)	-2003(16)	1589(5)	25(6)
H(44C)	6782(17)	-1670(17)	1951(6)	42(7)
H(45A)	3579(16)	2938(16)	1463(6)	38(6)
H(45B)	3413(17)	2292(16)	1900(6)	37(6)
H(45C)	4271(17)	3375(18)	1878(6)	44(7)
H(46A)	5299(16)	2781(15)	1146(6)	32(6)
H(46B)	6280(18)	2067(17)	1317(6)	38(7)
H(46C)	6051(18)	3250(20)	1569(7)	60(8)

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**Table 6. Crystal data and structure refinement for 4.**

Empirical formula	$C_{51}H_{61}BO_3P_2Pt$ [ $C_{43}H_{45}BOP_2Pt \cdot 2(C_4H_8O)$ ]
Formula weight	989.87 [845.67 · 2(72.10)]
Crystal Habit	blocks
Crystal size	Not measured
Crystal color	Colorless

**Data Collection**

Type of diffractometer	CCD area detector
Wavelength	0.71073 Å MoK $\alpha$
Temperature	98 K
Unit cell dimensions	a = 12.210(4) Å $\alpha$ = 109.614(5)° b = 12.803(4) Å $\beta$ = 104.361(5)° c = 16.205(5) Å $\gamma$ = 96.489(5)°
Volume	2257.6(12) Å <sup>3</sup>
Z	2
Crystal system	Triclinic
Space group	P1 (#2)
Density (calculated)	1.456 g/cm <sup>3</sup>
F(000)	1008
Theta range for data collection	1.73 to 23.35°
Completeness to theta = 23.35°	99.1 %
Index ranges	-13 ≤ h ≤ 13, -13 ≤ k ≤ 14, -18 ≤ l ≤ 18
Data collection scan type	omega scans at 5 f settings
Reflections collected	18315
Independent reflections	6521 [R(int) = 0.0837]
Absorption coefficient	3.220 mm <sup>-1</sup>
Absorption correction	None

**Structure solution and Refinement**

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	6521 / 0 / 523
Treatment of hydrogen atoms	Unrestrained

Goodness-of-fit on $F^2$	1.404
Final R indices [ $I > 2\sigma(I)$ ]	R1 = 0.043, wR2 = 0.083
R indices (all data)	R1 = 0.057, wR2 = 0.087
Largest diff. peak and hole	1.48 and -1.82 e.Å <sup>-3</sup>

**Table 7. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for 4.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{\text{ij}}$  tensor.**

	x	y	z	$U(\text{eq})$
Pt(1)	951(1)	2561(1)	3443(1)	23(1)
P(1)	2157(2)	1516(2)	3993(1)	27(1)
P(2)	1879(2)	2653(2)	2463(1)	25(1)
B	3874(7)	1653(7)	3039(6)	25(2)
C(1)	4246(6)	415(7)	2756(5)	27(2)
C(2)	5125(6)	201(6)	3379(5)	25(2)
C(3)	5491(6)	-822(6)	3149(5)	25(2)
C(4)	5002(6)	-1647(7)	2289(5)	29(2)
C(5)	4160(6)	-1452(7)	1661(5)	32(2)
C(6)	3792(6)	-432(7)	1895(5)	28(2)
C(7)	4990(6)	2584(7)	3109(5)	28(2)
C(8)	5513(6)	3595(7)	3830(5)	28(2)
C(9)	6463(6)	4336(7)	3850(5)	33(2)
C(10)	6920(7)	4044(8)	3109(6)	38(2)
C(11)	6407(6)	3031(7)	2374(6)	34(2)
C(12)	5476(6)	2314(6)	2377(5)	29(2)
C(13)	3639(6)	2009(6)	4055(5)	29(2)
C(14)	2717(6)	1600(6)	2211(5)	25(2)
C(15)	2155(6)	1726(6)	5165(5)	23(2)
C(16)	2796(6)	2714(6)	5877(5)	28(2)
C(17)	2737(6)	2952(6)	6762(5)	29(2)
C(18)	2020(6)	2195(7)	6952(5)	28(2)
C(19)	1387(6)	1201(7)	6240(5)	30(2)
C(20)	1448(5)	970(6)	5355(5)	25(2)
C(21)	1802(7)	-13(6)	3418(5)	29(2)
C(22)	2538(7)	-668(8)	3691(6)	44(2)
C(23)	2267(11)	-1824(10)	3255(8)	62(3)
C(24)	1235(11)	-2349(8)	2549(8)	61(3)
C(25)	472(8)	-1726(8)	2271(6)	44(2)
C(26)	776(7)	-576(7)	2700(5)	31(2)
C(27)	2812(5)	4001(6)	2761(5)	25(2)
C(28)	2909(6)	4912(6)	3563(5)	29(2)
C(29)	3608(6)	5951(7)	3780(6)	36(2)
C(30)	4238(6)	6102(7)	3201(6)	39(2)
C(31)	4162(6)	5196(7)	2415(6)	37(2)
C(32)	3458(6)	4142(7)	2186(5)	30(2)
C(33)	803(6)	2432(7)	1381(5)	26(2)
C(34)	-2(7)	1402(7)	918(5)	36(2)
C(35)	-855(8)	1214(7)	112(5)	42(2)
C(36)	-926(7)	2047(8)	-238(5)	42(2)
C(37)	-166(7)	3051(9)	196(6)	58(3)
C(38)	699(6)	3262(8)	1009(7)	55(3)
C(39)	-72(6)	3622(6)	3059(5)	31(2)
O	-120(4)	2397(4)	4299(3)	25(1)
C(40)	24(6)	3279(7)	5172(5)	35(2)
C(41)	-1165(6)	3350(7)	5249(6)	43(2)
C(42)	-1883(7)	2214(7)	4614(5)	40(2)
C(43)	-1349(6)	1909(7)	3839(6)	39(2)
O(3)	6617(14)	4739(13)	2(9)	240(8)
C(45)	6910(30)	4620(20)	873(10)	390(30)
C(46)	8190(15)	5051(14)	1279(8)	132(6)
C(47)	8382(7)	5731(10)	844(6)	77(4)
C(48)	7535(15)	5320(20)	77(17)	350(20)

O(2)	7751(12)	8518(13)	573(12)	175(6)
C(50)	7951(8)	9440(11)	1367(7)	59(3)
C(51)	6966(18)	9908(10)	1265(12)	124(7)
C(52)	6187(9)	9130(20)	414(10)	173(11)
C(53)	6600(20)	8250(11)	105(11)	204(12)

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**Table 8. Bond lengths [Å] and angles [°] for 4.**

Pt(1)-C(39)	2.087(6)	C(24)-C(25)	1.381(13)
Pt(1)-O	2.170(4)	C(24)-H(24)	0.9300
Pt(1)-P(2)	2.1927(16)	C(25)-C(26)	1.360(10)
Pt(1)-P(1)	2.3130(19)	C(25)-H(25)	0.9300
P(1)-C(21)	1.809(8)	C(26)-H(26)	0.9300
P(1)-C(13)	1.814(6)	C(27)-C(28)	1.392(10)
P(1)-C(15)	1.827(7)	C(27)-C(32)	1.403(8)
P(2)-C(14)	1.784(7)	C(28)-C(29)	1.379(9)
P(2)-C(27)	1.798(7)	C(28)-H(28)	0.9300
P(2)-C(33)	1.823(7)	C(29)-C(30)	1.400(9)
B-C(1)	1.646(11)	C(29)-H(29)	0.9300
B-C(7)	1.660(10)	C(30)-C(31)	1.379(11)
B-C(13)	1.661(9)	C(30)-H(30)	0.9300
B-C(14)	1.667(10)	C(31)-C(32)	1.393(10)
C(1)-C(6)	1.376(10)	C(31)-H(31)	0.9300
C(1)-C(2)	1.401(9)	C(32)-H(32)	0.9300
C(2)-C(3)	1.393(9)	C(33)-C(38)	1.389(10)
C(2)-H(2)	0.9300	C(33)-C(34)	1.394(10)
C(3)-C(4)	1.368(10)	C(34)-C(35)	1.379(10)
C(3)-H(3)	0.9300	C(34)-H(34)	0.9300
C(4)-C(5)	1.365(9)	C(35)-C(36)	1.368(10)
C(4)-H(4)	0.9300	C(35)-H(35)	0.9300
C(5)-C(6)	1.392(10)	C(36)-C(37)	1.344(11)
C(5)-H(5)	0.9300	C(36)-H(36)	0.9300
C(6)-H(6)	0.9300	C(37)-C(38)	1.388(11)
C(7)-C(8)	1.370(10)	C(37)-H(37)	0.9300
C(7)-C(12)	1.414(9)	C(38)-H(38)	0.9300
C(8)-C(9)	1.400(9)	C(39)-H(39A)	0.9600
C(8)-H(8)	0.9300	C(39)-H(39B)	0.9600
C(9)-C(10)	1.404(9)	C(39)-H(39C)	0.9600
C(9)-H(9)	0.9300	O-C(40)	1.438(8)
C(10)-C(11)	1.379(11)	O-C(43)	1.456(8)
C(10)-H(10)	0.9300	C(40)-C(41)	1.498(9)
C(11)-C(12)	1.380(9)	C(40)-H(40A)	0.9700
C(11)-H(11)	0.9300	C(40)-H(40B)	0.9700
C(12)-H(12)	0.9300	C(41)-C(42)	1.484(10)
C(13)-H(13A)	0.9700	C(41)-H(41A)	0.9700
C(13)-H(13B)	0.9700	C(41)-H(41B)	0.9700
C(14)-H(14A)	0.9700	C(42)-C(43)	1.512(9)
C(14)-H(14B)	0.9700	C(42)-H(42A)	0.9700
C(15)-C(16)	1.376(9)	C(42)-H(42B)	0.9700
C(15)-C(20)	1.385(8)	C(43)-H(43A)	0.9700
C(16)-C(17)	1.385(9)	C(43)-H(43B)	0.9700
C(16)-H(16)	0.9300	O(3)-C(48)	1.230(16)
C(17)-C(18)	1.394(9)	O(3)-C(45)	1.430(19)
C(17)-H(17)	0.9300	C(45)-C(46)	1.50(3)
C(18)-C(19)	1.378(10)	C(45)-H(45A)	0.9700
C(18)-H(18)	0.9300	C(45)-H(45B)	0.9700
C(19)-C(20)	1.387(9)	C(46)-C(47)	1.325(14)
C(19)-H(19)	0.9300	C(46)-H(46A)	0.9700
C(20)-H(20)	0.9300	C(46)-H(46B)	0.9700
C(21)-C(22)	1.389(10)	C(47)-C(48)	1.30(2)
C(21)-C(26)	1.397(10)	C(47)-H(47A)	0.9700
C(22)-C(23)	1.368(12)	C(47)-H(47B)	0.9700
C(22)-H(22)	0.9300	C(48)-H(48A)	0.9700
C(23)-C(24)	1.387(13)	C(48)-H(48B)	0.9700
C(23)-H(23)	0.9300	O(2)-C(53)	1.36(2)



O(2)-C(50)	1.367(15)	C(7)-C(8)-C(9)	122.8(7)
C(50)-C(51)	1.402(17)	C(7)-C(8)-H(8)	118.6
C(50)-H(50A)	0.9700	C(9)-C(8)-H(8)	118.6
C(50)-H(50B)	0.9700	C(8)-C(9)-C(10)	119.6(7)
C(51)-C(52)	1.417(19)	C(8)-C(9)-H(9)	120.2
C(51)-H(51A)	0.9700	C(10)-C(9)-H(9)	120.2
C(51)-H(51B)	0.9700	C(11)-C(10)-C(9)	118.7(7)
C(52)-C(53)	1.29(2)	C(11)-C(10)-H(10)	120.7
C(52)-H(52A)	0.9700	C(9)-C(10)-H(10)	120.7
C(52)-H(52B)	0.9700	C(10)-C(11)-C(12)	120.4(7)
C(53)-H(53A)	0.9700	C(10)-C(11)-H(11)	119.8
C(53)-H(53B)	0.9700	C(12)-C(11)-H(11)	119.8
		C(11)-C(12)-C(7)	122.4(7)
C(39)-Pt(1)-O	87.39(19)	C(11)-C(12)-H(12)	118.8
C(39)-Pt(1)-P(2)	89.90(17)	C(7)-C(12)-H(12)	118.8
O-Pt(1)-P(2)	174.44(14)	B-C(13)-P(1)	114.2(5)
C(39)-Pt(1)-P(1)	174.9(2)	B-C(13)-H(13A)	108.7
O-Pt(1)-P(1)	91.17(12)	P(1)-C(13)-H(13A)	108.7
P(2)-Pt(1)-P(1)	91.93(6)	B-C(13)-H(13B)	108.7
C(21)-P(1)-C(13)	107.5(3)	P(1)-C(13)-H(13B)	108.7
C(21)-P(1)-C(15)	103.1(3)	H(13A)-C(13)-H(13B)	107.6
C(13)-P(1)-C(15)	106.9(3)	B-C(14)-P(2)	118.8(5)
C(21)-P(1)-Pt(1)	118.8(3)	B-C(14)-H(14A)	107.6
C(13)-P(1)-Pt(1)	111.0(2)	P(2)-C(14)-H(14A)	107.6
C(15)-P(1)-Pt(1)	108.8(2)	B-C(14)-H(14B)	107.6
C(14)-P(2)-C(27)	107.0(3)	P(2)-C(14)-H(14B)	107.6
C(14)-P(2)-C(33)	106.7(3)	H(14A)-C(14)-H(14B)	107.0
C(27)-P(2)-C(33)	104.6(3)	C(16)-C(15)-C(20)	118.3(6)
C(14)-P(2)-Pt(1)	114.5(2)	C(16)-C(15)-P(1)	119.2(5)
C(27)-P(2)-Pt(1)	115.6(2)	C(20)-C(15)-P(1)	122.2(6)
C(33)-P(2)-Pt(1)	107.61(19)	C(15)-C(16)-C(17)	121.0(6)
C(1)-B-C(7)	106.0(5)	C(15)-C(16)-H(16)	119.5
C(1)-B-C(13)	110.4(5)	C(17)-C(16)-H(16)	119.5
C(7)-B-C(13)	109.4(6)	C(16)-C(17)-C(18)	120.6(7)
C(1)-B-C(14)	109.2(6)	C(16)-C(17)-H(17)	119.7
C(7)-B-C(14)	109.5(5)	C(18)-C(17)-H(17)	119.7
C(13)-B-C(14)	112.1(5)	C(19)-C(18)-C(17)	118.3(7)
C(6)-C(1)-C(2)	116.0(7)	C(19)-C(18)-H(18)	120.9
C(6)-C(1)-B	123.4(6)	C(17)-C(18)-H(18)	120.9
C(2)-C(1)-B	120.5(7)	C(18)-C(19)-C(20)	120.7(6)
C(3)-C(2)-C(1)	121.9(7)	C(18)-C(19)-H(19)	119.6
C(3)-C(2)-H(2)	119.0	C(20)-C(19)-H(19)	119.6
C(1)-C(2)-H(2)	119.0	C(15)-C(20)-C(19)	121.0(7)
C(4)-C(3)-C(2)	120.1(7)	C(15)-C(20)-H(20)	119.5
C(4)-C(3)-H(3)	120.0	C(19)-C(20)-H(20)	119.5
C(2)-C(3)-H(3)	120.0	C(22)-C(21)-C(26)	117.8(8)
C(5)-C(4)-C(3)	119.3(7)	C(22)-C(21)-P(1)	120.7(7)
C(5)-C(4)-H(4)	120.4	C(26)-C(21)-P(1)	121.6(6)
C(3)-C(4)-H(4)	120.4	C(23)-C(22)-C(21)	120.5(9)
C(4)-C(5)-C(6)	120.5(7)	C(23)-C(22)-H(22)	119.7
C(4)-C(5)-H(5)	119.7	C(21)-C(22)-H(22)	119.7
C(6)-C(5)-H(5)	119.7	C(22)-C(23)-C(24)	119.7(9)
C(1)-C(6)-C(5)	122.2(7)	C(22)-C(23)-H(23)	120.1
C(1)-C(6)-H(6)	118.9	C(24)-C(23)-H(23)	120.1
C(5)-C(6)-H(6)	118.9	C(25)-C(24)-C(23)	121.3(10)
C(8)-C(7)-C(12)	116.0(6)	C(25)-C(24)-H(24)	119.3
C(8)-C(7)-B	125.8(6)	C(23)-C(24)-H(24)	119.3
C(12)-C(7)-B	118.1(7)	C(26)-C(25)-C(24)	117.7(9)

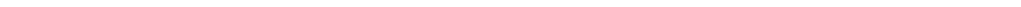
C(26)-C(25)-H(25)	121.1	C(40)-C(41)-H(41A)	111.0
C(24)-C(25)-H(25)	121.1	C(42)-C(41)-H(41B)	111.0
C(25)-C(26)-C(21)	122.8(8)	C(40)-C(41)-H(41B)	111.0
C(25)-C(26)-H(26)	118.6	H(41A)-C(41)-H(41B)	109.0
C(21)-C(26)-H(26)	118.6	C(41)-C(42)-C(43)	102.9(5)
C(28)-C(27)-C(32)	119.3(6)	C(41)-C(42)-H(42A)	111.2
C(28)-C(27)-P(2)	120.7(5)	C(43)-C(42)-H(42A)	111.2
C(32)-C(27)-P(2)	120.0(6)	C(41)-C(42)-H(42B)	111.2
C(29)-C(28)-C(27)	120.5(6)	C(43)-C(42)-H(42B)	111.2
C(29)-C(28)-H(28)	119.7	H(42A)-C(42)-H(42B)	109.1
C(27)-C(28)-H(28)	119.7	O-C(43)-C(42)	103.9(6)
C(28)-C(29)-C(30)	120.4(7)	O-C(43)-H(43A)	111.0
C(28)-C(29)-H(29)	119.8	C(42)-C(43)-H(43A)	111.0
C(30)-C(29)-H(29)	119.8	O-C(43)-H(43B)	111.0
C(31)-C(30)-C(29)	119.2(7)	C(42)-C(43)-H(43B)	111.0
C(31)-C(30)-H(30)	120.4	H(43A)-C(43)-H(43B)	109.0
C(29)-C(30)-H(30)	120.4	C(48)-O(3)-C(45)	100.0(17)
C(30)-C(31)-C(32)	121.0(6)	O(3)-C(45)-C(46)	105.5(14)
C(30)-C(31)-H(31)	119.5	O(3)-C(45)-H(45A)	110.7
C(32)-C(31)-H(31)	119.5	C(46)-C(45)-H(45A)	110.6
C(31)-C(32)-C(27)	119.6(7)	O(3)-C(45)-H(45B)	110.6
C(31)-C(32)-H(32)	120.2	C(46)-C(45)-H(45B)	110.7
C(27)-C(32)-H(32)	120.2	H(45A)-C(45)-H(45B)	108.8
C(38)-C(33)-C(34)	117.5(7)	C(47)-C(46)-C(45)	101.2(12)
C(38)-C(33)-P(2)	123.4(7)	C(47)-C(46)-H(46A)	111.5
C(34)-C(33)-P(2)	119.0(5)	C(45)-C(46)-H(46A)	111.5
C(35)-C(34)-C(33)	120.9(7)	C(47)-C(46)-H(46B)	111.5
C(35)-C(34)-H(34)	119.5	C(45)-C(46)-H(46B)	111.6
C(33)-C(34)-H(34)	119.5	H(46A)-C(46)-H(46B)	109.3
C(36)-C(35)-C(34)	120.0(8)	C(48)-C(47)-C(46)	104.3(11)
C(36)-C(35)-H(35)	120.0	C(48)-C(47)-H(47A)	110.9
C(34)-C(35)-H(35)	120.0	C(46)-C(47)-H(47A)	110.9
C(37)-C(36)-C(35)	120.4(8)	C(48)-C(47)-H(47B)	110.8
C(37)-C(36)-H(36)	119.8	C(46)-C(47)-H(47B)	110.9
C(35)-C(36)-H(36)	119.8	H(47A)-C(47)-H(47B)	108.9
C(36)-C(37)-C(38)	120.7(7)	O(3)-C(48)-C(47)	121.6(16)
C(36)-C(37)-H(37)	119.6	O(3)-C(48)-H(48A)	106.8
C(38)-C(37)-H(37)	119.6	C(47)-C(48)-H(48A)	106.9
C(37)-C(38)-C(33)	120.5(8)	O(3)-C(48)-H(48B)	107.0
C(37)-C(38)-H(38)	119.8	C(47)-C(48)-H(48B)	107.0
C(33)-C(38)-H(38)	119.8	H(48A)-C(48)-H(48B)	106.7
Pt(1)-C(39)-H(39A)	109.5	C(53)-O(2)-C(50)	108.4(12)
Pt(1)-C(39)-H(39B)	109.5	O(2)-C(50)-C(51)	106.6(11)
H(39A)-C(39)-H(39B)	109.5	O(2)-C(50)-H(50A)	110.4
Pt(1)-C(39)-H(39C)	109.5	C(51)-C(50)-H(50A)	110.4
H(39A)-C(39)-H(39C)	109.5	O(2)-C(50)-H(50B)	110.4
H(39B)-C(39)-H(39C)	109.5	C(51)-C(50)-H(50B)	110.4
C(40)-O-C(43)	109.0(5)	H(50A)-C(50)-H(50B)	108.6
C(40)-O-Pt(1)	121.7(4)	C(50)-C(51)-C(52)	104.0(11)
C(43)-O-Pt(1)	117.2(4)	C(50)-C(51)-H(51A)	111.0
O-C(40)-C(41)	106.8(6)	C(52)-C(51)-H(51A)	110.9
O-C(40)-H(40A)	110.4	C(50)-C(51)-H(51B)	111.0
C(41)-C(40)-H(40A)	110.4	C(52)-C(51)-H(51B)	111.0
O-C(40)-H(40B)	110.4	H(51A)-C(51)-H(51B)	109.0
C(41)-C(40)-H(40B)	110.4	C(53)-C(52)-C(51)	110.1(12)
H(40A)-C(40)-H(40B)	108.6	C(53)-C(52)-H(52A)	109.6
C(42)-C(41)-C(40)	103.8(6)	C(51)-C(52)-H(52A)	109.6
C(42)-C(41)-H(41A)	111.0	C(53)-C(52)-H(52B)	109.6

C(51)-C(52)-H(52B)	109.7	O(2)-C(53)-H(53A)	110.2
H(52A)-C(52)-H(52B)	108.2	C(52)-C(53)-H(53B)	110.2
C(52)-C(53)-O(2)	107.5(14)	O(2)-C(53)-H(53B)	110.2
C(52)-C(53)-H(53A)	110.2	H(53A)-C(53)-H(53B)	108.5

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**Table 9. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for 4. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$**

	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	U <sup>13</sup>	U <sup>12</sup>
Pt(1)	17(1)	33(1)	36(1)	24(1)	18(1)	14(1)
P(1)	23(1)	43(1)	37(1)	31(1)	22(1)	21(1)
P(2)	18(1)	36(1)	37(1)	26(1)	17(1)	14(1)
B	25(5)	34(6)	34(5)	24(5)	19(4)	14(4)
C(1)	20(4)	45(5)	33(5)	30(4)	18(4)	11(4)
C(2)	22(4)	38(5)	29(4)	19(4)	21(4)	11(4)
C(3)	22(4)	29(5)	33(5)	19(4)	13(4)	11(4)
C(4)	24(4)	33(5)	44(5)	22(5)	19(4)	15(4)
C(5)	21(4)	42(6)	33(5)	16(4)	8(4)	4(4)
C(6)	21(4)	43(6)	37(5)	26(5)	18(4)	16(4)
C(7)	27(4)	41(6)	43(5)	36(5)	19(4)	29(4)
C(8)	35(4)	34(5)	38(5)	26(5)	24(4)	24(4)
C(9)	36(5)	34(5)	43(5)	26(4)	17(4)	18(4)
C(10)	32(5)	58(7)	61(6)	51(6)	28(5)	31(5)
C(11)	29(4)	48(6)	46(5)	29(5)	24(4)	20(4)
C(12)	24(4)	38(5)	40(5)	24(4)	20(4)	13(4)
C(13)	24(4)	46(5)	37(5)	29(4)	19(4)	19(4)
C(14)	20(4)	34(5)	40(5)	26(4)	19(4)	14(4)
C(15)	19(4)	28(5)	35(5)	21(4)	16(4)	13(4)
C(16)	23(4)	29(5)	50(5)	29(5)	22(4)	17(4)
C(17)	23(4)	32(5)	38(5)	18(4)	8(4)	13(4)
C(18)	22(4)	42(6)	28(4)	22(4)	11(4)	6(4)
C(19)	20(4)	44(6)	35(5)	22(4)	16(4)	4(4)
C(20)	15(4)	34(5)	32(4)	16(4)	11(3)	7(4)
C(21)	42(5)	37(5)	34(5)	27(4)	33(4)	24(4)
C(22)	53(6)	56(7)	67(6)	47(6)	49(5)	39(5)
C(23)	99(9)	65(9)	96(9)	69(7)	80(8)	65(7)
C(24)	107(9)	40(7)	76(8)	30(6)	78(8)	34(7)
C(25)	59(6)	39(6)	55(6)	23(5)	42(5)	13(5)
C(26)	45(5)	29(6)	36(5)	15(4)	32(5)	11(4)
C(27)	16(4)	31(5)	41(5)	28(4)	14(4)	10(4)
C(28)	24(4)	32(5)	55(5)	37(5)	24(4)	16(4)
C(29)	32(4)	45(6)	55(5)	33(5)	25(4)	26(4)
C(30)	28(4)	43(6)	72(6)	41(5)	28(5)	21(4)
C(31)	24(4)	54(6)	69(6)	51(6)	29(4)	26(4)
C(32)	25(4)	48(6)	40(5)	35(4)	17(4)	19(4)
C(33)	24(4)	46(6)	34(4)	30(4)	24(4)	28(4)
C(34)	58(6)	20(5)	32(5)	9(4)	14(4)	20(5)
C(35)	64(6)	36(6)	20(4)	8(4)	4(4)	21(5)
C(36)	22(4)	84(7)	41(5)	40(6)	17(4)	28(5)
C(37)	21(5)	109(9)	87(7)	89(7)	14(5)	19(6)
C(38)	18(4)	78(7)	101(7)	75(7)	17(5)	11(5)
C(39)	19(4)	48(5)	57(5)	42(5)	29(4)	21(4)
O	25(3)	37(3)	26(3)	17(3)	17(2)	17(2)
C(40)	38(5)	43(5)	42(5)	25(5)	25(4)	21(4)
C(41)	34(5)	47(6)	54(6)	11(5)	32(5)	11(5)
C(42)	34(5)	44(6)	58(6)	23(5)	34(4)	14(4)
C(43)	22(4)	45(6)	70(6)	35(5)	27(4)	10(4)
O(3)	272(18)	265(16)	153(11)	96(11)	45(12)	-63(14)
C(45)	480(50)	450(40)	41(10)	79(16)	-23(16)	-330(40)
C(46)	208(17)	184(16)	62(9)	70(10)	59(11)	152(15)
C(47)	13(4)	166(11)	51(6)	81(7)	-18(4)	-42(6)
C(48)	114(14)	540(40)	450(40)	460(40)	-80(19)	-160(20)
O(2)	177(12)	261(16)	228(14)	172(14)	143(12)	155(12)
C(50)	37(5)	94(9)	50(7)	43(6)	12(5)	-9(6)
C(51)	234(19)	55(8)	180(16)	67(10)	178(16)	73(11)
C(52)	35(7)	480(30)	87(11)	175(18)	44(7)	82(13)
C(53)	300(30)	44(9)	122(13)	-9(8)	-140(15)	65(13)



**Table 10. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for 4.**

	x	y	z	U(eq)
H(2)	5475	758	3964	30
H(3)	6069	-943	3581	30
H(4)	5241	-2332	2134	35
H(5)	3830	-2005	1073	38
H(6)	3219	-318	1454	34
H(8)	5224	3799	4328	34
H(9)	6790	5018	4351	39
H(10)	7555	4523	3113	46
H(11)	6689	2831	1872	41
H(12)	5157	1628	1880	35
H(13A)	3845	2829	4360	35
H(13B)	4147	1700	4431	35
H(14A)	2205	864	2001	30
H(14B)	2974	1631	1698	30
H(16)	3275	3231	5762	33
H(17)	3182	3622	7235	35
H(18)	1970	2358	7544	34
H(19)	913	678	6354	36
H(20)	1008	298	4882	30
H(22)	3220	-318	4174	53
H(23)	2772	-2257	3430	75
H(24)	1053	-3135	2257	73
H(25)	-226	-2081	1806	53
H(26)	280	-146	2507	37
H(28)	2500	4818	3956	34
H(29)	3660	6555	4315	44
H(30)	4702	6805	3344	46
H(31)	4587	5290	2032	44
H(32)	3417	3536	1656	36
H(34)	37	835	1155	43
H(35)	-1381	520	-192	50
H(36)	-1504	1919	-780	50
H(37)	-220	3610	-50	70
H(38)	1212	3963	1306	66
H(39A)	-264	4091	3581	37
H(39B)	-769	3169	2580	37
H(39C)	348	4094	2838	37
H(40A)	422	3998	5203	42
H(40B)	475	3098	5669	42
H(41A)	-1176	3500	5874	52
H(41B)	-1438	3942	5061	52
H(42A)	-2687	2248	4393	47
H(42B)	-1833	1670	4911	47
H(43A)	-1486	1092	3528	47
H(43B)	-1661	2236	3395	47
H(45A)	6695	3834	794	463
H(45B)	6509	5067	1267	463
H(46A)	8411	5465	1939	158
H(46B)	8599	4438	1149	158
H(47A)	8373	6507	1201	92
H(47B)	9123	5714	727	92
H(48A)	7346	5965	-78	417
H(48B)	7836	4876	-395	417
H(50A)	8631	9990	1462	70
H(50B)	8075	9212	1891	70
H(51A)	7148	10657	1249	149
H(51B)	6643	9965	1765	149
H(52A)	5449	8907	496	208

H(52B)	6058	9500	-28	208
H(53A)	6493	8040	-549	245
H(53B)	6216	7614	198	245

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