Oxidative Addition of Aryl Tosylates to Palladium(0) and Coupling of Unactivated Aryl Tosylates at Room Temperature

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General Methods. ^1H , ^{13}C , and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 or 500 MHz Spectrometer, General Electric QE 300 MHz spectrometer, or a General Electric Omega 500 spectrometer with tetramethylsilane or residual protiated solvent as a reference. Elemental analyses were performed by Robertson Microlabs, Inc., Madison, NJ. All ^{31}P and ^{13}C NMR spectra were proton decoupled. GC and GC/MS analyses were conducted with an HP-1 methyl silicone column. Ether, toluene, tetrahydrofuran, benzene, and pentane were distilled from sodium/benzophenone. PPF-*t*-Bu (PPF-*t*-Bu = 1-diphenylphosphino-2-di-*t*-butylphosphinoethylferrocene) was purchased from Strem Chemicals and used without further purification. CyPF-*t*-Bu (CyPF-*t*-Bu = 1-dicyclohexylphosphino-2-di-*t*-butylphosphinoethylferrocene) was obtained from Solvias AG and Strem Chemicals and used without further purification. $\{\text{Pd}[\text{P}(o\text{-tolyl})_3](\text{Ph})(\mu\text{-Br})\}_2$,¹ $\text{Pd}(\text{dba})_2$,² and $\text{Pd}[\text{P}(o\text{-tolyl})_3]_2$ ¹ were prepared by literature procedures.

Synthesis of $\text{Pd}(\text{PPF-}t\text{-Bu})[\text{P}(o\text{-tolyl})_3]$ (1). A solution of 9.1 mg (0.017 mmol) of PPF-*t*-Bu in C_6D_6 was added dropwise to a suspension of 12.0 mg (0.0168 mmol) of $\text{Pd}[\text{P}(o\text{-tolyl})_3]_2$ in C_6D_6 . The suspension was stirred at room temperature for 10 min, during which time the solids dissolved. The solution was transferred to an NMR sample tube and was characterized by ^1H and $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. ^1H NMR (C_6D_6) 0.98 (d, $J=12.0$ Hz, 9H), 1.24 (d, $J=11.2$ Hz, 9H), 1.86 (t, $J=6.4$ Hz, 3H), 2.35 (s, 9H, coordinated $\text{P}(o\text{-tolyl})_3$), 3.32 (m, 1H), 3.72 (br s, 1H), 3.86 (s, 5H), 3.91 (t, $J=2.8$ Hz, 1H), 4.18 (br s, 1H), 6.87-7.18 (m, 16H), 7.32 (t, $J=10.0$ Hz, 2H), 7.90 (br s, 2H), 7.99 (br t, $J=8.0$ Hz, 2H). $^{31}\text{P}\{^1\text{H}\}$ NMR (C_6D_6) 1.28 (t, $J=87.9$ Hz), 19.78 (br t, $J=83.0$ Hz), 71.69 (br t, $J=87.9$ Hz).³

Synthesis of $\text{Pd}(\text{CyPF-}t\text{-Bu})[\text{P}(o\text{-tolyl})_3]$ (2). A solution of 9.4 mg (0.017 mmol) of CyPF-*t*-Bu in C_6D_6 was added dropwise to a suspension of 12.1 mg (0.0169 mmol) of $\text{Pd}[\text{P}(o\text{-tolyl})_3]_2$ in C_6D_6 . The suspension was stirred at room temperature for 10 min, during which time the solids dissolved. The solution was transferred to an NMR sample tube and was characterized by ^1H and $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. ^1H NMR (C_6D_6) 0.90-2.0 (m, 22 Cy H), 1.05 (br d, $J=8.8$ Hz, 9H), 1.26 (d, $J=11.6$ Hz, 9H), 1.88 (t, $J=6.8$ Hz, 3H), 2.2-2.6 (br, 9H), 3.15 (m, 1H), 4.05 (t, $J=2.0$ Hz, 1H), 4.13 (s, 5H), 4.25 (br s, 1H), 4.36 (br s, 1H), 6.99-7.10 (m, 12H). $^{31}\text{P}\{^1\text{H}\}$ NMR (C_6D_6) 11.72 (br), 21.42 (br), 69.59 (t, $J=87.9$ Hz).³

Independent Synthesis of $\text{Pd}(\text{PPF-}t\text{-Bu})(\text{Ph})(\text{OTs})$ (3). A solution of 200 mg (0.240 mmol) of $\text{Pd}(\text{PPF-}t\text{-Bu})(\text{Ph})(\text{Br})$ (5) in C_6H_6 was added to a suspension of 68 mg (0.24 mmol) of AgOTs in C_6H_6 . The reaction was complete after stirring for 20 min, as determined by $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. The suspension was filtered through Celite, and the orange solution was concentrated under vacuum. Addition of pentane and stirring of the resulting mixture led to

precipitation of the product as an orange solid. The product was filtered, washed with pentane, and dried under vacuum to give 162 mg (75% yield) of product. ^1H NMR (CD_2Cl_2) 1.36 (d, $J=13.2$ Hz, 9H), 1.63 (d, $J=12.0$ Hz, 9H), 2.01 (t, $J=7.2$ Hz, 3H), 2.21 (s, 3H), 3.28 (m, 1H), 3.69 (s, 5H), 4.10 (br s, 1H), 4.34 (br s, 1H), 4.61 (br s, 1H), 6.45 (br, 2H), 6.45 (t, $J=7.2$ Hz, 1H), 6.58 (br, 2H), 6.80 (d, $J=8.4$ Hz, 2H), 6.90 (d, $J=7.6$ Hz, 2H), 7.09-7.19 (m, 4H), 7.26-7.31 (m, 1H), 7.59-7.64 (m, 3H), 8.17-8.22 (m, 2H). ^{13}C NMR (CD_2Cl_2 , 40 °C) 17.71 (d, $J=5.0$ Hz), 21.30, 31.64 (d, $J=5.9$ Hz), 31.70 (d, $J=6.8$ Hz), 32.43 (m), 37.47, 38.52, 68.62 (d, $J=7.0$ Hz), 70.88 (d, $J=8.3$ Hz), 70.95, 75.73 (d, $J=5.2$ Hz), 78.26 (dd, $J=47.63$, 10.80 Hz), 95.96 (dd, $J=16.8$, 6.5 Hz), 123.22, 126.40, 126.77, 128.04 (d, $J=11.3$ Hz), 128.31, 128.77 (d, $J=11.4$ Hz), 129.92 ($J=2.8$ Hz), 131.77 (br), 132.12 (d, $J=58.1$ Hz), 132.24 (d, $J=58.1$ Hz), 133.73 (d, $J=9.6$ Hz), 136.02 (d, $J=12.6$ Hz), 136.67 (br), 139.05, 140.95, 157.83 (d, $J=103.3$ Hz). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2) 25.4 (d, $J=37.4$ Hz), 67.5 (d, $J=37.5$ Hz). Anal. calc'd for $\text{C}_{45}\text{H}_{52}\text{P}_2\text{O}_3\text{SFePd}$: C, 60.24; H, 5.85. Found: C, 59.94; H, 5.78.

Independent Synthesis of $\text{Pd}(\text{CyPF-}t\text{-Bu})(\text{Ph})(\text{OTs})$ (4). A solution of 113 mg (0.140 mmol) of $\text{Pd}(\text{CyPF-}t\text{-Bu})(\text{Ph})(\text{Br})$ (**6**) in C_6H_6 was added to a suspension of 48 mg (0.17 mmol) of AgOTs in C_6H_6 . The reaction was complete after stirring for 20 min, as determined by $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. The suspension was filtered through Celite, and the orange solution was concentrated under vacuum. Addition of pentane and stirring of the resulting mixture led to precipitation of the product as an orange solid. The product was filtered, washed with pentane, and dried under vacuum to give 90 mg (71% yield) of product. ^1H NMR (CD_2Cl_2)

0.76 – 2.95 (m, 22 Cy H), 1.11 (d, $J=13.5$ Hz, 9H), 1.62 (d, $J=12.0$ Hz, 9H), 1.92 (t, $J=8.0$ Hz, 3H), 2.26 (s, 3H), 3.02 (m, 1H), 4.26 (s, 5H), 4.52 (t, $J=2.0$ Hz, 1H), 4.56 (br s, 1H), 4.95 (br s, 1H), 6.79-6.84 (m, 4H), 6.89 (d, $J=7.0$ Hz, 2H), 7.05-7.12 (m, 3H). $^{31}\text{P}\{^1\text{H}\}$ NMR (C_7H_8 , -40 °C) 25.2 (d, $J=31.9$ Hz), 71.6 (d, $J=30.5$ Hz). Anal. calc'd for $\text{C}_{45}\text{H}_{64}\text{P}_2\text{O}_3\text{SFePd}$: C, 59.44; H, 7.11. Found: C, 59.71; H, 7.34.

Independent Synthesis of $\text{Pd}(\text{PPF-}t\text{-Bu})(\text{Ph})(\text{Br})$ (5). A solution of 218 mg (0.400 mmol) of PPF-*t*-Bu in THF was added dropwise to a suspension of 200 mg (0.180 mmol) $\{\text{Pd}[\text{P}(o\text{-tolyl})_3](\text{Ph})(\mu\text{-Br})\}_2$ in THF. The reaction mixture was stirred at room temperature for 20 min, during which time all the solids dissolved. After 20 min the reaction was complete, as determined by $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. The orange solution was filtered through Celite and concentrated. Addition of pentane led to precipitation of 226 mg (78% yield) of the product as an orange solid. The product was filtered, washed with pentane, and dried under vacuum. ^1H NMR (CD_2Cl_2) 1.39 (d, $J=12.8$ Hz, 9H), 1.72 (d, $J=12.0$ Hz, 9H), 2.07 (t, $J=8.0$ Hz, 3H), 3.43 (m, 1H), 3.67 (s, 5H), 4.22 (m, 1H), 4.33 (m, 1H), 4.59 (m, 1H), 6.36-7.28 (m, 10H), 7.60-7.66 (m, 3H), 8.18-8.24 (m, 2H). ^{13}C NMR (CD_2Cl_2 , -15 °C) 18.18 (d, $J=3.0$ Hz), 31.05 (d, $J=4.3$

Hz), 32.00 (br), 32.66 (dd, $J=5.7, 3.1$ Hz), 37.48 (s at 25 °C, d with $J=1.8$ Hz at -80 °C), 38.86, 67.92 (d, $J=7.0$ Hz), 70.20 (d, $J=7.8$ Hz), 70.42, 75.28 (d, $J=4.7$ Hz), 78.00 (dd, $J=41.1, 8.7$ Hz), 95.61 (dd, $J=18.2, 6.3$ Hz), 121.07, 124.51 (d, $J=7.5$ Hz), 127.38 (d, $J=12.1$ Hz), 127.65 (d, $J=7.0$ Hz), 128.57 (d, $J=11.6$ Hz), 128.74 (d, $J=1.3$ Hz), 131.33 (d, $J=2.0$ Hz), 131.80 (d, $J=51.9$ Hz), 132.49 (d, $J=64.1$ Hz), 132.61 (d, $J=7.8$ Hz), 133.33 (d, $J=6.0$ Hz), 136.23 (d, $J=14.0$ Hz), 139.56, 160.44 (d, $J=113.1$ Hz). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2) 21.2 (d, $J=38.0$ Hz), 67.6 (d, $J=38.0$ Hz). Anal. calc'd for $\text{C}_{38}\text{H}_{45}\text{P}_2\text{BrFePd}$: C, 56.63; H, 5.64. Found: C, 56.59; H, 5.49.

Independent Synthesis of $\text{Pd}(\text{CyPF-}t\text{-Bu})(\text{Ph})(\text{Br})$ (6). A solution of 157 mg (0.28 mmol) $\text{CyPF-}t\text{-Bu}$ in THF was added dropwise to a suspension of 150 mg (0.13 mmol) $\{\text{Pd}[\text{P}(o\text{-tolyl})_3](\text{Ph})(\mu\text{-Br})\}_2$ in THF. The reaction mixture was stirred at room temperature for 20 min during which time all the solids dissolved. After 20 min the reaction was complete, as determined by $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy. The orange solution was filtered through Celite and concentrated. Addition of pentane led to precipitation of the product as an orange solid. The solid was filtered, washed with cold pentane, and dried under vacuum to give 126 mg (58% yield) of the product. ^1H NMR (C_6D_6) 0.80 – 2.86 (m, 22 Cy H), 1.32 (d, $J=12.8$ Hz, 9H), 1.61 (t, $J=7.2$ Hz, 3H), 1.67 (d, $J=11.6$ Hz, 9H), 2.19 (s, 1H, 1/3 molecule of toluene), 3.10 (m, 1H), 3.98 (s, 5H), 4.01 (m, 1H), 4.04 (br s, 1H), 4.64 (br s, 1H), 7.01-7.06 (m, 2H), 7.10-7.15 (m, C_6D_6 and 1/3 molecule of toluene), 7.35 (br, 1H), 7.84 (br, 2H). $^{31}\text{P}\{^1\text{H}\}$ NMR (C_7H_8 , -20 °C) 16.0 (d, $J=34.7$ Hz), 72.0 (d, $J=34.7$ Hz). Anal. calc'd for $\text{C}_{38}\text{H}_{57}\text{P}_2\text{BrFePd} \cdot (1/3)\text{C}_7\text{H}_8$: C, 57.1; H, 7.05. Found: C, 57.32; H, 7.19.

Synthesis of $\text{Pd}(\text{PPF-}t\text{-Bu})\text{Cl}_2$. A suspension of PdCl_2 (150 mg, 0.85 mmol) in 15 mL of CH_3CN was stirred at 80 °C until all solid dissolved. The warm solution was poured into a vial containing $\text{PPF-}t\text{-Bu}$ (490 mg, 0.903 mmol), and a red precipitate formed immediately. The suspension was allowed to cool to room temperature, and the red solid was isolated by filtration. The product was washed with pentane and dried under vacuum to produce $\text{Pd}(\text{PPF-}t\text{-Bu})\text{Cl}_2$ (544 mg, 85% yield) as a red, air-stable solid. ^1H NMR (CDCl_3) 1.42 (d, $J=15.2$ Hz, 9H), 1.59 (d, $J=13.6$ Hz, 9H), 2.07 (t, $J=8.8$ Hz, 3H), 3.17 (m, 1H), 3.86 (s, 1H), 3.95 (s, 5H), 4.40 (s, 1H), 4.60 (s, 1H), 7.30-7.40 (m, 4H), 7.59 (br s, 4H), 8.47-8.52 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3) 17.95 (d, $J=5.1$ Hz), 31.33 (d, 2.1 Hz), 31.61 (d, 2.9 Hz), 33.81 (br t, $J=9.8$ Hz), 40.45 (d, $J=11.0$ Hz), 41.59 (d, $J=8.7$ Hz), 69.09 (d, $J=11.1$ Hz), 69.65 (d, $J=7.7$ Hz), 70.50, 74.18 (d, $J=5.6$ Hz), 127.53 (d, $J=12.6$ Hz), 128.35 (d, $J=11.6$ Hz), 129.85 (d, $J=55.7$ Hz), 130.46 (d, $J=3.5$ Hz), 131.71 (d, $J=2.6$ Hz), 132.19 (d, $J=71.8$ Hz), 134.02 (d, $J=9.7$ Hz), 135.46 (d, $J=12.0$ Hz). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2) 24.3 (s), 112.2 (s). Anal. calc'd for $\text{C}_{32}\text{H}_{40}\text{P}_2\text{Cl}_2\text{FePd}$: C, 53.40; H, 5.61. Found: C, 53.19; H, 5.65.

General Procedure for the Oxidative Addition of PhOTs to 1 and 2. Into a small vial was placed 10.0 mg (0.0140 mmol) of $\text{Pd}[\text{P}(o\text{-tol})_3]_2$, 7.8 mg (0.0140 mol) of $\text{CyPF-}t\text{-Bu}$, and 5.4 mg of PMes_3 as internal standard. The solid materials were dissolved in 0.60 mL C_6H_6 , and the solution was transferred to an NMR tube. A $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum was obtained. The solution was poured into a vial containing 17.3 mg (0.0697 mmol) of PhOTs, and the resulting solution was stirred at room temperature. After 30 min, a second $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum was obtained, and this spectrum showed all starting material had been consumed and that compound **4** was produced in 92% yield.

Measurement of Rate Constant for Oxidative Addition of Aryl Tosylate to Palladium(0):

Representative Procedure. Into a small vial was placed 12.0 mg (0.0170 mmol) of $\text{Pd}[\text{P}(o\text{-tolyl})_3]_2$, 9.2 mg (0.017 mmol) of $\text{PPF-}t\text{-Bu}$, 34.0 mg (0.140 mmol) of PhOTs, 92.0 mg (0.170 mmol) of $\text{N}(\text{octyl})_4\text{Br}$, 26.0 mg (0.0850 mmol) of $\text{P}(o\text{-tolyl})_3$, and PMes_3 (as internal standard). The solids were dissolved in 0.60 mL THF, and the solution was transferred to an NMR tube with a septum-lined, screw cap. The sample was heated at 55 °C, and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra were obtained every 3 min for at least 5 half-lives by an automated program.

Kinetic data:

Figure S1. $[\text{Pd}(0)]$ versus time for the oxidative addition of PhOTs to **1**.

Table S1. Dependence of k_{obs} on $[\text{Pd}]_0$.

$[\text{Pd}]_0$ (M)	k_{obs} (s^{-1}) $\times 10^4$
0.0280	3.61
0.111	3.49

Figure S2. Dependence of k_{obs} on $[\text{ArOTs}]$.Table S2. Dependence of k_{obs} on $[\text{ArOTs}]$.

$[\text{ArOTs}]$	k_{obs} (s^{-1}) $\times 10^4$
0.0651	1.58
0.0946	2.74
0.148	4.78
0.226	7.50
0.280	9.17
0.331	12.6
0.414	15.0
0.447	16.2

Figure S3. Dependence of k_{obs} on $[P(o\text{-tol})_3]$.

Table S3. Dependence of k_{obs} on $[P(o\text{-tol})_3]$.

$[P(o\text{-tol})_3]$ (M)	k_{obs} (s^{-1}) $\times 10^4$	$1/k_{\text{obs}}$ (s) $\times 10^{-3}$
0.0882	9.38	1.07
0.110	7.03	1.42
0.142	5.06	1.98
0.275	2.79	3.58

Figure S4. Dependence of k_{obs} on $[\text{N(octyl)}_4\text{Br}]$.

Table S4. Dependence of k_{obs} on $[\text{N(octyl)}_4\text{Br}]$.

$[\text{N(octyl)}_4\text{Br}] \text{ (M)}$	$k_{\text{obs}} \text{ (s}^{-1}\text{)} \times 10^4$
0	4.11
0.0329	4.63
0.0628	5.83
0.165	7.50
0.244	10.1
0.277	11.3
0.335	12.6

Figure S5. Dependence of k_{obs} on $[\text{NBu}_4\text{PF}_6]$.

Table S5. Dependence of k_{obs} on $[\text{NBu}_4\text{PF}_6]$.

$[\text{NBu}_4\text{PF}_6] \text{ (M)}$	$k_{\text{obs}} \text{ (s}^{-1}\text{)} \times 10^4$
0	4.11
0.0116	4.41
0.0426	6.81
0.0865	13.2
0.140	18.8
0.239	27.9

General Procedure for the Reaction of Aryl Tosylates with Arylmagnesium Bromides. The reaction conditions and average yields for each reaction are shown in Table 1. A typical procedure is given for the first entry in Table 1.

4-Methoxy-4'-methylbiphenyl.⁴ Into a small vial was placed 5.8 mg (0.010 mmol) of Pd(dba)₂, 5.4 mg (0.010 mmol) of PPF-*t*-Bu, and 278.3 mg (1.0 mmol) of 4-MeOC₆H₄OTs. The solids were dissolved in 1.5 mL of toluene. To the vial, 2.00 mL (2.0 mmol) of a 1.0 M solution of *p*-tolylMgBr in ether were added by syringe. The vial was sealed with a cap containing a PTFE septum, and the mixture was stirred at room temperature until the aryl tosylate was consumed, as determined by GC. The mixture was hydrolyzed with 1.0 M HCl, and the product was extracted with diethyl ether. The organic extracts were washed with saturated sodium bicarbonate and brine and then dried over MgSO₄. After filtration and evaporation of the solvent, the residue was purified by chromatography on silica gel (hexane/ethyl acetate = 99/1) to give 174.5 mg (88% yield) of 4-methoxy-4'-methylbiphenyl. ¹H NMR (C₆D₆) 2.17 (s, 3H), 3.33 (s, 3H), 6.85 (m, 2H), 7.07 (d, J=4.0 Hz, 2H), 7.44 (m, 4H). ¹³C NMR (CDCl₃) 21.01, 55.26, 114.13, 126.54, 127.90, 129.40, 133.70, 136.28, 137.93, 158.91.

4-Methoxy-2'-methylbiphenyl.⁵ 4-MeOC₆H₄OTs (282 mg, 1.01 mmol), *o*-tolylMgBr (1.0 mL of a 2M solution in ether, 2.00 mmol), Pd(dba)₂ (5.8 mg, 0.010 mmol), and PPF-*t*-Bu (5.4 mg, 0.010 mmol) were used. The crude product was purified by chromatography on silica gel (hexane/ethyl acetate = 99/1) to give 147 mg (73% yield) of 4-methoxy-2'-methylbiphenyl. ¹H NMR (CD₂Cl₂) 2.26 (s, 3H), 3.84 (s, 3H), 6.94-6.96 (m, 2H), 7.20-7.26 (m, 6H). ¹³C NMR (CD₂Cl₂) 20.70, 55.61, 113.85, 126.12, 127.33, 130.19, 130.63, 130.66, 134.70, 135.86, 141.97, 159.05.

4-Methyl-4'-trifluoromethylbiphenyl.⁶ 4-CF₃C₆H₄OTs (316 mg, 1.00 mmol), *p*-tolylMgBr (2.0 mL of a 1.0 M solution in ether, 2.00 mmol), Pd(dba)₂ (5.8 mg, 0.010 mmol), and PPF-*t*-Bu (5.4 mg, 0.010 mmol) were used. The crude product was purified by chromatography on silica gel (hexane/ethyl acetate = 99/1) to give 154 mg (65% yield) of 4-methyl-4'-trifluoromethylbiphenyl. ¹H NMR (CD₂Cl₂) 2.41 (s, 3H), 7.30 (dd, J=8.5 Hz, 1.0 Hz, 2H), 7.53 (dd, J=6.3 Hz, 1.5 Hz, 2H), 7.68-7.73 (m, 4H). ¹³C NMR (CD₂Cl₂) 21.63, 124.89 (q, J=272 Hz, CF₃), 125.98 (q, J=3.8 Hz), 127.36, 127.50, 129.08 (q, J=32.0 Hz), 130.05, 136.98, 138.73, 145.03.

1-*p*-Tolyl naphthalene.⁷ Toluene-4-sulfonic acid-[1]naphthyl ester (298 mg, 1.00 mmol), *p*-tolylMgBr (2.0 mL of a 1.0 M solution in ether, 2.00 mmol), Pd(dba)₂ (5.8 mg, 0.010 mmol), and PPF-*t*-Bu (5.4 mg, 0.010 mmol) were used. The crude product was purified by chromatography

on silica gel (hexanes). The residue was dissolved in ether, layered with pentane, and recrystallized at -35 °C to give 171 mg (78% yield) of 1-*p*-tolynaphthalene. ¹H NMR (CD₂Cl₂) 2.46 (s, 3H), 7.32-7.55 (m, 8H), 7.86 (d, 6.5 Hz, 1H), 7.92 (d, 4.1 Hz, 2H). ¹³C NMR (CD₂Cl₂) 21.55, 126.00, 126.30, 126.53, 126.55, 127.19, 127.47, 127.96, 128.81, 129.55, 130.49, 132.28, 134.48, 137.65, 140.40.

4-Fluoro-4'-methylbiphenyl.⁸ 4-MeC₆H₄OTs (262 mg, 1.00 mmol), *p*-FC₆H₄MgBr (1.0 mL of a 2.0 M solution in tetrahydrofuran, 2.00 mmol), Pd(dba)₂ and PPF-*t*-Bu (100 µL of a 0.010 M solution of precatalyst and ligand, 0.0010 mmol) were used. The residue was dissolved in ether, layered with pentane, and recrystallized at -35 °C to give 125 mg (67% yield) of 4-fluoro-4'-methylbiphenyl. ¹H NMR (CD₂Cl₂) 2.38 (s, 3H), 7.10-7.15 (m, 2H), 7.25 (d, *J*=4.0 Hz, 2H), 7.45 (d, *J*=3.2 Hz, 2H), 7.51-7.57 (m, 2H). ¹³C NMR (CD₂Cl₂) 21.35, 115.97 (d, *J*=20.9 Hz), 127.29, 128.99 (d, *J*=7.5 Hz), 130.08, 137.74, 137.78, 137.86 (d, *J*=4.4 Hz), 162.87 (d, *J*=245.3 Hz).

2-Methoxy-4'-methylbiphenyl.⁹ 2-MeOC₆H₄OTs (278 mg, 1.00 mmol), *p*-tolylMgBr (2.0 mL, 1.0 M solution in ether, 2.00 mmol), Pd(dba)₂ (5.8 mg, 0.010 mmol), and PPF-*t*-Bu (5.4 mg, 0.010 mmol) were used. The crude product was purified by chromatography on silica gel (hexane/ethyl acetate = 99/1) to give 90 mg (45% yield) of 2-methoxy-4'-methylbiphenyl. ¹H NMR (CD₂Cl₂) 2.38 (s, 3H), 3.80 (s, 3H), 6.98-7.02 (m, 2H), 7.21 (d, *J*=4.0 Hz, 2H), 7.27-7.32 (m, 2H), 7.38-7.40 (m, 2H). ¹³C NMR (CD₂Cl₂) 20.22, 55.85, 111.22, 121.00, 129.21, 130.12, 130.58, 131.36, 131.46, 137.49, 139.47, 157.23.

2-Methoxy-2'-methylbiphenyl.⁵ 2-MeOC₆H₄OTs (278 mg, 1.00 mmol), *o*-tolylMgBr (1.0 mL, 2.0 M solution in ether, 2.00 mmol), Pd(dba)₂ and PPF-*t*-Bu (100 µL of a 0.010 M solution of precatalyst and ligand, 0.0010 mmol) were used. The crude product was purified by column chromatography on silica gel (hexanes to elute 2,2'-dimethylbiphenyl, then 95/5 hexane/ethyl acetate to elute 2-methoxy-2'-methylbiphenyl) to give 112 mg (56% yield) of 2-methoxy-2'-methylbiphenyl. ¹H NMR (CD₂Cl₂) 2.11 (s, 3H), 3.76 (s, 3H), 6.98-7.03 (m, 2H), 7.11-7.15 (m, 2H), 7.19-7.25 (m, 3H), 7.33-7.39 (m, 1H). ¹³C NMR (CD₂Cl₂) 20.02, 55.65, 110.01, 120.80, 125.78, 127.59, 129.00, 129.92, 130.37, 131.16, 131.25, 137.29, 139.27, 157.03.

2-Methyl-2',4',6'-trimethylbiphenyl.⁸ MesOTs (290 mg, 1.00 mmol), *p*-tolylMgBr (2.0 mL of a 1.0 M solution, 2.00 mmol), Pd(dba)₂ (5.8 mg, 0.010 mmol), and PPF-*t*-Bu (5.4 mg, 0.010 mmol) were used. The crude product was purified by column chromatography on silica gel (hexanes), then recrystallization from pentane at -35 °C provided 84 mg (40% yield) of 2-methyl-2',4',6'-trimethylbiphenyl. ¹H NMR (CDCl₃) 1.99 (s, 6H), 2.30 (s, 3H), 2.38 (s, 3H),

6.91 (s, 2H), 7.00 (d, $J=4.0$ Hz, 2H), 7.20 (d, $J=4.0$ Hz, 2H). ^{13}C NMR (CD_2Cl_2) 20.88, 21.09, 21.30, 128.28, 129.36, 129.50 (2 overlapping C), 129.59, 129.78, 129.97, 136.22, 136.40, 136.64.

***N*-octylaniline.**¹⁰ Into a 20 mL vial was placed 5.8 mg (0.010 mmol) $\text{PdCl}_2(\text{PPF-}t\text{-Bu})$, 202 mg (0.81 mmol) of PhOTs, and 200 mg (2.08 mmol) of $\text{NaO}(t\text{-Bu})$. The solids were dissolved in 15 mL of toluene, and 160 μL (0.97 mmol) of octylamine were added by syringe into the vial. The mixture was stirred at room temperature until the aryl tosylate was consumed, as determined by GC, and then diluted with EtOAc. After filtration and evaporation of the solvent, the residue was purified by chromatography on silica gel (hexane/ethyl acetate = 95/5) to give 128 mg (76% yield) of *N*-octylaniline. ^1H NMR (CD_2Cl_2) 0.87-0.91 (m, 3H), 1.29-1.41 (m, 10H), 1.59 (quintet, $J=7.6$ Hz, 2H), 3.08 (t, $J=7.2$ Hz, 2H), 3.66 (br s, 1H), 6.57-6.65 (m, 3H), 7.11-7.15 (m, 2H). ^{13}C NMR (CD_2Cl_2) 14.54, 23.34, 27.84, 29.95, 30.10, 30.22, 32.52, 44.57, 113.13, 117.38, 129.72, 149.42.

References:

- 1)Paul, F.; Patt, J.; Hartwig, J. F. *Organometallics* **1995**, *14*, 3030.
- 2)Komiyama, S. ; John Wiley & Sons: New York, 1997, pp 290.
- 3)Burckhardt, U.; Gramlich, V.; Hofmann, P.; Nesper, R.; Pregosin, P. S.; Salzmänn, R.; Togni, A. *Organometallics* **1996**, *15*, 3496.
- 4)Farina, V.; Krishnan, B.; Marshall, D. R.; Roth, G. P. *J. Org. Chem.* **1993**, *58*, 5434.
- 5)Lipshutz, B. H.; Siegmänn, K.; Garcia, E.; Kayser, F. *J. Am. Chem. Soc.* **1993**, *115*, 9276.
- 6)Gouda, K.; Hagiwara, E.; Hatanaka, Y.; Hiyama, T. *J. Org. Chem.* **1996**, 7232.
- 7)Leznoff, C. C.; Hayward, R. J. *Can. J. Chem.* **1970**, *48*, 1842.
- 8)Bell, H. C.; Kalman, J. R.; May, G. L.; Pinhey, J. T.; Sternhell, S. *Aust. J. Chem.* **1979**, *32*, 1531.
- 9)Wolfe, J. P.; Singer, R. A.; Yang, B. H.; Buchwald, S. L. *J. Am. Chem. Soc.* **1999**, *121*, 9550.
- 10)Cho, C. S.; Kim, J. S.; Kim, H. S.; Kim, T. J.; Shim, S. C. *Synth. Commun.* **2001**, *31*, 3791.

Crystallographic data for **5**.

Figure S6. X-ray crystal structure of **5**.

Data Collection

An orange needle crystal of $C_{42}H_{53}OP_2BrFePd$ having approximate dimensions of 0.10 x 0.15 x 0.29 mm was mounted on a glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-K α radiation.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using ten (1° in ω , 10s exposure, de-zingered) data frames, corresponded to a primitive monoclinic cell with dimensions: $a = 9.4928(2)$ Å, $b = 15.1888(5)$ Å, $c = 13.5344(4)$ Å.

$\text{\AA}$, $= 91.7135^\circ$ and $V = 1950.57(8) \text{\AA}^3$. For $Z = 2$ and F.W. = 877.98, the calculated density is 1.50 g/cm^3 . Based on the systematic absences of: $0k0: k = 2n+1$; packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be: $P2_1$ (#4). In this acentric space group, the enantiomer was selected on the basis of refinement of the flack parameter and on the least-squares refinement R values (the R values of the opposite hand were r, rw: 6.37, 7.46%).

The data were collected at a temperature of $-90 \pm 1^\circ\text{C}$ to a maximum 2θ value of 61.2° . One phi scan consisting of 140 data frames and two omega scans consisting of 44 and 32 data frames, respectively, were collected with a scan width of 1° and a detector-to-crystal distance, Dx, of 35mm. Each frame was exposed twice (for the purpose of de-zingering) for 20s. The data frames were processed and scaled using the DENZO software package. (Z. Otwinowski and W. Minor, "Processing of X-Ray Diffraction Data Collected in Oscillation Mode," Methods in Enzymology, vol. 276: Macromolecular Crystallography, part A, 307-326, 1997, C.W. Carter, Jr. & R.M. Sweet, Eds., Academic Press).

Data Reduction

Of the 13797 reflections which were collected, 5823 were unique ($R_{\text{int}} = 0.061$); equivalent reflections were merged. No decay correction was applied. The linear absorption coefficient, μ , for Mo-K radiation is 19.7 cm^{-1} and a SORTAV absorption correction was applied (MaXus: Mackay, S., Edwards, C., Henderson, A., Gilmore, C., Stewart, N., Shankland, K. and Donald, A. (1998) Chemistry Department, The University of Glasgow, Scotland.) The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = $2.82590\text{e-}07$).

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement³ was based on 4363 observed reflections ($I > 3.00F(I)$) and 432 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \frac{||F_o| - |F_c||}{|F_o|} = 0.042$$

$$R_w = [\sum w (|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.041$$

The standard deviation of an observation of unit weight⁴ was 1.37. The weighting scheme was based on counting statistics and included a factor ($p = 0.020$) to downweight the intense reflections. Plots of $\sum w (|F_o| - |F_c|)^2$ versus $|F_o|$, reflection order in data collection, $\sin \theta / \lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.70 and -1.34 e-/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for f' and f'' were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

- (1) ORIENT: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least-Squares:

Function minimized $\sum w(|F_o| - |F_c|)^2$

where $w = 4F_o^2 / 2(F_o^2)$

and $2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2] / Lp^2$

S = Scan rate

C = Total integrated peak count

R = Ratio of scan time to background counting time

B = Total background count
 L_p = Lorentz-polarization factor
 p = p-factor

(4) Standard deviation of an observation of unit weight:

$$[w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

where N_o = number of observations

N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992)

EXPERIMENTAL DETAILS FOR 5

A. Crystal Data

Empirical Formula	$\text{C}_{42}\text{H}_{53}\text{OP}_2\text{BrFePd}$
Formula Weight	877.98
Crystal Color, Habit	orange, needle
Crystal Dimensions	0.10 X 0.15 X 0.29 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	$a = 9.4928(2) \text{ \AA}$ $b = 15.1888(5) \text{ \AA}$ $c = 13.5344(4) \text{ \AA}$ $\beta = 91.7135^\circ$ $V = 1950.57(8) \text{ \AA}^3$
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.495 g/cm^3
F_{000}	900.00
$\mu(\text{MoK}\alpha)$	19.74 cm^{-1}

B. Intensity Measurements

Diffractometer	Nonius KappaCCD
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated
Take-off Angle	2.8°
Crystal to Detector Distance	35 mm
Temperature	-90.0°C
Scan Rate	20s/frame
Scan Width	1°/frame
$2\theta_{\text{max}}$	61.2°
No. of Reflections Measured	Total: 13797 Unique: 5823 (R_{int} = 0.061)
Corrections	Lorentz-polarization Absorption (SORTAV) Secondary Extinction (coefficient: 2.82590e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares
Function Minimized	$w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o)$
p-factor	0.0200
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00F(I)$)	4363
No. Variables	432
Reflection/Parameter Ratio	10.10
Residuals: R; R _w	0.042 ; 0.041
Goodness of Fit Indicator	1.37
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	$0.70 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-1.34 \text{ e}^-/\text{\AA}^3$

Table S6. Atomic coordinates and B_{iso}/B_{eq} for **5**.

atom	x	y	z	B _{eq}
Pd(1)	-0.70967(4)	-0.3340	-0.66182(3)	1.560(8)
Br(1)	-0.47271(6)	-0.34817(6)	-0.57262(4)	2.79(1)
Fe(1)	-1.13095(9)	-0.15114(7)	-0.82300(7)	1.90(2)
P(1)	-0.8636(1)	-0.3176(1)	-0.78920(10)	1.43(3)
P(2)	-0.8206(2)	-0.2302(1)	-0.5511(1)	1.64(3)
O(1)	-0.8012(8)	0.1275(6)	-0.9032(8)	8.8(3)
C(1)	-0.6591(6)	-0.4483(4)	-0.7323(4)	1.9(1)
C(2)	-0.5779(6)	-0.4615(5)	-0.8146(5)	2.3(1)
C(3)	-0.5545(6)	-0.5456(5)	-0.8500(5)	2.7(1)
C(4)	-0.6112(7)	-0.6183(5)	-0.8035(5)	2.7(1)
C(5)	-0.6903(7)	-0.6056(5)	-0.7194(5)	2.7(1)
C(6)	-0.7127(6)	-0.5214(5)	-0.6869(5)	2.4(1)
C(7)	-0.7867(5)	-0.3118(4)	-0.9119(4)	1.7(1)
C(8)	-0.6571(6)	-0.2694(5)	-0.9197(5)	2.3(1)
C(9)	-0.6071(6)	-0.2512(5)	-1.0124(5)	2.7(1)
C(10)	-0.6841(7)	-0.2764(6)	-1.0968(5)	3.2(2)
C(11)	-0.8113(7)	-0.3179(6)	-1.0886(4)	3.0(2)
C(12)	-0.8654(5)	-0.3345(6)	-0.9965(4)	2.2(1)
C(13)	-0.9969(6)	-0.4038(4)	-0.7997(4)	1.7(1)
C(14)	-0.9638(6)	-0.4829(5)	-0.8472(5)	2.1(1)
C(15)	-1.0606(7)	-0.5507(5)	-0.8516(5)	2.6(1)
C(16)	-1.1881(7)	-0.5423(5)	-0.8076(5)	2.5(1)
C(17)	-1.2196(6)	-0.4671(5)	-0.7564(5)	2.4(1)
C(18)	-1.1246(6)	-0.3968(5)	-0.7519(4)	1.9(1)
C(19)	-0.9400(5)	-0.2077(4)	-0.7889(4)	1.4(1)
C(20)	-0.9285(6)	-0.1440(5)	-0.8660(4)	2.3(1)
C(21)	-0.9710(6)	-0.0615(5)	-0.8299(5)	2.5(1)
C(22)	-1.0111(6)	-0.0720(5)	-0.7307(5)	2.5(1)
C(23)	-0.9889(5)	-0.1618(4)	-0.7030(4)	1.6(1)
C(24)	-1.3226(6)	-0.2054(6)	-0.7951(6)	3.2(2)
C(25)	-1.2694(7)	-0.2395(5)	-0.8844(5)	3.0(2)
C(26)	-1.2509(7)	-0.1686(6)	-0.9501(5)	3.1(2)

Table S6. Atomic coordinates and B_{iso}/B_{eq} for **5** (continued)

atom	x	y	z	B _{eq}
C(27)	-1.2900(7)	-0.0891(6)	-0.9014(6)	3.5(2)
C(28)	-1.3365(6)	-0.1133(6)	-0.8062(6)	3.4(2)
C(29)	-1.0030(6)	-0.2033(5)	-0.6036(4)	2.0(1)
C(30)	-1.1137(7)	-0.1543(6)	-0.5424(5)	2.9(1)
C(31)	-0.8620(7)	-0.2869(5)	-0.4318(4)	2.3(1)
C(32)	-0.7340(7)	-0.3404(6)	-0.3921(4)	3.1(1)
C(33)	-0.9786(7)	-0.3556(6)	-0.4561(4)	3.2(2)
C(34)	-0.9097(8)	-0.2267(6)	-0.3481(5)	3.2(2)
C(35)	-0.7189(6)	-0.1251(4)	-0.5287(5)	2.2(1)
C(36)	-0.6508(7)	-0.1033(5)	-0.6268(5)	2.9(2)
C(37)	-0.8074(7)	-0.0464(5)	-0.4989(5)	2.6(1)
C(38)	-0.5981(7)	-0.1376(5)	-0.4511(5)	3.0(2)
C(40)	-0.791(1)	0.148(1)	-0.801(1)	10.7(5)
C(41)	-0.644(2)	0.122(1)	-0.768(1)	9.6(5)
C(42)	-0.6103(10)	0.0545(9)	-0.8455(10)	6.3(3)
C(43)	-0.692(1)	0.079(1)	-0.9312(8)	7.4(4)
H(1)	-0.5379	-0.4123	-0.8469	2.7806
H(2)	-0.4991	-0.5536	-0.9067	3.1831
H(3)	-0.5963	-0.6758	-0.8285	3.2465
H(4)	-0.7280	-0.6545	-0.6853	3.2688
H(5)	-0.7679	-0.5131	-0.6302	2.9095
H(6)	-0.6039	-0.2533	-0.8619	2.7150
H(7)	-0.5199	-0.2213	-1.0185	3.2522
H(8)	-0.6484	-0.2648	-1.1604	3.8276
H(9)	-0.8628	-0.3355	-1.1465	3.6418
H(10)	-0.9554	-0.3611	-0.9912	2.5980
H(11)	-0.8746	-0.4899	-0.8763	2.5574
H(12)	-1.0387	-0.6035	-0.8855	3.1049
H(13)	-1.2551	-0.5887	-0.8126	2.9874
H(14)	-1.3066	-0.4629	-0.7237	2.8469
H(15)	-1.1467	-0.3448	-0.7167	2.3329
H(16)	-0.8973	-0.1554	-0.9308	2.7224

Table S6. Atomic coordinates and B_{iso}/B_{eq} for **5** (continued)

atom	x	y	z	B _{eq}
H(17)	-0.9725	-0.0079	-0.8660	3.0102
H(18)	-1.0467	-0.0270	-0.6896	3.0004
H(19)	-1.3448	-0.2386	-0.7381	3.8296
H(20)	-1.2496	-0.2996	-0.8975	3.6361
H(21)	-1.2180	-0.1729	-1.0155	3.7129
H(22)	-1.2859	-0.0312	-0.9276	4.1909
H(23)	-1.3711	-0.0740	-0.7580	4.0198
H(24)	-1.0445	-0.2591	-0.6172	2.3488
H(25)	-1.0804	-0.0969	-0.5268	3.4853
H(26)	-1.1291	-0.1858	-0.4831	3.4853
H(27)	-1.1998	-0.1502	-0.5799	3.4853
H(28)	-0.7059	-0.3810	-0.4412	3.7110
H(29)	-0.7592	-0.3716	-0.3344	3.7110
H(30)	-0.6583	-0.3015	-0.3764	3.7110
H(31)	-1.0621	-0.3262	-0.4780	3.8230
H(32)	-0.9972	-0.3889	-0.3985	3.8230
H(33)	-0.9481	-0.3939	-0.5066	3.8230
H(34)	-0.8361	-0.1867	-0.3306	3.8831
H(35)	-0.9317	-0.2614	-0.2922	3.8831
H(36)	-0.9909	-0.1946	-0.3696	3.8831
H(37)	-0.5924	-0.1509	-0.6456	3.4351
H(38)	-0.5955	-0.0515	-0.6193	3.4351
H(39)	-0.7223	-0.0941	-0.6763	3.4351
H(40)	-0.7485	0.0039	-0.4910	3.1358
H(41)	-0.8512	-0.0587	-0.4382	3.1358
H(42)	-0.8776	-0.0352	-0.5487	3.1358
H(43)	-0.5409	-0.1858	-0.4697	3.6022
H(44)	-0.6365	-0.1491	-0.3884	3.6022
H(45)	-0.5426	-0.0856	-0.4474	3.6022
H(46)	-0.8586	0.1162	-0.7650	12.8082
H(47)	-0.8050	0.2097	-0.7907	12.8082
H(48)	-0.6415	0.0974	-0.7034	11.5774

Table S6. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ for **5** (continued)

atom	x	y	z	B_{eq}
H(49)	-0.5805	0.1708	-0.7694	11.5774
H(50)	-0.6357	-0.0027	-0.8239	7.6082
H(51)	-0.5128	0.0556	-0.8592	7.6082
H(52)	-0.6360	0.1115	-0.9747	8.8949
H(53)	-0.7261	0.0271	-0.9639	8.8949
$B_{\text{eq}} = 8/3 \left(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \quad + 2U_{13}(aa^*cc^*)\cos \quad + 2U_{23}(bb^*cc^*)\cos \right)$				

Table S7. Anisotropic Displacement Parameters for **5**

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Pd(1)	0.0191(2)	0.0238(2)	0.0162(2)	0.0025(2)	-0.0023(1)	-0.0011(2)
Br(1)	0.0278(3)	0.0442(5)	0.0333(3)	0.0100(3)	-0.0104(2)	-0.0066(3)
Fe(1)	0.0207(4)	0.0269(5)	0.0242(5)	0.0035(4)	-0.0048(3)	0.0021(4)
P(1)	0.0149(6)	0.0238(9)	0.0155(6)	0.0000(6)	0.0000(5)	0.0002(6)
P(2)	0.0252(7)	0.0207(8)	0.0163(7)	0.0008(6)	-0.0020(6)	-0.0010(6)
O(1)	0.062(5)	0.092(7)	0.181(9)	0.029(4)	0.018(5)	0.060(7)
C(1)	0.023(3)	0.030(4)	0.020(3)	0.006(3)	-0.003(2)	0.000(3)
C(2)	0.017(3)	0.036(4)	0.034(4)	0.004(3)	-0.004(2)	-0.005(3)
C(3)	0.026(3)	0.042(5)	0.032(3)	0.013(3)	-0.003(3)	-0.014(3)
C(4)	0.036(3)	0.034(4)	0.032(4)	0.008(3)	-0.008(3)	-0.013(3)
C(5)	0.042(4)	0.027(4)	0.034(4)	0.007(3)	-0.001(3)	-0.001(3)
C(6)	0.037(3)	0.027(4)	0.028(3)	0.002(3)	0.002(3)	-0.007(3)
C(7)	0.022(3)	0.028(4)	0.015(2)	0.000(2)	0.000(2)	-0.002(2)
C(8)	0.016(3)	0.041(4)	0.028(3)	0.001(3)	-0.005(2)	0.001(3)
C(9)	0.028(3)	0.044(5)	0.032(3)	-0.001(3)	0.006(3)	0.013(3)
C(10)	0.036(4)	0.060(5)	0.026(3)	0.000(4)	0.007(3)	0.007(4)
C(11)	0.044(3)	0.055(6)	0.016(3)	-0.002(4)	-0.004(2)	-0.001(3)
C(12)	0.030(3)	0.033(3)	0.019(2)	-0.006(3)	0.001(2)	-0.002(4)
C(13)	0.023(3)	0.024(3)	0.016(3)	0.000(2)	-0.003(2)	0.004(2)
C(14)	0.022(3)	0.030(4)	0.029(3)	0.001(3)	0.003(2)	-0.002(3)
C(15)	0.043(4)	0.022(3)	0.033(4)	0.002(3)	-0.004(3)	0.000(3)
C(16)	0.032(3)	0.031(4)	0.032(3)	-0.005(3)	-0.008(3)	0.008(3)
C(17)	0.019(3)	0.043(4)	0.028(3)	-0.009(3)	-0.002(2)	0.006(3)
C(18)	0.019(3)	0.034(4)	0.021(3)	-0.002(3)	-0.002(2)	0.003(3)
C(19)	0.015(2)	0.017(3)	0.022(3)	0.000(2)	-0.004(2)	0.005(2)
C(20)	0.022(3)	0.037(4)	0.026(3)	0.002(3)	0.002(2)	0.001(3)
C(21)	0.029(3)	0.028(4)	0.038(4)	-0.006(3)	-0.009(3)	0.008(3)
C(22)	0.030(3)	0.026(4)	0.039(4)	0.001(3)	-0.007(3)	0.001(3)
C(23)	0.021(3)	0.019(3)	0.022(3)	0.002(2)	-0.004(2)	-0.002(3)
C(24)	0.017(3)	0.055(5)	0.049(4)	-0.002(3)	-0.005(3)	0.011(4)
C(25)	0.030(3)	0.039(4)	0.045(4)	-0.003(3)	-0.019(3)	0.002(4)
C(26)	0.038(4)	0.052(5)	0.027(3)	-0.002(4)	-0.011(3)	-0.003(4)

Table S7. Anisotropic Displacement Parameters for **5** (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(27)	0.029(3)	0.057(5)	0.046(4)	0.004(3)	-0.020(3)	0.019(4)
C(28)	0.018(3)	0.058(6)	0.051(5)	0.014(3)	-0.002(3)	0.002(4)
C(29)	0.026(3)	0.027(3)	0.021(3)	0.000(3)	0.001(2)	-0.008(3)
C(30)	0.033(3)	0.045(5)	0.032(4)	0.014(3)	-0.001(3)	-0.007(3)
C(31)	0.039(3)	0.027(4)	0.020(3)	-0.005(3)	-0.003(3)	0.007(3)
C(32)	0.057(4)	0.036(4)	0.024(3)	0.008(4)	0.000(3)	0.014(4)
C(33)	0.052(4)	0.043(5)	0.027(3)	-0.015(4)	0.007(3)	0.003(3)
C(34)	0.057(5)	0.045(5)	0.021(3)	0.003(4)	-0.003(3)	0.002(3)
C(35)	0.031(3)	0.023(3)	0.029(3)	-0.005(3)	-0.007(2)	0.000(3)
C(36)	0.039(4)	0.028(4)	0.042(4)	-0.008(3)	-0.006(3)	0.008(3)
C(37)	0.038(4)	0.021(4)	0.039(4)	0.000(3)	-0.012(3)	-0.004(3)
C(38)	0.033(3)	0.038(4)	0.043(4)	-0.003(3)	-0.012(3)	-0.007(4)
C(40)	0.077(8)	0.09(1)	0.24(2)	-0.049(8)	0.08(1)	-0.09(1)
C(41)	0.17(1)	0.10(1)	0.096(10)	-0.03(1)	-0.014(9)	0.049(9)
C(42)	0.057(6)	0.078(8)	0.107(9)	0.024(5)	0.024(6)	0.041(7)
C(43)	0.054(6)	0.15(1)	0.082(8)	-0.006(7)	0.002(5)	0.050(8)

The general temperature factor expression:

$$\exp(-2 \text{ }^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$$

Table S8. Bond Lengths(Å)for **5**

atom	atom	distance	atom	atom	distance
Pd(1)	Br(1)	2.5301(6)	Pd(1)	P(1)	2.240(1)
Pd(1)	P(2)	2.436(2)	Pd(1)	C(1)	2.045(6)
Fe(1)	C(19)	2.046(5)	Fe(1)	C(20)	2.028(6)
Fe(1)	C(21)	2.043(6)	Fe(1)	C(22)	2.053(7)
Fe(1)	C(23)	2.086(5)	Fe(1)	C(24)	2.043(7)
Fe(1)	C(25)	2.038(7)	Fe(1)	C(26)	2.052(6)
Fe(1)	C(27)	2.049(7)	Fe(1)	C(28)	2.053(6)
P(1)	C(7)	1.837(5)	P(1)	C(13)	1.823(6)
P(1)	C(19)	1.821(6)	P(2)	C(29)	1.896(6)
P(2)	C(31)	1.882(6)	P(2)	C(35)	1.886(7)
O(1)	C(40)	1.42(2)	O(1)	C(43)	1.34(1)
C(1)	C(2)	1.387(9)	C(1)	C(6)	1.374(10)
C(2)	C(3)	1.385(10)	C(3)	C(4)	1.39(1)
C(4)	C(5)	1.395(9)	C(5)	C(6)	1.372(10)
C(7)	C(8)	1.394(8)	C(7)	C(12)	1.392(7)
C(8)	C(9)	1.383(9)	C(9)	C(10)	1.392(10)
C(10)	C(11)	1.369(9)	C(11)	C(12)	1.385(8)
C(13)	C(14)	1.403(9)	C(13)	C(18)	1.395(8)
C(14)	C(15)	1.380(9)	C(15)	C(16)	1.371(9)
C(16)	C(17)	1.37(1)	C(17)	C(18)	1.398(9)
C(19)	C(20)	1.429(9)	C(19)	C(23)	1.444(8)
C(20)	C(21)	1.41(1)	C(21)	C(22)	1.415(9)
C(22)	C(23)	1.429(9)	C(23)	C(29)	1.495(8)
C(24)	C(25)	1.42(1)	C(24)	C(28)	1.41(1)
C(25)	C(26)	1.41(1)	C(26)	C(27)	1.43(1)
C(27)	C(28)	1.42(1)	C(29)	C(30)	1.548(8)
C(31)	C(32)	1.544(9)	C(31)	C(33)	1.549(9)
C(31)	C(34)	1.536(10)	C(35)	C(36)	1.530(9)
C(35)	C(37)	1.523(10)	C(35)	C(38)	1.543(9)
C(40)	C(41)	1.51(2)	C(41)	C(42)	1.51(2)
C(42)	C(43)	1.42(1)			

Table S9. Bond Lengths for the Hydrogen Atoms (Å) for **5**

atom	atom	distance	atom	atom	distance
C(2)	H(1)	0.95	C(3)	H(2)	0.95
C(4)	H(3)	0.95	C(5)	H(4)	0.95
C(6)	H(5)	0.95	C(8)	H(6)	0.95
C(9)	H(7)	0.95	C(10)	H(8)	0.95
C(11)	H(9)	0.95	C(12)	H(10)	0.95
C(14)	H(11)	0.95	C(15)	H(12)	0.95
C(16)	H(13)	0.95	C(17)	H(14)	0.95
C(18)	H(15)	0.95	C(20)	H(16)	0.95
C(21)	H(17)	0.95	C(22)	H(18)	0.95
C(24)	H(19)	0.95	C(25)	H(20)	0.95
C(26)	H(21)	0.95	C(27)	H(22)	0.95
C(28)	H(23)	0.95	C(29)	H(24)	0.95
C(30)	H(25)	0.95	C(30)	H(26)	0.95
C(30)	H(27)	0.95	C(32)	H(28)	0.95
C(32)	H(29)	0.95	C(32)	H(30)	0.95
C(33)	H(31)	0.95	C(33)	H(32)	0.95
C(33)	H(33)	0.95	C(34)	H(34)	0.95
C(34)	H(35)	0.95	C(34)	H(36)	0.95
C(36)	H(37)	0.95	C(36)	H(38)	0.95
C(36)	H(39)	0.95	C(37)	H(40)	0.95
C(37)	H(41)	0.95	C(37)	H(42)	0.95
C(38)	H(43)	0.95	C(38)	H(44)	0.95
C(38)	H(45)	0.95	C(40)	H(46)	0.95
C(40)	H(47)	0.95	C(41)	H(48)	0.95
C(41)	H(49)	0.95	C(42)	H(50)	0.95
C(42)	H(51)	0.95	C(43)	H(52)	0.95
C(43)	H(53)	0.95			

Table S10. Bond Angles(°) for **5**

atom	atom	atom	angle	atom	atom	atom	angle
Br(1)	Pd(1)	P(1)	157.94(4)	Br(1)	Pd(1)	P(2)	98.97(4)
Br(1)	Pd(1)	C(1)	86.1(2)	P(1)	Pd(1)	P(2)	96.73(5)
P(1)	Pd(1)	C(1)	83.8(2)	P(2)	Pd(1)	C(1)	161.5(2)
C(19)	Fe(1)	C(20)	41.1(2)	C(19)	Fe(1)	C(21)	68.6(2)
C(19)	Fe(1)	C(22)	68.7(2)	C(19)	Fe(1)	C(23)	40.9(2)
C(19)	Fe(1)	C(24)	125.2(3)	C(19)	Fe(1)	C(25)	111.8(3)
C(19)	Fe(1)	C(26)	126.9(3)	C(19)	Fe(1)	C(27)	160.8(3)
C(19)	Fe(1)	C(28)	158.6(3)	C(20)	Fe(1)	C(21)	40.5(3)
C(20)	Fe(1)	C(22)	68.3(3)	C(20)	Fe(1)	C(23)	68.4(2)
C(20)	Fe(1)	C(24)	158.7(3)	C(20)	Fe(1)	C(25)	121.6(3)
C(20)	Fe(1)	C(26)	105.9(3)	C(20)	Fe(1)	C(27)	121.1(3)
C(20)	Fe(1)	C(28)	158.3(3)	C(21)	Fe(1)	C(22)	40.4(3)
C(21)	Fe(1)	C(23)	67.9(2)	C(21)	Fe(1)	C(24)	160.8(3)
C(21)	Fe(1)	C(25)	153.0(3)	C(21)	Fe(1)	C(26)	116.3(3)
C(21)	Fe(1)	C(27)	102.0(3)	C(21)	Fe(1)	C(28)	121.9(3)
C(22)	Fe(1)	C(23)	40.4(3)	C(22)	Fe(1)	C(24)	127.2(3)
C(22)	Fe(1)	C(25)	166.5(3)	C(22)	Fe(1)	C(26)	150.4(3)
C(22)	Fe(1)	C(27)	115.6(3)	C(22)	Fe(1)	C(28)	106.3(3)
C(23)	Fe(1)	C(24)	112.5(3)	C(23)	Fe(1)	C(25)	131.3(3)
C(23)	Fe(1)	C(26)	166.5(3)	C(23)	Fe(1)	C(27)	152.7(3)
C(23)	Fe(1)	C(28)	122.0(3)	C(24)	Fe(1)	C(25)	40.8(3)
C(24)	Fe(1)	C(26)	68.2(3)	C(24)	Fe(1)	C(27)	68.6(3)
C(24)	Fe(1)	C(28)	40.3(3)	C(25)	Fe(1)	C(26)	40.4(3)
C(25)	Fe(1)	C(27)	68.6(3)	C(25)	Fe(1)	C(28)	68.0(3)
C(26)	Fe(1)	C(27)	40.8(3)	C(26)	Fe(1)	C(28)	67.9(3)
C(27)	Fe(1)	C(28)	40.6(3)	Pd(1)	P(1)	C(7)	115.7(2)
Pd(1)	P(1)	C(13)	114.5(2)	Pd(1)	P(1)	C(19)	110.6(2)
C(7)	P(1)	C(13)	104.9(3)	C(7)	P(1)	C(19)	97.3(3)
C(13)	P(1)	C(19)	112.5(3)	Pd(1)	P(2)	C(29)	108.4(2)
Pd(1)	P(2)	C(31)	109.7(2)	Pd(1)	P(2)	C(35)	114.7(2)
C(29)	P(2)	C(31)	102.0(3)	C(29)	P(2)	C(35)	109.6(3)

Table S10. Bond Angles($^{\circ}$) for **5** (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(31)	P(2)	C(35)	111.6(3)	C(40)	O(1)	C(43)	111.7(10)
Pd(1)	C(1)	C(2)	129.8(5)	Pd(1)	C(1)	C(6)	112.4(4)
C(2)	C(1)	C(6)	117.7(6)	C(1)	C(2)	C(3)	120.6(7)
C(2)	C(3)	C(4)	120.5(6)	C(3)	C(4)	C(5)	119.1(7)
C(4)	C(5)	C(6)	118.8(7)	C(1)	C(6)	C(5)	123.1(6)
P(1)	C(7)	C(8)	117.8(4)	P(1)	C(7)	C(12)	120.9(4)
C(8)	C(7)	C(12)	120.4(5)	C(7)	C(8)	C(9)	119.2(6)
C(8)	C(9)	C(10)	120.3(6)	C(9)	C(10)	C(11)	120.1(6)
C(10)	C(11)	C(12)	120.6(6)	C(7)	C(12)	C(11)	119.3(5)
P(1)	C(13)	C(14)	119.1(4)	P(1)	C(13)	C(18)	121.3(5)
C(14)	C(13)	C(18)	119.2(6)	C(13)	C(14)	C(15)	120.0(6)
C(14)	C(15)	C(16)	120.5(6)	C(15)	C(16)	C(17)	120.3(6)
C(16)	C(17)	C(18)	120.4(6)	C(13)	C(18)	C(17)	119.4(6)
Fe(1)	C(19)	P(1)	137.3(3)	Fe(1)	C(19)	C(20)	68.8(3)
Fe(1)	C(19)	C(23)	71.1(3)	P(1)	C(19)	C(20)	125.4(5)
P(1)	C(19)	C(23)	125.6(4)	C(20)	C(19)	C(23)	107.2(5)
Fe(1)	C(20)	C(19)	70.1(3)	Fe(1)	C(20)	C(21)	70.4(3)
C(19)	C(20)	C(21)	108.6(5)	Fe(1)	C(21)	C(20)	69.2(4)
Fe(1)	C(21)	C(22)	70.1(4)	C(20)	C(21)	C(22)	108.5(6)
Fe(1)	C(22)	C(21)	69.5(4)	Fe(1)	C(22)	C(23)	71.1(4)
C(21)	C(22)	C(23)	108.4(6)	Fe(1)	C(23)	C(19)	68.0(3)
Fe(1)	C(23)	C(22)	68.5(3)	Fe(1)	C(23)	C(29)	131.3(4)
C(19)	C(23)	C(22)	107.3(5)	C(19)	C(23)	C(29)	124.1(6)
C(22)	C(23)	C(29)	128.5(6)	Fe(1)	C(24)	C(25)	69.4(4)
Fe(1)	C(24)	C(28)	70.2(4)	C(25)	C(24)	C(28)	107.7(7)
Fe(1)	C(25)	C(24)	69.8(4)	Fe(1)	C(25)	C(26)	70.3(4)
C(24)	C(25)	C(26)	108.2(7)	Fe(1)	C(26)	C(25)	69.3(4)
Fe(1)	C(26)	C(27)	69.5(4)	C(25)	C(26)	C(27)	108.4(6)
Fe(1)	C(27)	C(26)	69.7(4)	Fe(1)	C(27)	C(28)	69.8(4)
C(26)	C(27)	C(28)	106.9(7)	Fe(1)	C(28)	C(24)	69.4(4)
Fe(1)	C(28)	C(27)	69.6(4)	C(24)	C(28)	C(27)	108.8(7)

Table S10. Bond Angles($^{\circ}$) for **5** (continued)

atom	atom	atom	angle	atom	atom	atom	angle
P(2)	C(29)	C(23)	108.8(4)	P(2)	C(29)	C(30)	122.1(4)
C(23)	C(29)	C(30)	111.0(6)	P(2)	C(31)	C(32)	110.9(4)
P(2)	C(31)	C(33)	107.0(4)	P(2)	C(31)	C(34)	115.7(5)
C(32)	C(31)	C(33)	105.6(6)	C(32)	C(31)	C(34)	107.6(5)
C(33)	C(31)	C(34)	109.4(5)	P(2)	C(35)	C(36)	105.7(4)
P(2)	C(35)	C(37)	115.1(4)	P(2)	C(35)	C(38)	111.8(5)
C(36)	C(35)	C(37)	108.1(6)	C(36)	C(35)	C(38)	107.0(5)
C(37)	C(35)	C(38)	108.7(5)	O(1)	C(40)	C(41)	105(1)
C(40)	C(41)	C(42)	100(1)	C(41)	C(42)	C(43)	105(1)
O(1)	C(43)	C(42)	108(1)				

Table S11. Bond Angles for the Hydrogen Atoms (°) for **5**

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	C(2)	H(1)	119.7	C(3)	C(2)	H(1)	119.7
C(2)	C(3)	H(2)	119.7	C(4)	C(3)	H(2)	119.7
C(3)	C(4)	H(3)	120.4	C(5)	C(4)	H(3)	120.4
C(4)	C(5)	H(4)	120.6	C(6)	C(5)	H(4)	120.6
C(1)	C(6)	H(5)	118.4	C(5)	C(6)	H(5)	118.4
C(7)	C(8)	H(6)	120.4	C(9)	C(8)	H(6)	120.4
C(8)	C(9)	H(7)	119.9	C(10)	C(9)	H(7)	119.9
C(9)	C(10)	H(8)	119.9	C(11)	C(10)	H(8)	119.9
C(10)	C(11)	H(9)	119.7	C(12)	C(11)	H(9)	119.7
C(7)	C(12)	H(10)	120.3	C(11)	C(12)	H(10)	120.3
C(13)	C(14)	H(11)	120.0	C(15)	C(14)	H(11)	120.0
C(14)	C(15)	H(12)	119.7	C(16)	C(15)	H(12)	119.8
C(15)	C(16)	H(13)	119.8	C(17)	C(16)	H(13)	119.8
C(16)	C(17)	H(14)	119.8	C(18)	C(17)	H(14)	119.8
C(13)	C(18)	H(15)	120.3	C(17)	C(18)	H(15)	120.3
Fe(1)	C(20)	H(16)	125.4	C(19)	C(20)	H(16)	125.7
C(21)	C(20)	H(16)	125.7	Fe(1)	C(21)	H(17)	126.5
C(20)	C(21)	H(17)	125.8	C(22)	C(21)	H(17)	125.8
Fe(1)	C(22)	H(18)	125.2	C(21)	C(22)	H(18)	125.8
C(23)	C(22)	H(18)	125.8	Fe(1)	C(24)	H(19)	125.8
C(25)	C(24)	H(19)	126.1	C(28)	C(24)	H(19)	126.1
Fe(1)	C(25)	H(20)	125.5	C(24)	C(25)	H(20)	125.9
C(26)	C(25)	H(20)	125.9	Fe(1)	C(26)	H(21)	127.0
C(25)	C(26)	H(21)	125.8	C(27)	C(26)	H(21)	125.8
Fe(1)	C(27)	H(22)	125.5	C(26)	C(27)	H(22)	126.5
C(28)	C(27)	H(22)	126.5	Fe(1)	C(28)	H(23)	127.0
C(24)	C(28)	H(23)	125.6	C(27)	C(28)	H(23)	125.6
P(2)	C(29)	H(24)	104.4	C(23)	C(29)	H(24)	104.4
C(30)	C(29)	H(24)	104.4	C(29)	C(30)	H(25)	109.5
C(29)	C(30)	H(26)	109.5	C(29)	C(30)	H(27)	109.5
H(25)	C(30)	H(26)	109.5	H(25)	C(30)	H(27)	109.5

Table S11. Bond Angles for the Hydrogen Atoms (°) for **5** (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(26)	C(30)	H(27)	109.5	C(31)	C(32)	H(28)	109.5
C(31)	C(32)	H(29)	109.5	C(31)	C(32)	H(30)	109.5
H(28)	C(32)	H(29)	109.5	H(28)	C(32)	H(30)	109.5
H(29)	C(32)	H(30)	109.5	C(31)	C(33)	H(31)	109.5
C(31)	C(33)	H(32)	109.5	C(31)	C(33)	H(33)	109.5
H(31)	C(33)	H(32)	109.5	H(31)	C(33)	H(33)	109.5
H(32)	C(33)	H(33)	109.5	C(31)	C(34)	H(34)	109.5
C(31)	C(34)	H(35)	109.5	C(31)	C(34)	H(36)	109.5
H(34)	C(34)	H(35)	109.5	H(34)	C(34)	H(36)	109.5
H(35)	C(34)	H(36)	109.5	C(35)	C(36)	H(37)	109.5
C(35)	C(36)	H(38)	109.5	C(35)	C(36)	H(39)	109.5
H(37)	C(36)	H(38)	109.5	H(37)	C(36)	H(39)	109.5
H(38)	C(36)	H(39)	109.5	C(35)	C(37)	H(40)	109.5
C(35)	C(37)	H(41)	109.5	C(35)	C(37)	H(42)	109.5
H(40)	C(37)	H(41)	109.5	H(40)	C(37)	H(42)	109.5
H(41)	C(37)	H(42)	109.5	C(35)	C(38)	H(43)	109.5
C(35)	C(38)	H(44)	109.5	C(35)	C(38)	H(45)	109.5
H(43)	C(38)	H(44)	109.5	H(43)	C(38)	H(45)	109.5
H(44)	C(38)	H(45)	109.5	O(1)	C(40)	H(46)	110.5
O(1)	C(40)	H(47)	110.5	C(41)	C(40)	H(46)	110.5
C(41)	C(40)	H(47)	110.5	H(46)	C(40)	H(47)	109.5
C(40)	C(41)	H(48)	111.6	C(40)	C(41)	H(49)	111.6
C(42)	C(41)	H(48)	111.6	C(42)	C(41)	H(49)	111.6
H(48)	C(41)	H(49)	109.5	C(41)	C(42)	H(50)	110.4
C(41)	C(42)	H(51)	110.4	C(43)	C(42)	H(50)	110.4
C(43)	C(42)	H(51)	110.4	H(50)	C(42)	H(51)	109.5
O(1)	C(43)	H(52)	109.6	O(1)	C(43)	H(53)	109.7
C(42)	C(43)	H(52)	109.7	C(42)	C(43)	H(53)	109.7
H(52)	C(43)	H(53)	109.5				

Table S12. Torsion Angles(°) for **5**

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Pd(1)	P(1)	C(7)	C(8)	-35.6(6)	Pd(1)	P(1)	C(7)	C(12)	155.7(5)
Pd(1)	P(1)	C(13)	C(14)	-83.2(5)	Pd(1)	P(1)	C(13)	C(18)	89.4(5)
Pd(1)	P(1)	C(19)	Fe(1)	-144.2(4)	Pd(1)	P(1)	C(19)	C(20)	119.1(5)
Pd(1)	P(1)	C(19)	C(23)	-43.5(5)	Pd(1)	P(2)	C(29)	C(23)	-61.1(5)
Pd(1)	P(2)	C(29)	C(30)	167.6(5)	Pd(1)	P(2)	C(31)	C(32)	47.3(5)
Pd(1)	P(2)	C(31)	C(33)	-67.4(5)	Pd(1)	P(2)	C(31)	C(34)	170.3(4)
Pd(1)	P(2)	C(35)	C(36)	35.9(5)	Pd(1)	P(2)	C(35)	C(37)	155.1(4)
Pd(1)	P(2)	C(35)	C(38)	-80.3(5)	Pd(1)	C(1)	C(2)	C(3)	179.0(4)
Pd(1)	C(1)	C(6)	C(5)	-178.6(5)	Br(1)	Pd(1)	P(1)	C(7)	2.8(3)
Br(1)	Pd(1)	P(1)	C(13)	125.1(2)	Br(1)	Pd(1)	P(1)	C(19)	-106.5(2)
Br(1)	Pd(1)	P(2)	C(29)	177.9(2)	Br(1)	Pd(1)	P(2)	C(31)	-71.5(2)
Br(1)	Pd(1)	P(2)	C(35)	55.1(2)	Br(1)	Pd(1)	C(1)	C(2)	-83.0(5)
Br(1)	Pd(1)	C(1)	C(6)	94.9(4)	Fe(1)	C(19)	P(1)	C(7)	94.8(5)
Fe(1)	C(19)	P(1)	C(13)	-14.8(5)	Fe(1)	C(19)	C(20)	C(21)	60.1(4)
Fe(1)	C(19)	C(23)	C(22)	-57.4(4)	Fe(1)	C(19)	C(23)	C(29)	125.9(5)
Fe(1)	C(20)	C(19)	P(1)	133.7(4)	Fe(1)	C(20)	C(19)	C(23)	-61.0(4)
Fe(1)	C(20)	C(21)	C(22)	59.3(4)	Fe(1)	C(21)	C(20)	C(19)	-59.9(4)
Fe(1)	C(21)	C(22)	C(23)	60.7(4)	Fe(1)	C(22)	C(21)	C(20)	-58.7(4)
Fe(1)	C(22)	C(23)	C(19)	57.1(4)	Fe(1)	C(22)	C(23)	C(29)	-126.4(6)
Fe(1)	C(23)	C(19)	P(1)	-135.2(4)	Fe(1)	C(23)	C(19)	C(20)	59.6(4)
Fe(1)	C(23)	C(22)	C(21)	-59.7(4)	Fe(1)	C(23)	C(29)	P(2)	157.6(4)
Fe(1)	C(23)	C(29)	C(30)	-65.4(7)	Fe(1)	C(24)	C(25)	C(26)	-60.1(5)
Fe(1)	C(24)	C(28)	C(27)	58.6(5)	Fe(1)	C(25)	C(24)	C(28)	60.0(5)
Fe(1)	C(25)	C(26)	C(27)	-58.7(5)	Fe(1)	C(26)	C(25)	C(24)	59.8(4)
Fe(1)	C(26)	C(27)	C(28)	-60.2(4)	Fe(1)	C(27)	C(26)	C(25)	58.5(5)
Fe(1)	C(27)	C(28)	C(24)	-58.5(5)	Fe(1)	C(28)	C(24)	C(25)	-59.5(4)
Fe(1)	C(28)	C(27)	C(26)	60.1(5)	P(1)	Pd(1)	P(2)	C(29)	13.4(2)
P(1)	Pd(1)	P(2)	C(31)	124.0(2)	P(1)	Pd(1)	P(2)	C(35)	-109.4(2)
P(1)	Pd(1)	C(1)	C(2)	77.3(5)	P(1)	Pd(1)	C(1)	C(6)	-104.7(4)
P(1)	C(7)	C(8)	C(9)	-169.6(5)	P(1)	C(7)	C(12)	C(11)	171.1(6)
P(1)	C(13)	C(14)	C(15)	176.8(5)	P(1)	C(13)	C(18)	C(17)	-175.6(5)

Table S12. Torsion Angles(°) for **5** (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
P(1)	C(19)	Fe(1)	C(20)	-119.8(6)	P(1)	C(19)	Fe(1)	C(21)	-157.2(5)
P(1)	C(19)	Fe(1)	C(22)	159.3(5)	P(1)	C(19)	Fe(1)	C(23)	122.3(6)
(1)	C(19)	Fe(1)	C(24)	38.0(6)	P(1)	C(19)	Fe(1)	C(25)	-6.2(5)
P(1)	C(19)	Fe(1)	C(26)	-49.7(6)	P(1)	C(19)	Fe(1)	C(27)	-93.7(10)
P(1)	C(19)	Fe(1)	C(28)	79.0(10)	P(1)	C(19)	C(20)	C(21)	-166.2(4)
P(1)	C(19)	C(23)	C(22)	167.4(4)	P(1)	C(19)	C(23)	C(29)	-9.3(8)
P(2)	Pd(1)	P(1)	C(7)	138.0(2)	P(2)	Pd(1)	P(1)	C(13)	-99.7(2)
P(2)	Pd(1)	P(1)	C(19)	28.7(2)	P(2)	Pd(1)	C(1)	C(2)	170.1(3)
P(2)	Pd(1)	C(1)	C(6)	-12.0(9)	P(2)	C(29)	C(23)	C(19)	67.9(6)
P(2)	C(29)	C(23)	C(22)	-108.1(6)	O(1)	C(40)	C(41)	C(42)	-23(1)
O(1)	C(43)	C(42)	C(41)	-22(1)	C(1)	Pd(1)	P(1)	C(7)	-60.6(3)
PC(1)	Pd(1)	P(1)	C(13)	61.7(3)	C(1)	Pd(1)	P(1)	C(19)	-169.9(3)
C(1)	Pd(1)	P(2)	C(29)	-77.2(6)	C(1)	Pd(1)	P(2)	C(31)	33.4(5)
C(1)	Pd(1)	P(2)	C(35)	159.9(5)	C(1)	C(2)	C(3)	C(4)	-0.4(9)
C(1)	C(6)	C(5)	C(4)	0(1)	C(2)	C(1)	C(6)	C(5)	-0.5(10)
C(2)	C(3)	C(4)	C(5)	-1.0(9)	C(3)	C(2)	C(1)	C(6)	1.1(9)
C(3)	C(4)	C(5)	C(6)	1.7(10)	C(7)	P(1)	C(13)	C(14)	44.8(5)
C(7)	P(1)	C(13)	C(18)	-142.7(5)	C(7)	P(1)	C(19)	C(20)	-1.9(5)
C(7)	P(1)	C(19)	C(23)	-164.5(5)	C(7)	C(8)	C(9)	C(10)	-1(1)
C(7)	C(12)	C(11)	C(10)	-2(1)	C(8)	C(7)	P(1)	C(13)	-162.8(5)
C(8)	C(7)	P(1)	C(19)	81.5(5)	C(8)	C(7)	C(12)	C(11)	2(1)
C(8)	C(9)	C(10)	C(11)	1(1)	C(9)	C(8)	C(7)	C(12)	0(1)
C(9)	C(10)	C(11)	C(12)	0(1)	C(12)	C(7)	P(1)	C(13)	28.5(6)
C(12)	C(7)	P(1)	C(19)	-87.3(6)	C(13)	P(1)	C(19)	C(20)	-111.4(5)
C(13)	P(1)	C(19)	C(23)	86.0(5)	C(13)	C(14)	C(15)	C(16)	-1.7(10)
C(13)	C(18)	C(17)	C(16)	-0.3(9)	C(14)	C(13)	P(1)	C(19)	149.4(5)
C(14)	C(13)	C(18)	C(17)	-3.0(9)	C(14)	C(15)	C(16)	C(17)	-1(1)
C(15)	C(14)	C(13)	C(18)	4.0(9)	C(15)	C(16)	C(17)	C(18)	2(1)
C(18)	C(13)	P(1)	C(19)	-38.0(6)	C(19)	Fe(1)	C(20)	C(21)	-119.3(5)
C(19)	Fe(1)	C(21)	C(20)	38.0(4)	C(19)	Fe(1)	C(21)	C(22)	-81.9(4)
C(19)	Fe(1)	C(22)	C(21)	81.6(4)	C(19)	Fe(1)	C(22)	C(23)	-37.4(3)

Table S12. Torsion Angles(°) for **5** (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(19)	Fe(1)	C(23)	C(22)	120.2(5)	C(19)	Fe(1)	C(23)	C(29)	-116.8(7)
C(19)	Fe(1)	C(24)	C(25)	-83.0(5)	C(19)	Fe(1)	C(24)	C(28)	158.3(4)
C(19)	Fe(1)	C(25)	C(24)	119.1(4)	C(19)	Fe(1)	C(25)	C(26)	-121.9(4)
C(19)	Fe(1)	C(26)	C(25)	80.4(5)	C(19)	Fe(1)	C(26)	C(27)	-159.5(4)
C(19)	Fe(1)	C(27)	C(26)	58(1)	C(19)	Fe(1)	C(27)	C(28)	175.9(8)
C(19)	Fe(1)	C(28)	C(24)	-55.8(10)	C(19)	Fe(1)	C(28)	C(27)	-176.3(7)
C(19)	C(20)	Fe(1)	C(21)	119.3(5)	C(19)	C(20)	Fe(1)	C(22)	82.1(4)
C(19)	C(20)	Fe(1)	C(23)	38.5(4)	C(19)	C(20)	Fe(1)	C(24)	-58.2(8)
C(19)	C(20)	Fe(1)	C(25)	-87.6(4)	C(19)	C(20)	Fe(1)	C(26)	-128.6(4)
C(19)	C(20)	Fe(1)	C(27)	-170.3(4)	C(19)	C(20)	Fe(1)	C(28)	161.5(7)
C(19)	C(20)	C(21)	C(22)	-0.6(7)	C(19)	C(23)	Fe(1)	C(20)	-38.7(4)
C(19)	C(23)	Fe(1)	C(21)	-82.4(4)	C(19)	C(23)	Fe(1)	C(22)	-120.2(5)
C(19)	C(23)	Fe(1)	C(24)	118.3(4)	C(19)	C(23)	Fe(1)	C(25)	75.0(5)
C(19)	C(23)	Fe(1)	C(26)	28(1)	C(19)	C(23)	Fe(1)	C(27)	-155.0(6)
C(19)	C(23)	Fe(1)	C(28)	162.8(4)	C(19)	C(23)	C(22)	C(21)	-2.6(7)
C(19)	C(23)	C(29)	C(30)	-155.1(5)	C(20)	Fe(1)	C(19)	C(23)	117.9(5)
C(20)	Fe(1)	C(21)	C(22)	-119.9(5)	C(20)	Fe(1)	C(22)	C(21)	37.3(4)
C(20)	Fe(1)	C(22)	C(23)	-81.7(4)	C(20)	Fe(1)	C(23)	C(22)	81.5(4)
C(20)	Fe(1)	C(23)	C(29)	-155.5(7)	C(20)	Fe(1)	C(24)	C(25)	-39.8(9)
C(20)	Fe(1)	C(24)	C(28)	-158.6(6)	C(20)	Fe(1)	C(25)	C(24)	164.1(4)
C(20)	Fe(1)	C(25)	C(26)	-76.9(5)	C(20)	Fe(1)	C(26)	C(25)	120.4(5)
C(20)	Fe(1)	C(26)	C(27)	-119.6(5)	C(20)	Fe(1)	C(27)	C(26)	77.8(5)
C(20)	Fe(1)	C(27)	C(28)	-164.4(5)	C(20)	Fe(1)	C(28)	C(24)	159.0(7)
C(20)	Fe(1)	C(28)	C(27)	38(1)	C(20)	C(19)	Fe(1)	C(21)	-37.4(4)
C(20)	C(19)	Fe(1)	C(22)	-81.0(4)	C(20)	C(19)	Fe(1)	C(23)	-117.9(5)
C(20)	C(19)	Fe(1)	C(24)	157.8(4)	C(20)	C(19)	Fe(1)	C(25)	113.5(4)
C(20)	C(19)	Fe(1)	C(26)	70.0(5)	C(20)	C(19)	Fe(1)	C(27)	26(1)
C(20)	C(19)	Fe(1)	C(28)	-161.2(8)	C(20)	C(19)	C(23)	C(22)	2.2(6)
C(20)	C(19)	C(23)	C(29)	-174.5(5)	C(20)	C(21)	Fe(1)	C(22)	119.9(5)
C(20)	C(21)	Fe(1)	C(23)	82.2(4)	C(20)	C(21)	Fe(1)	C(24)	177.3(8)
C(20)	C(21)	Fe(1)	C(25)	-58.3(7)	C(20)	C(21)	Fe(1)	C(26)	-83.8(4)

Table S12. Torsion Angles(°) for **5** (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(20)	C(21)	Fe(1)	C(27)	-124.5(4)	C(20)	C(21)	Fe(1)	C(28)	163.0(4)
C(20)	C(21)	C(22)	C(23)	2.0(7)	C(21)	Fe(1)	C(19)	C(23)	80.5(4)
C(21)	Fe(1)	C(22)	C(23)	-119.0(5)	C(21)	Fe(1)	C(23)	C(22)	37.8(4)
C(21)	Fe(1)	C(23)	C(29)	160.8(7)	C(21)	Fe(1)	C(24)	C(25)	145.0(8)
C(21)	Fe(1)	C(24)	C(28)	26(1)	C(21)	Fe(1)	C(25)	C(24)	-155.4(6)
C(21)	Fe(1)	C(25)	C(26)	-36.5(8)	C(21)	Fe(1)	C(26)	C(25)	162.5(4)
C(21)	Fe(1)	C(26)	C(27)	-77.4(5)	C(21)	Fe(1)	C(27)	C(26)	116.5(5)
C(21)	Fe(1)	C(27)	C(28)	-125.7(5)	C(21)	Fe(1)	C(28)	C(24)	-170.1(4)
C(21)	Fe(1)	C(28)	C(27)	69.4(5)	C(21)	C(20)	Fe(1)	C(22)	-37.2(4)
C(21)	C(20)	Fe(1)	C(23)	-80.8(4)	C(21)	C(20)	Fe(1)	C(24)	-177.5(7)
C(21)	C(20)	Fe(1)	C(25)	153.1(4)	C(21)	C(20)	Fe(1)	C(26)	112.1(4)
C(21)	C(20)	Fe(1)	C(27)	70.4(5)	C(21)	C(20)	Fe(1)	C(28)	42.2(9)
C(21)	C(20)	C(19)	C(23)	-1.0(6)	C(21)	C(22)	Fe(1)	C(23)	119.0(5)
C(21)	C(22)	Fe(1)	C(24)	-159.6(4)	C(21)	C(22)	Fe(1)	C(25)	176(1)
C(21)	C(22)	Fe(1)	C(26)	-46.7(7)	C(21)	C(22)	Fe(1)	C(27)	-78.0(4)
C(21)	C(22)	Fe(1)	C(28)	-120.5(4)	C(21)	C(22)	C(23)	C(29)	173.9(5)
C(22)	Fe(1)	C(19)	C(23)	36.9(4)	C(22)	Fe(1)	C(23)	C(29)	123.0(7)
C(22)	Fe(1)	C(24)	C(25)	-171.7(4)	C(22)	Fe(1)	C(24)	C(28)	69.5(5)
C(22)	Fe(1)	C(25)	C(24)	29(1)	C(22)	Fe(1)	C(25)	C(26)	148(1)
C(22)	Fe(1)	C(26)	C(25)	-165.7(5)	C(22)	Fe(1)	C(26)	C(27)	-45.7(7)
C(22)	Fe(1)	C(27)	C(26)	156.9(4)	C(22)	Fe(1)	C(27)	C(28)	-85.3(5)
C(22)	Fe(1)	C(28)	C(24)	-128.9(5)	C(22)	Fe(1)	C(28)	C(27)	110.6(5)
C(22)	C(21)	Fe(1)	C(23)	-37.7(4)	C(22)	C(21)	Fe(1)	C(24)	57.4(10)
C(22)	C(21)	Fe(1)	C(25)	-178.2(6)	C(22)	C(21)	Fe(1)	C(26)	156.4(4)
C(22)	C(21)	Fe(1)	C(27)	115.7(4)	C(22)	C(21)	Fe(1)	C(28)	77.1(5)
C(22)	C(23)	Fe(1)	C(24)	-121.5(4)	C(22)	C(23)	Fe(1)	C(25)	-164.8(4)
C(22)	C(23)	Fe(1)	C(26)	148(1)	C(22)	C(23)	Fe(1)	C(27)	-34.9(7)
C(22)	C(23)	Fe(1)	C(28)	-77.0(5)	C(22)	C(23)	C(29)	C(30)	29.0(8)
C(23)	Fe(1)	C(24)	C(25)	-127.8(4)	C(23)	Fe(1)	C(24)	C(28)	113.4(5)
C(23)	Fe(1)	C(25)	C(24)	76.2(5)	C(23)	Fe(1)	C(25)	C(26)	-164.8(4)

Table S12. Torsion Angles(°) for **5** (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(23)	Fe(1)	C(26)	C(25)	57(1)	C(23)	Fe(1)	C(26)	C(27)	177.7(10)
C(23)	Fe(1)	C(27)	C(26)	-178.8(5)	C(23)	Fe(1)	C(27)	C(28)	-61.0(8)
C(23)	Fe(1)	C(28)	C(24)	-87.8(5)	C(23)	Fe(1)	C(28)	C(27)	151.7(5)
C(23)	C(19)	Fe(1)	C(24)	-84.3(5)	C(23)	C(19)	Fe(1)	C(25)	-128.6(4)
C(23)	C(19)	Fe(1)	C(26)	-172.1(4)	C(23)	C(19)	Fe(1)	C(27)	144.0(9)
C(23)	C(19)	Fe(1)	C(28)	-43.3(9)	C(23)	C(22)	Fe(1)	C(24)	81.4(5)
C(23)	C(22)	Fe(1)	C(25)	57(1)	C(23)	C(22)	Fe(1)	C(26)	165.7(5)
C(23)	C(22)	Fe(1)	C(27)	163.1(4)	C(23)	C(22)	Fe(1)	C(28)	120.6(4)
C(23)	C(29)	P(2)	C(31)	-176.8(4)	C(23)	C(29)	P(2)	C(35)	64.9(5)
C(24)	Fe(1)	C(23)	C(29)	1.5(7)	C(24)	Fe(1)	C(25)	C(26)	119.0(6)
C(24)	Fe(1)	C(26)	C(25)	-38.0(4)	C(24)	Fe(1)	C(26)	C(27)	82.1(5)
C(24)	Fe(1)	C(27)	C(26)	-81.0(5)	C(24)	Fe(1)	C(27)	C(28)	36.8(5)
C(24)	Fe(1)	C(28)	C(27)	-120.5(7)	C(24)	C(25)	Fe(1)	C(26)	-119.0(6)
C(24)	C(25)	Fe(1)	C(27)	-81.6(5)	C(24)	C(25)	Fe(1)	C(28)	-37.7(4)
C(24)	C(25)	C(26)	C(27)	1.1(7)	C(24)	C(28)	Fe(1)	C(25)	38.1(5)
C(24)	C(28)	Fe(1)	C(26)	81.9(5)	C(24)	C(28)	Fe(1)	C(27)	120.5(7)
C(24)	C(28)	C(27)	C(26)	1.6(8)	C(25)	Fe(1)	C(23)	C(29)	-41.8(7)
C(25)	Fe(1)	C(24)	C(28)	-118.7(6)	C(25)	Fe(1)	C(26)	C(27)	120.0(6)
C(25)	Fe(1)	C(27)	C(26)	-37.0(4)	C(25)	Fe(1)	C(27)	C(28)	80.8(5)
C(25)	Fe(1)	C(28)	C(27)	-82.3(5)	C(25)	C(24)	Fe(1)	C(26)	37.6(4)
C(25)	C(24)	Fe(1)	C(27)	81.7(5)	C(25)	C(24)	Fe(1)	C(28)	118.7(6)
C(25)	C(24)	C(28)	C(27)	-1.0(8)	C(25)	C(26)	Fe(1)	C(27)	-120.0(6)
C(25)	C(26)	Fe(1)	C(28)	-81.6(5)	C(25)	C(26)	C(27)	C(28)	-1.7(7)
C(26)	Fe(1)	C(23)	C(29)	-88(1)	C(26)	Fe(1)	C(24)	C(28)	-81.1(5)
C(26)	Fe(1)	C(27)	C(28)	117.8(7)	C(26)	Fe(1)	C(28)	C(27)	-38.6(5)
C(26)	C(25)	Fe(1)	C(27)	37.4(4)	C(26)	C(25)	Fe(1)	C(28)	81.2(5)
C(26)	C(25)	C(24)	C(28)	-0.1(7)	C(26)	C(27)	Fe(1)	C(28)	-117.8(7)
C(27)	Fe(1)	C(23)	C(29)	88.1(8)	C(27)	Fe(1)	C(24)	C(28)	-37.0(5)
C(27)	C(26)	Fe(1)	C(28)	38.4(5)	C(28)	Fe(1)	C(23)	C(29)	46.0(7)
C(29)	P(2)	C(31)	C(32)	162.1(5)	C(29)	P(2)	C(31)	C(33)	47.4(5)
C(29)	P(2)	C(31)	C(34)	-74.9(5)	C(29)	P(2)	C(35)	C(36)	-86.4(5)
C(29)	P(2)	C(35)	C(37)	32.8(5)	C(29)	P(2)	C(35)	C(38)	157.5(4)

Table S12. Torsion Angles($^{\circ}$) for **5** (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(30)	C(29)	P(2)	C(31)	51.9(6)	C(30)	C(29)	P(2)	C(35)	-66.5(6)
C(31)	P(2)	C(35)	C(36)	161.4(4)	C(31)	P(2)	C(35)	C(37)	-79.4(5)
C(31)	P(2)	C(35)	C(38)	45.3(5)	C(32)	C(31)	P(2)	C(35)	-81.0(6)
C(33)	C(31)	P(2)	C(35)	164.3(4)	C(34)	C(31)	P(2)	C(35)	42.0(6)
C(40)	O(1)	C(43)	C(42)	7(1)	C(40)	C(41)	C(42)	C(43)	27(1)
C(41)	C(40)	O(1)	C(43)	11(1)					

Table S13. Non-bonded Contacts out to 3.60 Å for **5**

atom	atom	distance	ADC	atom	atom	distance	ADC
O(1)	C(21)	3.45(1)	1	O(1)	C(12)	3.454(9)	35302
O(1)	C(25)	3.59(1)	35302	C(2)	C(17)	3.469(8)	65501
C(3)	C(16)	3.508(8)	65501	C(3)	C(17)	3.590(9)	65501
C(5)	C(38)	3.561(9)	44402	C(33)	C(37)	3.58(1)	34402

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	X,	Y,	Z	(2)	-X,	1/2+Y,	-Z

Crystallographic data for **6**.

Figure S7. X-ray crystal structure of **6**

Data Collection

The data were collected on a Nonius KappaCCD diffractometer with graphite monochromated Mo-K α radiation at a temperature of 183(2) K to a maximum 2θ value of 56.56 $^\circ$. Five omega scans consisting of 37, 37, 19, 19, and 20 data frames, respectively, were collected with a frame width of 2.0 $^\circ$ and a detector-to-crystal distance, Dx, of 35 mm. Each frame was exposed twice (for the purpose of de-zinging) for a total of 60 seconds. The data frames were processed and scaled using the DENZO software package.¹

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques². The non-hydrogen atoms, with exceptions noted, were refined anisotropically and hydrogen atoms were treated as idealized contributions.

REFERENCES

(1) Z. Otwinowski and W. Minor, "Processing of X-Ray Diffraction Data Collected in Oscillation Mode," *Methods in Enzymology*, vol. 276: *Macromolecular Crystallography*, part A, 307-326, 1997, C.W. Carter, Jr. & R.M. Sweet, Eds., Academic Press.

(2) *Acta Cryst.* **A46** (1990) 467-473

Structural Description

The molecule possesses a high degree of positional disorder in both the palladium complex and the co-crystallized solvent. Successful refinement in the space group $C222_1$ yielded a composite of two orientations of the palladium complex with equal occupancy factors. Two alternative positions for the unsubstituted Cp ring, C(6-10), and the phenyl-ring, C(33-38), were modeled and refined with anisotropic displacement parameters. The palladium complex co-crystallized with one ordered molecule of tetrahydrofuran and one severely disordered molecule of pentane. The co-crystallized pentane resides in an infinitely extending channel formed by adjacent palladium complexes and propagating along the crystallographic *c*-axis. Thus the structure refinement represents an approximate location and orientation of the pentane molecule. All solvent atoms were refined isotropically.

Table S14. Crystal data and structure refinement for **6**.

Empirical formula	$C_{47} H_{77} Br Fe O P_2 Pd$	
Formula weight	962.19	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	C222(1)	
Unit cell dimensions	$a = 22.412(5)$ Å	$= 90^\circ$.
	$b = 24.106(5)$ Å	$= 90^\circ$.
	$c = 16.388(3)$ Å	$= 90^\circ$.
Volume	$8854(3)$ Å ³	
Z	8	
Density (calculated)	1.444 g/cm ³	
Absorption coefficient	17.43 cm ⁻¹	
F(000)	4032	
Crystal size	0.35 x 0.20 x 0.20 mm ³	
Theta range for data collection	2.20 to 28.28°.	
Index ranges	$-29 \leq h \leq 29$, $-29 \leq k \leq 31$, $-21 \leq l \leq 21$	
Reflections collected	10177	
Independent reflections	10177 [R(int) = 0.0000]	
Completeness to theta = 28.28°	99.1 %	
Absorption correction	None	
Max. and min. transmission	0.7219 and 0.5806	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10177 / 0 / 481	
Goodness-of-fit on F ²	0.931	
Final R indices [I>2sigma(I)]	R1 = 0.0481, wR2 = 0.1213	
R indices (all data)	R1 = 0.0696, wR2 = 0.1361	
Absolute structure parameter	0.013(10)	
Largest diff. peak and hole	0.599 and -0.732 e.Å ⁻³	

Table S15. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Pd(1)	2037(1)	7075(1)	4595(1)	25(1)
Br(1)	2557(1)	7212(1)	3259(1)	33(1)
Fe(1)	1506(1)	5905(1)	7230(1)	32(1)
P(2)	2880(1)	6733(1)	5364(1)	22(1)
P(1)	1297(1)	6741(1)	5418(1)	26(1)
C(1)	1574(1)	6164(1)	6031(1)	26(1)
C(2)	2157(1)	6126(1)	6396(1)	25(1)
C(3)	2240(1)	5579(1)	6671(1)	32(1)
C(4)	1725(1)	5261(1)	6482(1)	33(1)
C(5)	1313(1)	5612(1)	6086(1)	31(1)
C(6)	1005(1)	6374(1)	7961(1)	33(1)
C(7)	1605(1)	6392(1)	8239(1)	26(1)
C(8)	1783(1)	5842(1)	8432(1)	29(1)
C(9)	1294(1)	5484(1)	8273(1)	33(1)
C(10)	813(1)	5813(1)	7982(1)	43(1)
C(6')	727(1)	6271(1)	7805(1)	45(1)
C(7')	1255(1)	6558(1)	8035(1)	49(1)
C(8')	1647(1)	6170(1)	8407(2)	51(1)
C(9')	1362(1)	5644(1)	8406(2)	47(1)
C(10')	794(1)	5706(1)	8034(1)	37(1)
C(11)	687(1)	6441(1)	4780(1)	34(1)
C(12)	131(1)	6241(1)	5219(1)	44(1)
C(13)	-355(1)	6093(1)	4601(1)	55(1)
C(14)	-123(1)	5662(1)	3984(1)	74(1)
C(15)	446(1)	5864(1)	3555(1)	61(1)
C(16)	930(1)	6019(1)	4167(1)	49(1)
C(17)	906(1)	7231(1)	6105(1)	30(1)
C(18)	1371(1)	7593(1)	6557(1)	46(1)
C(19)	1087(1)	8024(1)	7117(1)	81(1)
C(20)	638(1)	8378(1)	6646(2)	95(1)

C(21)	180(1)	8033(1)	6230(1)	65(1)
C(22)	457(1)	7609(1)	5645(1)	42(1)
C(23)	2615(1)	6581(1)	6433(1)	25(1)
C(24)	3064(1)	6478(1)	7129(1)	31(1)
C(25)	3459(1)	7299(1)	5526(1)	26(1)
C(26)	3179(1)	7729(1)	6100(1)	34(1)
C(27)	4063(1)	7099(1)	5869(1)	35(1)
C(28)	3586(1)	7602(1)	4716(1)	31(1)
C(29)	3229(1)	6081(1)	4920(1)	26(1)
C(30)	3598(1)	5743(1)	5528(1)	35(1)
C(31)	3631(1)	6233(1)	4201(1)	35(1)
C(32)	2708(1)	5736(1)	4577(1)	29(1)
C(33)	1425(1)	7588(1)	4081(1)	65(1)
C(35')	631(1)	8179(1)	3296(1)	66(1)
C(37)	894(1)	8524(1)	3737(1)	82(1)
C(34)	973(1)	7311(1)	3397(1)	25(1)
C(35)	563(2)	7686(2)	3043(2)	42(1)
C(38)	1360(1)	8053(1)	4198(2)	30(1)
C(34')	1046(1)	7601(1)	3581(2)	35(1)
C(37')	1309(2)	8675(2)	4199(2)	45(1)
C(38')	1634(2)	8215(1)	4439(2)	35(1)
O(1)	2092(2)	4244(1)	9345(2)	180(1)
C(39)	2692(2)	4178(2)	8837(2)	151(2)
C(40)	2479(2)	4233(2)	8031(2)	157(2)
C(41)	1919(2)	4057(2)	7947(3)	175(2)
C(42)	1689(1)	4165(1)	8653(1)	73(1)
C(43)	4551(3)	4969(4)	7224(4)	316(4)
C(44)	4123(3)	4973(4)	7949(3)	283(3)
C(45)	4707(3)	5063(3)	8718(4)	238(3)
C(46)	4457(4)	4908(4)	9494(3)	377(6)
C(47)	4870(6)	5000	10000	334(6)

Table S16. Bond lengths [Å] and angles [°] for **6**.

Pd(1)-C(33)	2.0291(19)	C(6')-C(10')	1.4200
Pd(1)-P(1)	2.2847(5)	C(7')-C(8')	1.4200
Pd(1)-P(2)	2.4157(5)	C(8')-C(9')	1.4200
Pd(1)-Br(1)	2.5031(4)	C(9')-C(10')	1.4200
Fe(1)-C(6)	1.9926(14)	C(11)-C(12)	1.517(2)
Fe(1)-C(10)	1.9953(18)	C(11)-C(16)	1.531(2)
Fe(1)-C(4)	2.0377(16)	C(12)-C(13)	1.528(2)
Fe(1)-C(7)	2.0403(17)	C(13)-C(14)	1.540(3)
Fe(1)-C(3)	2.0408(16)	C(14)-C(15)	1.535(3)
Fe(1)-C(9)	2.045(2)	C(15)-C(16)	1.524(3)
Fe(1)-C(5)	2.0498(15)	C(17)-C(18)	1.548(2)
Fe(1)-C(9')	2.054(3)	C(17)-C(22)	1.552(2)
Fe(1)-C(8')	2.057(3)	C(18)-C(19)	1.526(3)
Fe(1)-C(1)	2.0671(13)	C(19)-C(20)	1.529(3)
Fe(1)-C(2)	2.0691(14)	C(20)-C(21)	1.486(3)
Fe(1)-C(8)	2.072(2)	C(21)-C(22)	1.533(3)
P(2)-C(23)	1.8868(14)	C(23)-C(24)	1.543(2)
P(2)-C(25)	1.9004(15)	C(25)-C(26)	1.536(2)
P(2)-C(29)	1.9008(15)	C(25)-C(28)	1.5418(19)
P(1)-C(1)	1.8236(15)	C(25)-C(27)	1.544(2)
P(1)-C(17)	1.8525(15)	C(29)-C(31)	1.527(2)
P(1)-C(11)	1.8658(16)	C(29)-C(30)	1.529(2)
C(1)-C(2)	1.440(2)	C(29)-C(32)	1.541(2)
C(1)-C(5)	1.458(2)	C(33)-C(38)	1.147(3)
C(2)-C(3)	1.406(2)	C(33)-C(34')	1.182(3)
C(2)-C(23)	1.502(2)	C(33)-C(34)	1.653(3)
C(3)-C(4)	1.421(2)	C(33)-C(38')	1.687(4)
C(4)-C(5)	1.410(2)	C(35')-C(37)	1.250(3)
C(6)-C(7)	1.4200	C(35')-C(35)	1.269(4)
C(6)-C(10)	1.4200	C(35')-C(34')	1.741(4)
C(7)-C(8)	1.4200	C(37)-C(37')	1.252(4)
C(8)-C(9)	1.4200	C(37)-C(38)	1.717(4)
C(9)-C(10)	1.4200	C(34)-C(35)	1.414(4)
C(6')-C(7')	1.4200	C(37')-C(38')	1.383(5)

O(1)-C(42)	1.461(4)	C(7)-Fe(1)-C(5)	164.38(6)
O(1)-C(39)	1.590(6)	C(3)-Fe(1)-C(5)	68.10(6)
C(39)-C(40)	1.410(5)	C(9)-Fe(1)-C(5)	122.99(8)
C(40)-C(41)	1.332(7)	C(6)-Fe(1)-C(9')	61.38(8)
C(41)-C(42)	1.294(5)	C(10)-Fe(1)-C(9')	42.56(8)
C(43)-C(44)	1.526(9)	C(4)-Fe(1)-C(9')	111.59(9)
C(44)-C(45)	1.829(9)	C(7)-Fe(1)-C(9')	55.43(10)
C(45)-C(46)	1.439(9)	C(3)-Fe(1)-C(9')	115.40(8)
C(46)-C(47)	1.261(13)	C(9)-Fe(1)-C(9')	13.10(10)
C(46)-C(46)#1	1.717(12)	C(5)-Fe(1)-C(9')	136.00(10)
C(47)-C(46)#1	1.261(13)	C(6)-Fe(1)-C(8')	49.20(7)
		C(10)-Fe(1)-C(8')	64.82(8)
C(33)-Pd(1)-P(1)	88.27(5)	C(4)-Fe(1)-C(8')	139.74(9)
C(33)-Pd(1)-P(2)	162.27(6)	C(7)-Fe(1)-C(8')	17.11(9)
P(1)-Pd(1)-P(2)	98.045(19)	C(3)-Fe(1)-C(8')	114.57(8)
C(33)-Pd(1)-Br(1)	82.61(5)	C(9)-Fe(1)-C(8')	53.48(10)
P(1)-Pd(1)-Br(1)	154.346(11)	C(5)-Fe(1)-C(8')	175.93(9)
P(2)-Pd(1)-Br(1)	97.833(16)	C(9')-Fe(1)-C(8')	40.41(5)
C(6)-Fe(1)-C(10)	41.7	C(6)-Fe(1)-C(1)	116.26(6)
C(6)-Fe(1)-C(4)	158.28(6)	C(10)-Fe(1)-C(1)	132.76(7)
C(10)-Fe(1)-C(4)	118.26(6)	C(4)-Fe(1)-C(1)	68.96(6)
C(6)-Fe(1)-C(7)	41.2	C(7)-Fe(1)-C(1)	126.07(7)
C(10)-Fe(1)-C(7)	69.4	C(3)-Fe(1)-C(1)	68.31(6)
C(4)-Fe(1)-C(7)	154.08(6)	C(9)-Fe(1)-C(1)	164.17(7)
C(6)-Fe(1)-C(3)	160.41(6)	C(5)-Fe(1)-C(1)	41.47(6)
C(10)-Fe(1)-C(3)	149.49(6)	C(9')-Fe(1)-C(1)	175.21(8)
C(4)-Fe(1)-C(3)	40.78(6)	C(8')-Fe(1)-C(1)	141.88(10)
C(7)-Fe(1)-C(3)	119.80(6)	C(6)-Fe(1)-C(2)	130.42(6)
C(6)-Fe(1)-C(9)	69.36(5)	C(10)-Fe(1)-C(2)	170.36(6)
C(10)-Fe(1)-C(9)	41.1	C(4)-Fe(1)-C(2)	68.26(6)
C(4)-Fe(1)-C(9)	100.38(7)	C(7)-Fe(1)-C(2)	108.06(6)
C(7)-Fe(1)-C(9)	68.45(6)	C(3)-Fe(1)-C(2)	40.02(6)
C(3)-Fe(1)-C(9)	111.77(7)	C(9)-Fe(1)-C(2)	147.54(7)
C(6)-Fe(1)-C(5)	128.85(6)	C(5)-Fe(1)-C(2)	68.53(6)
C(10)-Fe(1)-C(5)	111.26(7)	C(9')-Fe(1)-C(2)	144.03(8)
C(4)-Fe(1)-C(5)	40.34(6)	C(8')-Fe(1)-C(2)	115.52(8)

C(1)-Fe(1)-C(2)	40.76(6)	C(2)-C(3)-C(4)	109.18(13)
C(6)-Fe(1)-C(8)	68.8	C(2)-C(3)-Fe(1)	71.08(9)
C(10)-Fe(1)-C(8)	68.76(6)	C(4)-C(3)-Fe(1)	69.49(9)
C(4)-Fe(1)-C(8)	116.32(7)	C(5)-C(4)-C(3)	108.02(14)
C(7)-Fe(1)-C(8)	40.4	C(5)-C(4)-Fe(1)	70.29(9)
C(3)-Fe(1)-C(8)	98.96(7)	C(3)-C(4)-Fe(1)	69.73(9)
C(9)-Fe(1)-C(8)	40.4	C(4)-C(5)-C(1)	108.27(13)
C(5)-Fe(1)-C(8)	155.18(7)	C(4)-C(5)-Fe(1)	69.36(9)
C(9')-Fe(1)-C(8)	29.76(10)	C(1)-C(5)-Fe(1)	69.90(8)
C(8')-Fe(1)-C(8)	23.69(9)	C(7)-C(6)-C(10)	108.0
C(1)-Fe(1)-C(8)	154.65(8)	C(7)-C(6)-Fe(1)	71.20(7)
C(2)-Fe(1)-C(8)	115.76(7)	C(10)-C(6)-Fe(1)	69.24(6)
C(23)-P(2)-C(25)	102.98(6)	C(8)-C(7)-C(6)	108.0
C(23)-P(2)-C(29)	108.92(6)	C(8)-C(7)-Fe(1)	71.00(6)
C(25)-P(2)-C(29)	111.42(6)	C(6)-C(7)-Fe(1)	67.59(6)
C(23)-P(2)-Pd(1)	107.71(5)	C(9)-C(8)-C(7)	108.0
C(25)-P(2)-Pd(1)	111.25(5)	C(9)-C(8)-Fe(1)	68.79(6)
C(29)-P(2)-Pd(1)	113.90(5)	C(7)-C(8)-Fe(1)	68.6
C(1)-P(1)-C(17)	108.21(7)	C(10)-C(9)-C(8)	108.0
C(1)-P(1)-C(11)	105.29(7)	C(10)-C(9)-Fe(1)	67.57(6)
C(17)-P(1)-C(11)	103.95(7)	C(8)-C(9)-Fe(1)	70.86(6)
C(1)-P(1)-Pd(1)	110.25(5)	C(9)-C(10)-C(6)	108.0
C(17)-P(1)-Pd(1)	118.48(5)	C(9)-C(10)-Fe(1)	71.29(6)
C(11)-P(1)-Pd(1)	109.74(5)	C(6)-C(10)-Fe(1)	69.04(5)
C(2)-C(1)-C(5)	106.30(12)	C(7')-C(6')-C(10')	108.0
C(2)-C(1)-P(1)	125.92(11)	C(7')-C(6')-Fe(1)	69.14(8)
C(5)-C(1)-P(1)	126.39(11)	C(10')-C(6')-Fe(1)	69.00(8)
C(2)-C(1)-Fe(1)	69.70(7)	C(8')-C(7')-C(6')	108.0
C(5)-C(1)-Fe(1)	68.63(8)	C(8')-C(7')-Fe(1)	67.45(8)
P(1)-C(1)-Fe(1)	136.73(8)	C(6')-C(7')-Fe(1)	72.30(7)
C(3)-C(2)-C(1)	108.22(13)	C(7')-C(8')-C(9')	108.0
C(3)-C(2)-C(23)	125.54(13)	C(7')-C(8')-Fe(1)	72.93(7)
C(1)-C(2)-C(23)	126.14(13)	C(9')-C(8')-Fe(1)	69.71(9)
C(3)-C(2)-Fe(1)	68.91(8)	C(10')-C(9')-C(8')	108.0
C(1)-C(2)-Fe(1)	69.55(8)	C(10')-C(9')-Fe(1)	72.86(7)
C(23)-C(2)-Fe(1)	130.02(10)	C(8')-C(9')-Fe(1)	69.87(8)

C(9')-C(10')-C(6')	108.0	C(34')-C(33)-C(34)	25.6(2)
C(9')-C(10')-Fe(1)	67.47(8)	C(38)-C(33)-C(38')	26.07(19)
C(6')-C(10')-Fe(1)	72.41(7)	C(34')-C(33)-C(38')	114.7(2)
C(12)-C(11)-C(16)	113.13(14)	C(34)-C(33)-C(38')	140.21(19)
C(12)-C(11)-P(1)	117.32(11)	C(38)-C(33)-Pd(1)	127.8(2)
C(16)-C(11)-P(1)	111.34(11)	C(34')-C(33)-Pd(1)	142.1(2)
C(11)-C(12)-C(13)	110.14(14)	C(34)-C(33)-Pd(1)	116.66(15)
C(12)-C(13)-C(14)	110.67(15)	C(38')-C(33)-Pd(1)	102.40(14)
C(15)-C(14)-C(13)	111.55(17)	C(37)-C(35')-C(35)	150.3(3)
C(16)-C(15)-C(14)	111.64(15)	C(37)-C(35')-C(34')	97.2(2)
C(15)-C(16)-C(11)	109.96(15)	C(35)-C(35')-C(34')	53.1(2)
C(18)-C(17)-C(22)	109.70(14)	C(35')-C(37)-C(37')	153.2(3)
C(18)-C(17)-P(1)	109.42(10)	C(35')-C(37)-C(38)	95.9(2)
C(22)-C(17)-P(1)	112.64(10)	C(37')-C(37)-C(38)	58.3(2)
C(19)-C(18)-C(17)	113.03(14)	C(35)-C(34)-C(33)	114.7(2)
C(18)-C(19)-C(20)	110.47(16)	C(35')-C(35)-C(34)	112.9(3)
C(21)-C(20)-C(19)	111.93(18)	C(33)-C(38)-C(37)	130.6(3)
C(20)-C(21)-C(22)	112.33(16)	C(33)-C(34')-C(35')	126.2(3)
C(21)-C(22)-C(17)	110.47(13)	C(37)-C(37')-C(38')	109.3(3)
C(2)-C(23)-C(24)	111.01(12)	C(37')-C(38')-C(33)	118.2(3)
C(2)-C(23)-P(2)	108.63(9)	C(42)-O(1)-C(39)	96.0(2)
C(24)-C(23)-P(2)	120.84(10)	C(40)-C(39)-O(1)	101.2(4)
C(26)-C(25)-C(28)	106.45(12)	C(41)-C(40)-C(39)	112.7(4)
C(26)-C(25)-C(27)	110.23(12)	C(42)-C(41)-C(40)	102.6(4)
C(28)-C(25)-C(27)	107.42(12)	C(41)-C(42)-O(1)	118.3(3)
C(26)-C(25)-P(2)	106.94(10)	C(43)-C(44)-C(45)	95.1(5)
C(28)-C(25)-P(2)	110.21(9)	C(46)-C(45)-C(44)	107.5(6)
C(27)-C(25)-P(2)	115.26(10)	C(47)-C(46)-C(45)	104.5(8)
C(31)-C(29)-C(30)	108.16(12)	C(47)-C(46)-C(46)#1	47.1(6)
C(31)-C(29)-C(32)	107.12(12)	C(45)-C(46)-C(46)#1	141.9(9)
C(30)-C(29)-C(32)	111.08(12)	C(46)#1-C(47)-C(46)	85.8(12)
C(31)-C(29)-P(2)	109.84(10)	C(38)-C(33)-C(34)	115.5(2)
C(30)-C(29)-P(2)	114.51(10)	C(38)-C(33)-C(34')	90.0(3)
C(32)-C(29)-P(2)	105.89(10)		

Symmetry transformations used to generate equivalent atoms: #1 x,-y+1,-z+2

Table S17. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6**. The anisotropic displacement factor exponent takes the form: $-2 \text{ }^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Pd(1)	27(1)	30(1)	18(1)	0(1)	1(1)	7(1)
Br(1)	32(1)	45(1)	22(1)	5(1)	3(1)	2(1)
Fe(1)	35(1)	37(1)	24(1)	1(1)	2(1)	-14(1)
P(2)	22(1)	23(1)	20(1)	2(1)	-2(1)	-3(1)
P(1)	21(1)	37(1)	20(1)	-4(1)	0(1)	5(1)
C(1)	25(1)	35(1)	19(1)	-1(1)	-2(1)	1(1)
C(2)	22(1)	32(1)	21(1)	5(1)	-1(1)	-3(1)
C(3)	31(1)	33(1)	32(1)	6(1)	-2(1)	-2(1)
C(4)	35(1)	25(1)	39(1)	-3(1)	-3(1)	-9(1)
C(5)	28(1)	37(1)	28(1)	-9(1)	3(1)	-8(1)
C(6)	26(1)	61(2)	11(1)	-3(1)	5(1)	10(2)
C(7)	41(2)	12(1)	25(1)	-4(1)	7(1)	1(1)
C(8)	50(2)	22(1)	15(1)	5(1)	-5(1)	-6(1)
C(9)	41(2)	33(2)	24(1)	14(1)	12(1)	-11(1)
C(10)	39(2)	38(2)	51(2)	5(1)	33(1)	-21(1)
C(6')	45(2)	45(2)	43(2)	1(2)	1(2)	-2(2)
C(7')	51(2)	50(2)	46(2)	-1(2)	4(2)	-6(2)
C(8')	52(2)	52(2)	49(2)	0(2)	0(2)	-1(2)
C(9')	49(2)	48(2)	43(2)	5(2)	3(2)	1(2)
C(10')	40(2)	34(2)	37(2)	1(1)	0(2)	1(2)
C(11)	31(1)	43(1)	28(1)	-8(1)	-12(1)	8(1)
C(12)	26(1)	58(1)	47(1)	-13(1)	-13(1)	-2(1)
C(13)	34(1)	63(1)	70(1)	-24(1)	-24(1)	4(1)
C(14)	55(1)	76(1)	92(1)	-39(1)	-45(1)	17(1)
C(15)	54(1)	73(1)	55(1)	-39(1)	-35(1)	25(1)
C(16)	47(1)	60(1)	39(1)	-18(1)	-22(1)	17(1)
C(17)	22(1)	43(1)	26(1)	-7(1)	0(1)	9(1)
C(18)	32(1)	68(1)	40(1)	-33(1)	-9(1)	16(1)
C(19)	50(1)	115(2)	78(1)	-67(1)	-22(1)	44(1)
C(20)	60(1)	105(1)	120(2)	-76(1)	-41(1)	59(1)

C(21)	40(1)	92(1)	61(1)	-38(1)	-11(1)	40(1)
C(22)	32(1)	64(1)	29(1)	-10(1)	-2(1)	21(1)
C(23)	27(1)	27(1)	22(1)	1(1)	-1(1)	-3(1)
C(24)	28(1)	40(1)	26(1)	4(1)	-7(1)	-10(1)
C(25)	27(1)	29(1)	24(1)	3(1)	-1(1)	-6(1)
C(26)	45(1)	24(1)	33(1)	-6(1)	-1(1)	-10(1)
C(27)	29(1)	44(1)	34(1)	8(1)	-6(1)	-15(1)
C(28)	36(1)	32(1)	26(1)	2(1)	1(1)	-17(1)
C(29)	25(1)	22(1)	31(1)	-4(1)	1(1)	3(1)
C(30)	32(1)	27(1)	45(1)	2(1)	-4(1)	0(1)
C(31)	31(1)	39(1)	36(1)	-6(1)	5(1)	-3(1)
C(32)	31(1)	25(1)	32(1)	-3(1)	-1(1)	1(1)
C(33)	51(1)	104(1)	41(1)	42(1)	27(1)	54(1)
C(35')	64(1)	79(1)	53(1)	-9(1)	19(1)	-26(1)
C(37)	64(1)	116(2)	66(1)	-25(1)	21(1)	-38(1)
C(34)	40(2)	25(1)	10(1)	-5(1)	-3(1)	-6(1)
C(35)	32(2)	59(2)	35(1)	15(2)	-8(1)	5(2)
C(38)	34(2)	19(1)	36(1)	1(1)	-15(1)	8(1)
C(34')	32(2)	49(2)	25(1)	6(1)	-2(1)	-17(2)
C(37')	45(2)	42(2)	49(2)	11(2)	11(2)	-5(2)
C(38')	46(2)	21(1)	37(1)	-17(1)	-15(1)	12(1)

Table S18. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6**.

	x	y	z	U(eq)
H(3A)	2603	5437	6957	38
H(4A)	1665	4858	6604	40
H(5A)	909	5501	5883	37
H(6A)	759	6700	7790	39
H(7A)	1860	6732	8280	31
H(8A)	2189	5726	8620	35
H(9A)	1291	5072	8341	39
H(10A)	408	5674	7828	51
H(6'A)	376	6435	7516	53
H(7'A)	1335	6962	7953	59
H(8'A)	2043	6257	8657	61
H(9'A)	1522	5296	8657	56
H(10B)	492	5406	7952	44
H(11A)	546	6758	4437	41
H(12A)	-16	6536	5589	52
H(12B)	228	5911	5553	52
H(13A)	-481	6432	4308	66
H(13B)	-706	5942	4891	66
H(14A)	-39	5310	4272	89
H(14B)	-436	5590	3571	89
H(15A)	596	5567	3192	73
H(15B)	350	6190	3214	73
H(16A)	1276	6180	3875	58
H(16B)	1067	5683	4459	58
H(17A)	680	7011	6520	36
H(18A)	1634	7350	6884	56
H(18B)	1622	7786	6149	56
H(19A)	1401	8266	7349	97
H(19B)	882	7835	7574	97

H(20A)	438	8636	7027	114
H(20B)	852	8602	6233	114
H(21A)	-94	8277	5921	78
H(21B)	-59	7834	6646	78
H(22A)	666	7805	5198	50
H(22B)	139	7378	5400	50
H(23A)	2388	6919	6602	30
H(24A)	2848	6409	7639	47
H(24B)	3320	6805	7195	47
H(24C)	3311	6155	6997	47
H(26A)	3465	8029	6200	51
H(26B)	3074	7551	6619	51
H(26C)	2818	7882	5848	51
H(27A)	4331	7418	5934	53
H(27B)	4242	6832	5491	53
H(27C)	4000	6922	6400	53
H(28A)	3884	7892	4807	47
H(28B)	3216	7769	4511	47
H(28C)	3738	7336	4314	47
H(30A)	3763	5416	5253	52
H(30B)	3343	5624	5981	52
H(30C)	3925	5972	5739	52
H(31A)	3810	5895	3975	53
H(31B)	3948	6483	4388	53
H(31C)	3394	6418	3779	53
H(32A)	2863	5392	4337	44
H(32B)	2500	5950	4156	44
H(32C)	2430	5645	5018	44
H(34A)	987	6929	3254	30
H(35A)	269	7580	2656	50
H(38A)	1603	8202	4620	36
H(34B)	959	7262	3307	42
H(37A)	1388	9046	4360	55
H(38B)	1966	8251	4795	42
H(39A)	2983	4473	8973	182
H(39B)	2878	3811	8928	182

H(40A)	2502	4628	7868	189
H(40B)	2741	4019	7660	189
H(41A)	1711	4261	7507	210
H(41B)	1906	3654	7827	210
H(42A)	1413	3858	8793	87
H(42B)	1445	4505	8597	87
H(43A)	4319	4944	6732	474
H(43B)	4752	5320	7241	474
H(43C)	4840	4675	7234	474
H(44A)	3904	4619	8011	339
H(44B)	3840	5288	7932	339
H(45A)	5055	4826	8588	285
H(45B)	4841	5455	8732	285
H(46A)	4289	4542	9529	453
H(46B)	4148	5175	9588	453
H(47A)	4717	4947	10542	500
H(47B)	5176	4731	9894	500
H(47C)	5034	5367	9953	500