Catalytic Copolymerization of CO and Ethylene with a Charge Neutral Palladium(II) Zwitterion

Supporting Information

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Experimental Section

All manipulations were carried out using standard Schlenk or glovebox techniques under a dinitrogen atmosphere. Unless otherwise noted, solvents were deoxygenated and dried by thorough sparging with N_2 gas, followed by passage through an activated alumina column. Diethyl ether, tetrahydrofuran, petroleum ether, and benzene were typically tested with a standard purple solution of sodium benzophenone ketyl in tetrahydrofuran in order to confirm effective oxygen and moisture removal. Deuterated acetone and benzene were purchased from Cambridge Isotope Laboratories, Inc. The solvents were dried over activated 3 Å molecular sieves and degassed by repeated freeze-pump-thaw cycles prior to use. The preparations of (tmeda)$PdMe_2$,[1,2] (dppp)$PdMe_2$,[3] [ASN][$Ph_2B(CH_2PPh_2)_2$],[4] and [$H(Et_2O)_2$][$B(C_6F_5)_4$][5] were carried out following literature procedures. CP grade ethylene and CO were used without further purification for polymerization. Elemental analyses were performed by Desert Analytics, Tucson, Az. A Varian Mercury-300 NMR spectrometer and a Varian Inova-500 NMR spectrometer was used to record ^{1}H and ^{13}C NMR spectra unless otherwise stated. ^{1}H and ^{13}C NMR chemical shifts were referenced to residual solvent. Proton peaks were assigned based on TOCSY-1D and ^{31}P-decoupled ^{1}H NMR studies. ^{31}P NMR chemical shifts are reported relative to an external standard of 85% H_3PO_4. IR measurements were obtained using a Bio Rad Excalibur FTS 3000 with a KBr solution cell. X-ray diffraction experiments were performed in the Beckman Institute Crystallographic Facility on a Bruker Smart 1000 CCD diffractometer. Polymer analyses were performed by MALDI-TOF MS on an Applied Biosystems Voyager DE-PRO with a matrix of 2-(4-hydroxyphenylazo)-benzoic acid in hexafluoroisopropanol with sodium iodide/ethanol [25 kV, 150 shots, linear, positive ion mode].

[ASN][$Ph_2B(CH_2PPh_2)_2PdMe_2$] (1). A benzene solution of (tmeda)$PdMe_2$ (0.466 g, 1.85 mmol) was added dropwise to a stirring suspension of [ASN][$Ph_2B(CH_2PPh_2)_2$] (1.273 g, 1.85 mmol) in acetonitrile (20 mL). Within 30 minutes, the reaction turned from cloudy white to a transparent yellow. The reaction was stirred overnight, and all volatiles were then removed in vacuo to give a mixture of off-white and yellow solids. The residue was submerged in Et_2O and stirred vigorously for 30 min to afford a fine powder. The solids were collected on a frit, washed with Et_2O, then with 15 mL Et_2O/CH_3CN (4:1), and dried in vacuo to give white **1** (1.284 g, 84% yield). ^{1}H NMR (d^6-acetone, 500 MHz) δ 7.35 (m, 8H, H_o of PPh_2), 7.11 (app t, J = 8.0 Hz, 4H, H_p of PPh_2), 7.05 (app t, J = 8.0 Hz, 8H, H_m of PPh_2), 6.95 (br s, 4H, H_o of BPh_2), 6.66 (app t, J = 7.0 Hz, 4H, H_m of BPh_2), 6.60 (tt, J = 1.0 and 7.0 Hz, 2H, H_p of BPh_2), 3.67 (m, 8H, $N(CH_2CH_2)_2$), 2.24 (m, 8H, $N(CH_2CH_2)_2$), 1.79 (br s, 4H, CH_2PPh_2), -0.18 (d, J = 0.5 Hz, 6H, $Pd(CH_3)_2$); ^{13}C NMR (d^6-acetone, 125 MHz) δ 166.8 (br, C_{ipso} of BPh_2), 141.2 (app t, J = 13.8 Hz, C_{ipso} of PPh_2), 134.4 (t, J = 5.3 Hz, C_o of PPh_2), 133.6 (C_o of BPh_2), 128.1 (C_m of BPh_2), 127.6 (t, J = 4.0 Hz, C_m of PPh_2), 126.3 (C_p of PPh_2), 122.1 (C_p of BPh_2), 63.8 (t, J = 3.1 Hz, $N(CH_2CH_2)_2$), 22.8 ($N(CH_2CH_2)_2$), 22.3 (br, CH_2PPh_2), 5.61 (br dd, $Pd(CH_3)_2$); ^{31}P NMR (d^6-acetone, 121 MHz) δ 22.4. Anal. Calcd for ($C_{48}H_{56}BNP_2Pd$): C 69.78; H 6.83; N 1.70. Found: C 69.69; H 6.91; N 1.62.

$Ph_2B(CH_2PPh_2)_2PdMe(THF)$ (2). A THF solution of [$HNEt^iPr_2$][BPh_4] (43.2 mg, 96.1 μmol) was added to a stirring THF solution of **1** (79.4 mg, 96.1 μmol). The reaction solution immediately turned cloudy pink. After stirring for 30 min, the reaction mixture was filtered through a glass wool pipette to remove white solids. A small amount of petroleum ether (5 mL) was added to the light pink filtrate to further precipitate the white solids. After filtering, the cycle was repeated until all of the [ASN][BPh_4] was removed (^{1}H NMR). Finally, copious petroleum ether (7 mL) was added to precipitate a light peach solid, which was collected and dried in vacuo to give 49.7 mg of product (71% yield). ^{1}H NMR (THF-d_8, 300 MHz, -70 °C) δ 7.40 - 7.25 (m, 12H, aryl H's of PPh_2 and P′Ph_2), 7.16 (app t, *J* = 7.20 Hz, 8H, aryl H's of PPh_2 and P′Ph_2), 6.68 (m, 4H, H_o of BPh_2), 6.59 - 6.57 (m, 6H, H_m and H_p of BPh_2), 3.62 (m, 4H, $(CH_2CH_2)_2O$), 2.01 (dd, *J* = 4.20 and 16.49 Hz, 2H, CH_2PPh_2), 1.82 - 1.76 (m, 6H, $(CH_2CH_2)_2O$ and C′H_2PPh_2), -0.12 (d, *J* = 7.80 Hz, 3H, $PdCH_3$); ^{31}P NMR (THF-d_8, 121 MHz, -70 °C) δ 47.1 (d, *J* = 47 Hz, P trans to Me), 19.1 (d, *J* = 47 Hz, P trans to THF). Anal. Calcd for ($C_{43}H_{45}BOP_4Pd$): C 68.23; H 5.99; N 0.00. Found: C 68.01; H 5.84; N 0.18.

[$Ph_2B(CH_2PPh_2)_2$]Pd(C(O)Me)(CO) (3). Obtaining spectroscopic data for the CO insertion product **3** was difficult due to the ease of formation of dimeric **4** at concentrations suitable for NMR analysis. However, when THF solvent was first sparged with CO gas, followed by dissolution of pale peach **2** in the CO-containing solvent, a rapid color change to yellow was observed. IR analysis of the solution showed two intense ν(CO) bands at 2108 and 1694 cm^{-1}, indicative of the CO insertion product **3**. Compound **3** reverts back to starting material **2** in the absence of a CO atmosphere. Spectroscopic NMR data was obtained by generating **3** in situ as described above CD_2Cl_2. ^{1}H NMR (CD_2Cl_2, 300 MHz) δ 7.41 - 7.32 (m, 6H, aryl H's), 7.27 – 7.02 (m, 18H, aryl H's), 6.93 – 6.82 (m, 6H, H_m and H_p of BPh_2), 3.68 (m, 4H, free THF), 2.07 (br d, *J* = 15 Hz, 2H, CH_2PPh_2), 1.95 (s, 3H, C(O)*Me*), 1.83 (m, 4H, free THF), 1.72 (br d, 2H, C′H_2P′Ph_2); ^{31}P NMR (CD_2Cl_2, 121 MHz) δ 22.3 (d, *J* = 71.4 Hz), 11.0 (d, *J* = 71.4 Hz); IR(CH_2Cl_2, KBr): ν(CO) = 2110 cm^{-1}, ν(*C*(*O*)Me) 1686 cm^{-1}.

{[$Ph_2B(CH_2PPh_2)_2$]Pd}$_2$ (4). A THF solution (5 mL) of compound **2** (39.8 mg, 52.6 μmol) was sparged with ethylene for 1 h. The dark red solution was concentrated in vacuo, and petroleum ether (5 mL) was added to precipitate red-orange solids (34 mg, 99%). Single crystals suitable for X-ray diffraction studies were grown from vapor diffusion of petroleum ether into a CH_2Cl_2/CH_3CN solution of **4** at –30 ºC. ^{1}H NMR (C_6D_6, 500 MHz) δ 7.46 (br d, *J* = 7.0 Hz, 4H, H_o of BPh_2), 7.23 (t, *J* = 7.50 Hz, 4H, H_m of BPh_2), 7.17 (t, *J* = 7.50 Hz, 2H, H_p of BPh_2), 7.05 (dd, *J* = 7.50 and 10.50 Hz, 4H, H_o of PPh_2), 6.87 (m, 2H, H_p of P′Ph_2), 6.75 (m, 8H, H_m of P′Ph_2, H_o of P′Ph_2), 6.66 (t, *J* = 7.00 Hz, 4H, H_m of PPh_2), 6.53 (t, *J* = 7.50 Hz, 2H, H_p of PPh_2), 2.24 (br d, *J* = 15.50 Hz, 2H, CH_2PPh_2), 1.97 (br d, *J* = 10.50 Hz, 2H, C′H_2P′Ph_2); ^{13}C NMR (C_6D_6, 125 MHz) δ 163.2 (br s, C_{ipso} of BPh_2), 134.9 (t, *J* = 16.0 Hz, C_{ipso} of PPh_2), 133.4 (m, C_o of BPh_2 , C_o of PPh_2), 131.7 (C_p of P′Ph_2), 130.6 (m, C_m of P′Ph_2), 129.4 (m, C_m of PPh_2), 128.9 (C_p of PPh_2), 127.5 (app t, *J* = 4.5 Hz, C_o of P′Ph_2), 127.2 (C_m of BPh_2), 123.4 (C_p of BPh_2), 112.4 (app d, *J* = 49.7 Hz, C_{ipso} of P′Ph_2), 25.3 (br, CH_2PPh_2), 21.1 (br, C′H_2P′Ph_2); ^{31}P NMR (THF, 121 MHz) δ 29.9 (d, *J* = 55 Hz), 11.3 (d, *J* = 55 Hz). Anal. Calcd for ($C_{76}H_{68}B_2P_4Pd_2$): C 68.13; H 5.11; N 0.00. Found: C 68.68; H 4.75; N 0.21.

$Ph_2Si(CH_2PPh_2)_2PdMe_2$. The compound (tmeda)$PdMe_2$ (0.190 g, 0.752 mmol) was dissolved in cold benzene (5 mL) and added dropwise to a cold (-30 °C) stirring suspension of $Ph_2Si(CH_2PPh_2)_2$ (0.437 g, 0.752 mmol) in acetonitrile (15 mL). Within ten minutes, the solution became homogeneous. After stirring overnight, all volatiles were removed in vacuo to give an off-white powder. The solids were collected on a frit, washed liberally with petroleum ether, and dried in vacuo to give an off-white powder (0.404 g, 75% yield). ^{1}H NMR (C_6D_6, 500 MHz) δ 7.55 (tt, *J* = 8.50 and 1.50 Hz, 8H, H_o of PPh_2), 7.04 - 6.93 (m, 14H, aryl H's of $SiPh_2$, H_p of PPh_2), 6.88 (br d, *J* = 4.00 Hz, 8H, H_m of PPh_2), 2.01 (br d, *J* = 7.50 Hz, 4H, CH_2PPh_2), 0.925 (d, *J* = 3.00 Hz, 6H, $Pd(CH_3)_2$); ^{13}C NMR (C_6D_6, 125 MHz) δ 136.7 (app t, *J* = 15 Hz, C_{ipso} of PPh_2), 136.4 (m, C_{ipso} of $SiPh_2$), 134.7, 134.1 (t, *J* = 5.91 Hz), 129.9, 129.6, 128.5 (m), 11.1 (br, CH_2PPh_2), 8.36 (dd, *J* = 14.95 and 95.5 Hz, $Pd(CH_3)_2$); ^{13}C NMR (d^6-acetone, 125 MHz) δ 136.9 - 136.6 (m, C_{ipso} of $SiPh_2$, C_{ipso} of PPh_2), 134.8 (C_o of $SiPh_2$), 134.2 (t, *J* = 6.4 Hz, C_o of PPh_2), 130.3 (C_p of $SiPh_2$), 129.9 (C_p of PPh_2), 128.7 (t, *J* = 4.3 Hz, C_m of PPh_2), 128.5 (C_m of $SiPh_2$), 10.8 (t, *J* = 5.0 Hz, CH_2PPh_2), 7.16 (dd, *J* = 97.3 and 15.0 Hz, $Pd(CH_3)_2$); ^{31}P NMR (C_6D_6, 121 MHz) δ 11.63. Anal. Calcd for ($C_{40}H_{40}P_2PdSi$): C 66.99; H 5.62; N 0.00. Found: C 66.70; H 5.75; N 0.13.

Generation of cationic catalysts 7 and 8. To a THF solution of the catalyst precursors (dppp)$PdMe_2$ (25.5 mg, 4.65 x10^{-5} mol) or $Ph_2Si(CH_2PPh_2)_2PdMe_2$ (33.3 mg, 4.65 x10^{-5} mol) was added $[H(Et_2O)_2][B(C_6F_5)_4]$ (38.5 mg, 4.65 x10^{-5} mol). Consumption of the starting neutral precursors was verified by ^{31}P NMR, which also established clean conversion to the cationic catalysts. Compound **7**: ^{31}P NMR (THF, 121 MHz) δ 33.1 (d, *J* = 40 Hz), 6.1 (d, *J* = 40 Hz). Compound **8**: δ 33.2 (d, *J* = 53 Hz), 0.5 (d, *J* = 53 Hz).

Ethylene and CO copolymerization studies. In a typical polymerization experiment, 9.3 x10^{-6} mol of palladium catalyst (**2**, **7**, or **8)** in 10 mL of THF, obtained from stock solutions, were transferred to a small bottleneck glass vessel with a 1 ¼" teflon stir bar. The glass vessels were placed inside a Parr reactor, which was then charged with 100 psi CO and 100 psi ethylene. After vigorously stirring for one hour at 23 °C, the reactions were quenched with MeOH. A white precipitate was collected, washed with MeOH, and thoroughly dried in vacuo. A total of 8 polymerization experiments were performed for each catalyst system to establish reliable polymer weights for TON value comparisons.

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Figure 1. Fully labeled drawing of Compound 4. Hydrogens have been omitted for clarity.

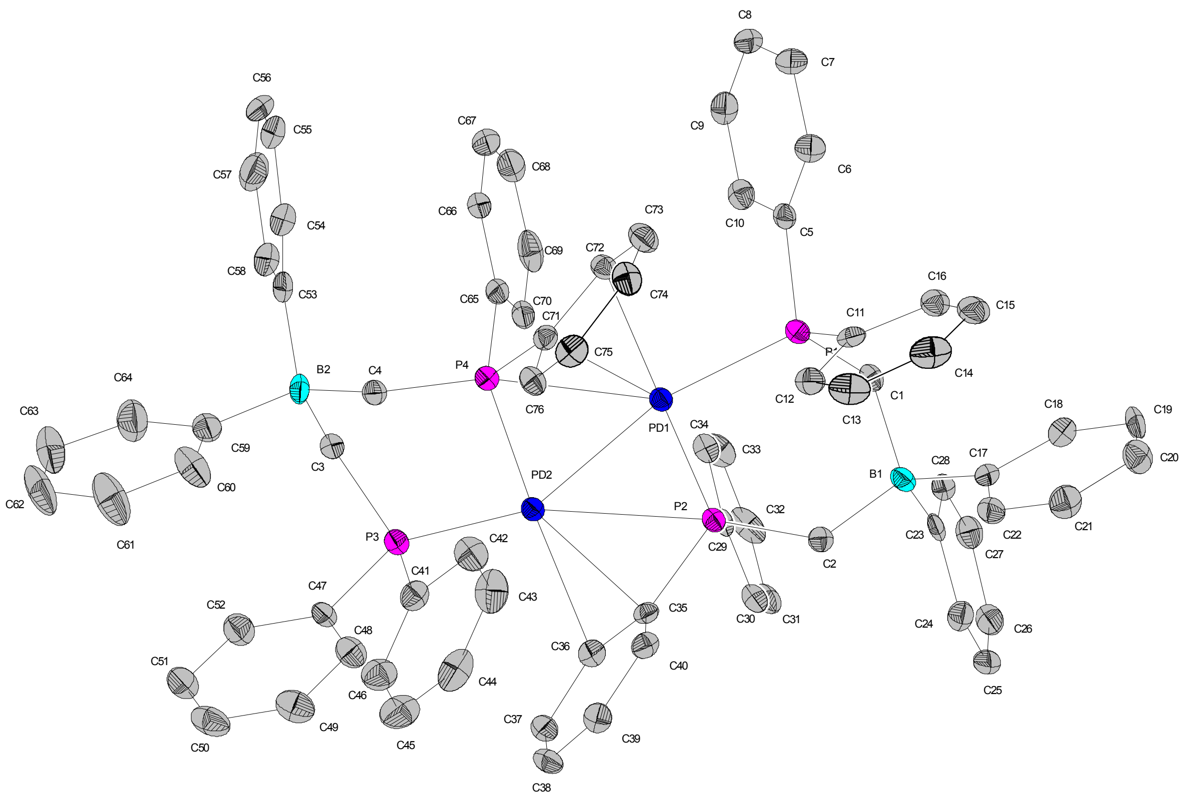

Table 1. Crystal data and structure refinement for CCL04 (CCDC 168698).

Empirical formula	$C_{76}H_{68}B_2P_4Pd_2 \cdot 3CH_2Cl_2$
Formula weight	1594.38
Crystallization Solvent	Dichloromethane
Crystal Habit	Plate
Crystal size	0.25 x 0.22 x 0.08 mm^3
Crystal color	Red-orange

Data Collection

Preliminary Photos	Rotation	
Type of diffractometer	CCD area detector	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	98(2) K	
θ range for 30493 reflections used in lattice determination	2.18 to 28.41°	
Unit cell dimensions	a = 22.389(3) Å	
	b = 20.690(2) Å	β= 106.779(2)°
	c = 16.3816(18) Å	
Volume	7265.2(14) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Density (calculated)	1.458 Mg/m^3	
F(000)	3248	
θ range for data collection	1.37 to 28.67°	
Completeness to θ = 28.67°	94.0 %	
Index ranges	$-29 \leq h \leq 28, -27 \leq k \leq 27, -21 \leq l \leq 21$	
Data collection scan type	ω scans at 6 ϕ settings	
Reflections collected	118272	
Independent reflections	17601 [R_{int}= 0.0819]	
Absorption coefficient	0.848 mm^{-1}	
Absorption correction	None	
Max. and min. transmission (calculated)	0.9368 and 0.8148	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Patterson method
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric sites
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	17601 / 64 / 846
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.522
Final R indices [$I>2\sigma(I)$, 12256 reflections]	R1 = 0.0606, *w*R2 = 0.1173
R indices (all data)	R1 = 0.0934, *w*R2 = 0.1207
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.007
Average shift/error	0.000
Largest diff. peak and hole	3.424 and -2.284 e.Å^{-3}

Special Refinement Details

The crystals contain solvent in three regions. One region contains a single ordered dichloromethane molecule, Cl(1)-C(77)-Cl(2). This molecule was refined with anisotropic displacement parameters. A second site contains two disordered molecules of dichloromethane, Cl(3)-C(78)-Cl(4) and Cl(5)-C(79)-Cl(6). These molecules were refined with isotropic displacement parameters and the geometry restrained to be similar to the geometry of the molecule in the ordered site. A further restraint was imposed to require the occupancy of these dichloromethane molecules sum to one. The third site contains four molecules of dichloromethane, Cl(7)-C(80)-Cl(8), Cl(9)-C(81)-Cl(10), Cl(11)-C(82)-Cl(12) and Cl(13)-C(83)-Cl(14). The same restraints were applied. All residual electron density greater than 1e^-/Å^3 is in this area.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for CCL04 (CCDC 168698). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Pd(1)	2227(1)	3736(1)	3555(1)	14(1)	1
Pd(2)	2887(1)	4824(1)	3446(1)	14(1)	1
P(1)	1962(1)	2797(1)	4157(1)	14(1)	1
P(2)	3231(1)	3658(1)	4295(1)	14(1)	1
P(3)	3029(1)	5948(1)	3393(1)	16(1)	1
P(4)	1854(1)	4911(1)	2893(1)	15(1)	1
B(1)	3221(3)	2379(3)	5100(4)	16(1)	1
B(2)	1737(3)	6294(3)	2450(4)	17(1)	1
C(1)	2522(2)	2601(2)	5163(3)	16(1)	1
C(2)	3433(2)	2810(2)	4355(3)	15(1)	1
C(3)	2329(2)	6400(2)	3337(3)	18(1)	1
C(4)	1727(2)	5542(2)	2096(3)	17(1)	1
C(5)	1213(2)	2913(2)	4366(3)	14(1)	1
C(6)	694(2)	2530(2)	4022(3)	20(1)	1
C(7)	119(2)	2678(3)	4147(3)	24(1)	1
C(8)	59(2)	3208(2)	4637(3)	23(1)	1
C(9)	577(3)	3586(2)	5000(3)	21(1)	1
C(10)	1146(2)	3446(2)	4862(3)	19(1)	1
C(11)	1884(2)	2093(2)	3467(3)	16(1)	1
C(12)	2000(2)	2161(2)	2682(3)	20(1)	1
C(13)	1965(3)	1638(3)	2154(3)	26(1)	1
C(14)	1809(3)	1039(3)	2394(4)	29(1)	1
C(15)	1704(2)	954(3)	3164(4)	25(1)	1
C(16)	1745(2)	1473(2)	3704(3)	19(1)	1
C(17)	3193(2)	1604(2)	4892(3)	16(1)	1
C(18)	3059(2)	1180(2)	5477(3)	20(1)	1
C(19)	3008(3)	521(2)	5351(3)	23(1)	1
C(20)	3091(3)	248(2)	4626(3)	25(1)	1
C(21)	3241(3)	649(2)	4042(4)	25(1)	1
C(22)	3288(2)	1314(2)	4170(3)	20(1)	1
C(23)	3754(2)	2483(2)	6018(3)	16(1)	1
C(24)	4384(2)	2365(2)	6087(3)	20(1)	1
C(25)	4863(2)	2454(2)	6817(3)	22(1)	1
C(26)	4727(2)	2683(2)	7544(3)	22(1)	1
C(27)	4117(2)	2786(2)	7515(3)	21(1)	1
C(28)	3640(2)	2687(2)	6768(3)	17(1)	1
C(29)	3476(2)	3983(2)	5366(3)	15(1)	1
C(30)	4105(2)	3958(2)	5839(3)	18(1)	1
C(31)	4288(3)	4177(2)	6675(3)	21(1)	1
C(32)	3853(3)	4425(2)	7035(3)	25(1)	1
C(33)	3229(3)	4451(2)	6574(3)	25(1)	1
C(34)	3038(3)	4231(2)	5747(3)	19(1)	1
C(35)	3719(2)	4068(2)	3761(3)	14(1)	1
C(36)	4024(2)	4652(2)	4049(3)	17(1)	1
C(37)	4419(2)	4927(2)	3617(3)	19(1)	1
C(38)	4495(2)	4639(2)	2890(3)	21(1)	1
C(39)	4159(2)	4079(2)	2566(3)	19(1)	1

C(40)	3778(2)	3801(2)	2987(3)	17(1)	1
C(41)	3606(2)	6208(2)	4377(3)	20(1)	1
C(42)	3511(3)	6007(3)	5142(3)	26(1)	1
C(43)	3936(3)	6158(3)	5919(4)	32(2)	1
C(44)	4459(3)	6512(3)	5943(4)	30(2)	1
C(45)	4557(3)	6713(3)	5192(4)	33(2)	1
C(46)	4132(3)	6561(3)	4413(4)	27(1)	1
C(47)	3307(2)	6264(2)	2539(3)	17(1)	1
C(48)	3460(2)	5849(3)	1973(3)	22(1)	1
C(49)	3659(3)	6085(3)	1295(3)	28(1)	1
C(50)	3691(3)	6747(3)	1178(3)	30(1)	1
C(51)	3532(3)	7166(3)	1738(4)	30(1)	1
C(52)	3337(3)	6927(2)	2412(3)	25(1)	1
C(53)	1073(3)	6443(2)	2642(3)	22(1)	1
C(54)	507(3)	6200(2)	2115(4)	26(1)	1
C(55)	-56(3)	6294(3)	2291(4)	33(2)	1
C(56)	-84(3)	6652(3)	2981(4)	34(2)	1
C(57)	448(3)	6929(3)	3480(4)	34(2)	1
C(58)	1019(3)	6819(2)	3320(4)	25(1)	1
C(59)	1799(2)	6802(2)	1717(3)	22(1)	1
C(60)	1904(3)	6652(3)	951(4)	34(2)	1
C(61)	1960(3)	7120(3)	369(4)	46(2)	1
C(62)	1900(3)	7766(3)	547(4)	44(2)	1
C(63)	1783(3)	7941(3)	1293(4)	43(2)	1
C(64)	1740(3)	7464(3)	1850(4)	36(2)	1
C(65)	1429(2)	5067(2)	3668(3)	17(1)	1
C(66)	791(2)	5084(2)	3458(3)	20(1)	1
C(67)	492(3)	5221(2)	4070(4)	27(1)	1
C(68)	836(3)	5346(2)	4901(4)	28(1)	1
C(69)	1473(3)	5325(2)	5126(4)	26(1)	1
C(70)	1776(3)	5191(2)	4514(3)	20(1)	1
C(71)	1494(2)	4191(2)	2340(3)	15(1)	1
C(72)	1106(2)	3777(2)	2636(3)	16(1)	1
C(73)	826(2)	3239(2)	2134(3)	24(1)	1
C(74)	937(2)	3111(2)	1377(3)	22(1)	1
C(75)	1342(2)	3506(2)	1093(3)	22(1)	1
C(76)	1621(2)	4025(2)	1561(3)	19(1)	1
Cl(1)	4966(1)	1130(1)	4526(1)	50(1)	1
Cl(2)	5349(1)	670(1)	6280(1)	52(1)	1
C(77)	4719(2)	774(3)	5358(3)	36(2)	1
Cl(3)	2973(4)	9157(3)	2500(4)	96(1)	0.354(3)
Cl(4)	2816(4)	8332(3)	3867(4)	96(1)	0.354(3)
C(78)	2611(11)	8456(8)	2760(5)	96(1)	0.354(3)
Cl(5)	2446(2)	9278(2)	2703(2)	96(1)	0.646(3)
Cl(6)	3148(2)	8315(2)	3903(2)	96(1)	0.646(3)
C(79)	3051(5)	8693(6)	2914(6)	96(1)	0.646(3)
Cl(7)	-133(3)	9618(4)	4525(5)	44(1)	0.206(3)
Cl(8)	1134(3)	10034(4)	4654(6)	44(1)	0.206(3)
C(80)	396(6)	9851(16)	3957(5)	44(1)	0.206(3)

Cl(9)	38(3)	-374(4)	4043(5)	44(1)	0.218(3)
Cl(10)	1375(3)	-130(4)	4662(5)	44(1)	0.218(3)
C(81)	642(4)	46(11)	4796(13)	44(1)	0.218(3)
Cl(11)	749(5)	9047(5)	2434(5)	131(2)	0.329(3)
Cl(12)	716(5)	9460(5)	4121(5)	131(2)	0.329(3)
C(82)	1062(14)	8933(13)	3548(6)	131(2)	0.329(3)
Cl(13)	1000(6)	9544(5)	2041(7)	131(2)	0.253(3)
Cl(14)	900(6)	8525(5)	3221(8)	131(2)	0.253(3)
C(83)	497(7)	8986(19)	2330(20)	131(2)	0.253(3)

Table 3. Bond lengths [Å] and angles [°] for CCL04 (CCDC 168698).

Pd(1)-P(2)	2.2316(14)	C(13)-H(13)	0.9500
Pd(1)-P(1)	2.3308(13)	C(14)-C(15)	1.361(7)
Pd(1)-P(4)	2.6945(14)	C(14)-H(14)	0.9500
Pd(1)-Pd(2)	2.7281(6)	C(15)-C(16)	1.377(7)
Pd(2)-P(4)	2.2351(14)	C(15)-H(15)	0.9500
Pd(2)-P(3)	2.3513(13)	C(16)-H(16)	0.9500
Pd(2)-P(2)	2.7808(13)	C(17)-C(18)	1.395(6)
P(1)-C(1)	1.805(5)	C(17)-C(22)	1.397(7)
P(1)-C(11)	1.821(5)	C(18)-C(19)	1.377(6)
P(1)-C(5)	1.822(5)	C(18)-H(18)	0.9500
P(2)-C(35)	1.797(5)	C(19)-C(20)	1.377(7)
P(2)-C(2)	1.807(5)	C(19)-H(19)	0.9500
P(2)-C(29)	1.810(5)	C(20)-C(21)	1.379(7)
P(3)-C(3)	1.804(5)	C(20)-H(20)	0.9500
P(3)-C(47)	1.811(5)	C(21)-C(22)	1.391(7)
P(3)-C(41)	1.831(5)	C(21)-H(21)	0.9500
P(4)-C(71)	1.808(5)	C(22)-H(22)	0.9500
P(4)-C(4)	1.811(5)	C(23)-C(28)	1.390(7)
P(4)-C(65)	1.821(5)	C(23)-C(24)	1.405(7)
B(1)-C(17)	1.636(7)	C(24)-C(25)	1.369(7)
B(1)-C(23)	1.642(8)	C(24)-H(24)	0.9500
B(1)-C(1)	1.664(7)	C(25)-C(26)	1.395(7)
B(1)-C(2)	1.686(7)	C(25)-H(25)	0.9500
B(2)-C(59)	1.631(7)	C(26)-C(27)	1.371(7)
B(2)-C(53)	1.634(8)	C(26)-H(26)	0.9500
B(2)-C(4)	1.659(7)	C(27)-C(28)	1.387(7)
B(2)-C(3)	1.675(7)	C(27)-H(27)	0.9500
C(1)-H(1A)	0.9900	C(28)-H(28)	0.9500
C(1)-H(1B)	0.9900	C(29)-C(30)	1.399(7)
C(2)-H(2A)	0.9900	C(29)-C(34)	1.404(7)
C(2)-H(2B)	0.9900	C(30)-C(31)	1.388(7)
C(3)-H(3A)	0.9900	C(30)-H(30)	0.9500
C(3)-H(3B)	0.9900	C(31)-C(32)	1.374(7)
C(4)-H(4A)	0.9900	C(31)-H(31)	0.9500
C(4)-H(4B)	0.9900	C(32)-C(33)	1.384(7)
C(5)-C(6)	1.385(7)	C(32)-H(32)	0.9500
C(5)-C(10)	1.404(6)	C(33)-C(34)	1.375(7)
C(6)-C(7)	1.395(7)	C(33)-H(33)	0.9500
C(6)-H(6)	0.9500	C(34)-H(34)	0.9500
C(7)-C(8)	1.387(7)	C(35)-C(36)	1.402(6)
C(7)-H(7)	0.9500	C(35)-C(40)	1.424(6)
C(8)-C(9)	1.383(7)	C(36)-C(37)	1.403(6)
C(8)-H(8)	0.9500	C(36)-H(36)	0.9500
C(9)-C(10)	1.385(7)	C(37)-C(38)	1.385(7)
C(9)-H(9)	0.9500	C(37)-H(37)	0.9500
C(10)-H(10)	0.9500	C(38)-C(39)	1.399(7)
C(11)-C(12)	1.391(7)	C(38)-H(38)	0.9500
C(11)-C(16)	1.401(6)	C(39)-C(40)	1.370(7)
C(12)-C(13)	1.372(7)	C(39)-H(39)	0.9500
C(12)-H(12)	0.9500	C(40)-H(40)	0.9500
C(13)-C(14)	1.376(7)	C(41)-C(46)	1.373(7)

C(41)-C(42)	1.394(7)	C(71)-C(72)	1.403(6)
C(42)-C(43)	1.388(7)	C(71)-C(76)	1.425(7)
C(42)-H(42)	0.9500	C(72)-C(73)	1.417(7)
C(43)-C(44)	1.372(8)	C(72)-H(72)	0.9500
C(43)-H(43)	0.9500	C(73)-C(74)	1.360(7)
C(44)-C(45)	1.375(8)	C(73)-H(73)	0.9500
C(44)-H(44)	0.9500	C(74)-C(75)	1.396(7)
C(45)-C(46)	1.390(7)	C(74)-H(74)	0.9500
C(45)-H(45)	0.9500	C(75)-C(76)	1.362(7)
C(46)-H(46)	0.9500	C(75)-H(75)	0.9500
C(47)-C(48)	1.377(7)	C(76)-H(76)	0.9500
C(47)-C(52)	1.391(7)	Cl(1)-C(77)	1.772(5)
C(48)-C(49)	1.398(7)	Cl(2)-C(77)	1.757(5)
C(48)-H(48)	0.9500	C(77)-H(77A)	0.9900
C(49)-C(50)	1.388(7)	C(77)-H(77B)	0.9900
C(49)-H(49)	0.9500	Cl(3)-C(78)	1.772(5)
C(50)-C(51)	1.382(7)	Cl(4)-C(78)	1.756(5)
C(50)-H(50)	0.9500	C(78)-H(78A)	0.9900
C(51)-C(52)	1.391(7)	C(78)-H(78B)	0.9900
C(51)-H(51)	0.9500	Cl(5)-C(79)	1.773(5)
C(52)-H(52)	0.9500	Cl(6)-C(79)	1.756(5)
C(53)-C(58)	1.392(7)	C(79)-H(79A)	0.9900
C(53)-C(54)	1.405(7)	C(79)-H(79B)	0.9900
C(54)-C(55)	1.386(7)	Cl(7)-C(80)	1.772(5)
C(54)-H(54)	0.9500	Cl(7)-Cl(7)#1	2.176(16)
C(55)-C(56)	1.368(8)	Cl(8)-C(80)	1.757(5)
C(55)-H(55)	0.9500	C(80)-H(80A)	0.9900
C(56)-C(57)	1.362(8)	C(80)-H(80B)	0.9900
C(56)-H(56)	0.9500	Cl(9)-C(81)	1.772(5)
C(57)-C(58)	1.394(7)	Cl(10)-C(81)	1.757(5)
C(57)-H(57)	0.9500	C(81)-H(81A)	0.9900
C(58)-H(58)	0.9500	C(81)-H(81B)	0.9900
C(59)-C(60)	1.375(7)	Cl(11)-C(82)	1.772(5)
C(59)-C(64)	1.400(7)	Cl(12)-C(82)	1.757(5)
C(60)-C(61)	1.391(7)	C(82)-H(82A)	0.9900
C(60)-H(60)	0.9500	C(82)-H(82B)	0.9900
C(61)-C(62)	1.383(8)	Cl(13)-C(83)	1.772(5)
C(61)-H(61)	0.9500	Cl(14)-C(83)	1.756(5)
C(62)-C(63)	1.371(8)	C(83)-H(83A)	0.9900
C(62)-H(62)	0.9500	C(83)-H(83B)	0.9900
C(63)-C(64)	1.367(7)		
C(63)-H(63)	0.9500	P(2)-Pd(1)-P(1)	92.83(5)
C(64)-H(64)	0.9500	P(2)-Pd(1)-P(4)	115.50(5)
C(65)-C(66)	1.370(7)	P(1)-Pd(1)-P(4)	146.60(5)
C(65)-C(70)	1.402(7)	P(2)-Pd(1)-Pd(2)	67.35(3)
C(66)-C(67)	1.385(7)	P(1)-Pd(1)-Pd(2)	157.56(4)
C(66)-H(66)	0.9500	P(4)-Pd(1)-Pd(2)	48.68(3)
C(67)-C(68)	1.381(8)	P(4)-Pd(2)-P(3)	92.33(5)
C(67)-H(67)	0.9500	P(4)-Pd(2)-Pd(1)	64.88(4)
C(68)-C(69)	1.368(8)	P(3)-Pd(2)-Pd(1)	154.16(4)
C(68)-H(68)	0.9500	P(4)-Pd(2)-P(2)	112.16(5)
C(69)-C(70)	1.390(7)	P(3)-Pd(2)-P(2)	148.83(5)
C(69)-H(69)	0.9500	Pd(1)-Pd(2)-P(2)	47.78(3)
C(70)-H(70)	0.9500	C(1)-P(1)-C(11)	107.7(2)

C(1)-P(1)-C(5)	107.0(2)	H(2A)-C(2)-H(2B)	107.4
C(11)-P(1)-C(5)	107.0(2)	B(2)-C(3)-P(3)	115.8(3)
C(1)-P(1)-Pd(1)	112.26(16)	B(2)-C(3)-H(3A)	108.3
C(11)-P(1)-Pd(1)	113.25(17)	P(3)-C(3)-H(3A)	108.3
C(5)-P(1)-Pd(1)	109.28(15)	B(2)-C(3)-H(3B)	108.3
C(35)-P(2)-C(2)	107.9(2)	P(3)-C(3)-H(3B)	108.3
C(35)-P(2)-C(29)	103.8(2)	H(3A)-C(3)-H(3B)	107.4
C(2)-P(2)-C(29)	107.9(2)	B(2)-C(4)-P(4)	116.2(3)
C(35)-P(2)-Pd(1)	111.10(16)	B(2)-C(4)-H(4A)	108.2
C(2)-P(2)-Pd(1)	107.22(17)	P(4)-C(4)-H(4A)	108.2
C(29)-P(2)-Pd(1)	118.38(17)	B(2)-C(4)-H(4B)	108.2
C(35)-P(2)-Pd(2)	57.72(15)	P(4)-C(4)-H(4B)	108.2
C(2)-P(2)-Pd(2)	153.95(16)	H(4A)-C(4)-H(4B)	107.4
C(29)-P(2)-Pd(2)	97.12(15)	C(6)-C(5)-C(10)	117.9(5)
Pd(1)-P(2)-Pd(2)	64.87(3)	C(6)-C(5)-P(1)	123.9(4)
C(3)-P(3)-C(47)	105.1(2)	C(10)-C(5)-P(1)	118.1(4)
C(3)-P(3)-C(41)	105.9(2)	C(5)-C(6)-C(7)	121.1(5)
C(47)-P(3)-C(41)	105.2(2)	C(5)-C(6)-H(6)	119.5
C(3)-P(3)-Pd(2)	112.95(16)	C(7)-C(6)-H(6)	119.5
C(47)-P(3)-Pd(2)	117.77(17)	C(8)-C(7)-C(6)	120.4(5)
C(41)-P(3)-Pd(2)	109.04(17)	C(8)-C(7)-H(7)	119.8
C(71)-P(4)-C(4)	106.1(2)	C(6)-C(7)-H(7)	119.8
C(71)-P(4)-C(65)	104.4(2)	C(9)-C(8)-C(7)	119.1(5)
C(4)-P(4)-C(65)	111.7(2)	C(9)-C(8)-H(8)	120.4
C(71)-P(4)-Pd(2)	113.36(17)	C(7)-C(8)-H(8)	120.4
C(4)-P(4)-Pd(2)	106.17(17)	C(8)-C(9)-C(10)	120.6(5)
C(65)-P(4)-Pd(2)	114.80(18)	C(8)-C(9)-H(9)	119.7
C(71)-P(4)-Pd(1)	59.99(15)	C(10)-C(9)-H(9)	119.7
C(4)-P(4)-Pd(1)	155.08(17)	C(9)-C(10)-C(5)	121.0(5)
C(65)-P(4)-Pd(1)	92.48(15)	C(9)-C(10)-H(10)	119.5
Pd(2)-P(4)-Pd(1)	66.44(4)	C(5)-C(10)-H(10)	119.5
C(17)-B(1)-C(23)	107.1(4)	C(12)-C(11)-C(16)	117.6(5)
C(17)-B(1)-C(1)	107.7(4)	C(12)-C(11)-P(1)	118.9(4)
C(23)-B(1)-C(1)	110.8(4)	C(16)-C(11)-P(1)	123.4(4)
C(17)-B(1)-C(2)	111.7(4)	C(13)-C(12)-C(11)	120.8(5)
C(23)-B(1)-C(2)	108.2(4)	C(13)-C(12)-H(12)	119.6
C(1)-B(1)-C(2)	111.4(4)	C(11)-C(12)-H(12)	119.6
C(59)-B(2)-C(53)	107.3(4)	C(12)-C(13)-C(14)	120.1(5)
C(59)-B(2)-C(4)	109.9(4)	C(12)-C(13)-H(13)	119.9
C(53)-B(2)-C(4)	108.8(4)	C(14)-C(13)-H(13)	119.9
C(59)-B(2)-C(3)	109.9(4)	C(15)-C(14)-C(13)	120.5(5)
C(53)-B(2)-C(3)	110.2(4)	C(15)-C(14)-H(14)	119.7
C(4)-B(2)-C(3)	110.7(4)	C(13)-C(14)-H(14)	119.7
B(1)-C(1)-P(1)	115.0(3)	C(14)-C(15)-C(16)	119.8(5)
B(1)-C(1)-H(1A)	108.5	C(14)-C(15)-H(15)	120.1
P(1)-C(1)-H(1A)	108.5	C(16)-C(15)-H(15)	120.1
B(1)-C(1)-H(1B)	108.5	C(15)-C(16)-C(11)	121.0(5)
P(1)-C(1)-H(1B)	108.5	C(15)-C(16)-H(16)	119.5
H(1A)-C(1)-H(1B)	107.5	C(11)-C(16)-H(16)	119.5
B(1)-C(2)-P(2)	115.8(3)	C(18)-C(17)-C(22)	115.3(5)
B(1)-C(2)-H(2A)	108.3	C(18)-C(17)-B(1)	118.5(4)
P(2)-C(2)-H(2A)	108.3	C(22)-C(17)-B(1)	126.2(4)
B(1)-C(2)-H(2B)	108.3	C(19)-C(18)-C(17)	122.9(5)
P(2)-C(2)-H(2B)	108.3	C(19)-C(18)-H(18)	118.5

C(17)-C(18)-H(18)	118.5	C(37)-C(36)-H(36)	120.1
C(18)-C(19)-C(20)	120.7(5)	C(38)-C(37)-C(36)	120.6(5)
C(18)-C(19)-H(19)	119.7	C(38)-C(37)-H(37)	119.7
C(20)-C(19)-H(19)	119.7	C(36)-C(37)-H(37)	119.7
C(19)-C(20)-C(21)	118.2(5)	C(37)-C(38)-C(39)	119.9(5)
C(19)-C(20)-H(20)	120.9	C(37)-C(38)-H(38)	120.1
C(21)-C(20)-H(20)	120.9	C(39)-C(38)-H(38)	120.1
C(20)-C(21)-C(22)	120.9(5)	C(40)-C(39)-C(38)	120.0(5)
C(20)-C(21)-H(21)	119.6	C(40)-C(39)-H(39)	120.0
C(22)-C(21)-H(21)	119.6	C(38)-C(39)-H(39)	120.0
C(21)-C(22)-C(17)	122.0(5)	C(39)-C(40)-C(35)	121.1(5)
C(21)-C(22)-H(22)	119.0	C(39)-C(40)-H(40)	119.4
C(17)-C(22)-H(22)	119.0	C(35)-C(40)-H(40)	119.4
C(28)-C(23)-C(24)	114.7(5)	C(46)-C(41)-C(42)	118.2(5)
C(28)-C(23)-B(1)	125.4(5)	C(46)-C(41)-P(3)	124.9(4)
C(24)-C(23)-B(1)	119.8(4)	C(42)-C(41)-P(3)	116.9(4)
C(25)-C(24)-C(23)	124.2(5)	C(43)-C(42)-C(41)	121.0(5)
C(25)-C(24)-H(24)	117.9	C(43)-C(42)-H(42)	119.5
C(23)-C(24)-H(24)	117.9	C(41)-C(42)-H(42)	119.5
C(24)-C(25)-C(26)	118.8(5)	C(44)-C(43)-C(42)	120.0(5)
C(24)-C(25)-H(25)	120.6	C(44)-C(43)-H(43)	120.0
C(26)-C(25)-H(25)	120.6	C(42)-C(43)-H(43)	120.0
C(27)-C(26)-C(25)	119.0(5)	C(43)-C(44)-C(45)	119.5(5)
C(27)-C(26)-H(26)	120.5	C(43)-C(44)-H(44)	120.3
C(25)-C(26)-H(26)	120.5	C(45)-C(44)-H(44)	120.3
C(26)-C(27)-C(28)	120.8(5)	C(44)-C(45)-C(46)	120.6(6)
C(26)-C(27)-H(27)	119.6	C(44)-C(45)-H(45)	119.7
C(28)-C(27)-H(27)	119.6	C(46)-C(45)-H(45)	119.7
C(27)-C(28)-C(23)	122.3(5)	C(41)-C(46)-C(45)	120.7(5)
C(27)-C(28)-H(28)	118.8	C(41)-C(46)-H(46)	119.6
C(23)-C(28)-H(28)	118.8	C(45)-C(46)-H(46)	119.6
C(30)-C(29)-C(34)	119.2(5)	C(48)-C(47)-C(52)	118.8(5)
C(30)-C(29)-P(2)	119.8(4)	C(48)-C(47)-P(3)	120.2(4)
C(34)-C(29)-P(2)	120.8(4)	C(52)-C(47)-P(3)	120.9(4)
C(31)-C(30)-C(29)	119.8(5)	C(47)-C(48)-C(49)	121.0(5)
C(31)-C(30)-H(30)	120.1	C(47)-C(48)-H(48)	119.5
C(29)-C(30)-H(30)	120.1	C(49)-C(48)-H(48)	119.5
C(32)-C(31)-C(30)	120.1(5)	C(50)-C(49)-C(48)	119.6(5)
C(32)-C(31)-H(31)	119.9	C(50)-C(49)-H(49)	120.2
C(30)-C(31)-H(31)	119.9	C(48)-C(49)-H(49)	120.2
C(31)-C(32)-C(33)	120.6(5)	C(51)-C(50)-C(49)	119.7(5)
C(31)-C(32)-H(32)	119.7	C(51)-C(50)-H(50)	120.2
C(33)-C(32)-H(32)	119.7	C(49)-C(50)-H(50)	120.2
C(34)-C(33)-C(32)	120.2(5)	C(50)-C(51)-C(52)	120.2(5)
C(34)-C(33)-H(33)	119.9	C(50)-C(51)-H(51)	119.9
C(32)-C(33)-H(33)	119.9	C(52)-C(51)-H(51)	119.9
C(33)-C(34)-C(29)	120.0(5)	C(51)-C(52)-C(47)	120.6(5)
C(33)-C(34)-H(34)	120.0	C(51)-C(52)-H(52)	119.7
C(29)-C(34)-H(34)	120.0	C(47)-C(52)-H(52)	119.7
C(36)-C(35)-C(40)	118.2(4)	C(58)-C(53)-C(54)	114.8(5)
C(36)-C(35)-P(2)	123.4(4)	C(58)-C(53)-B(2)	123.6(5)
C(40)-C(35)-P(2)	118.4(4)	C(54)-C(53)-B(2)	121.7(5)
C(35)-C(36)-C(37)	119.9(5)	C(55)-C(54)-C(53)	122.4(6)
C(35)-C(36)-H(36)	120.1	C(55)-C(54)-H(54)	118.8

C(53)-C(54)-H(54)	118.8	C(73)-C(72)-H(72)	120.2
C(56)-C(55)-C(54)	120.6(6)	C(74)-C(73)-C(72)	121.0(5)
C(56)-C(55)-H(55)	119.7	C(74)-C(73)-H(73)	119.5
C(54)-C(55)-H(55)	119.7	C(72)-C(73)-H(73)	119.5
C(57)-C(56)-C(55)	118.9(6)	C(73)-C(74)-C(75)	119.8(5)
C(57)-C(56)-H(56)	120.6	C(73)-C(74)-H(74)	120.1
C(55)-C(56)-H(56)	120.6	C(75)-C(74)-H(74)	120.1
C(56)-C(57)-C(58)	120.6(6)	C(76)-C(75)-C(74)	120.7(5)
C(56)-C(57)-H(57)	119.7	C(76)-C(75)-H(75)	119.6
C(58)-C(57)-H(57)	119.7	C(74)-C(75)-H(75)	119.6
C(53)-C(58)-C(57)	122.5(6)	C(75)-C(76)-C(71)	121.1(5)
C(53)-C(58)-H(58)	118.7	C(75)-C(76)-H(76)	119.5
C(57)-C(58)-H(58)	118.7	C(71)-C(76)-H(76)	119.5
C(60)-C(59)-C(64)	114.4(5)	Cl(2)-C(77)-Cl(1)	111.1(3)
C(60)-C(59)-B(2)	126.8(5)	Cl(2)-C(77)-H(77A)	109.4
C(64)-C(59)-B(2)	118.8(5)	Cl(1)-C(77)-H(77A)	109.4
C(59)-C(60)-C(61)	122.7(6)	Cl(2)-C(77)-H(77B)	109.4
C(59)-C(60)-H(60)	118.6	Cl(1)-C(77)-H(77B)	109.4
C(61)-C(60)-H(60)	118.6	H(77A)-C(77)-H(77B)	108.0
C(62)-C(61)-C(60)	119.7(6)	Cl(4)-C(78)-Cl(3)	111.5(3)
C(62)-C(61)-H(61)	120.2	Cl(4)-C(78)-H(78A)	109.3
C(60)-C(61)-H(61)	120.2	Cl(3)-C(78)-H(78A)	109.3
C(63)-C(62)-C(61)	119.9(6)	Cl(4)-C(78)-H(78B)	109.3
C(63)-C(62)-H(62)	120.1	Cl(3)-C(78)-H(78B)	109.3
C(61)-C(62)-H(62)	120.1	H(78A)-C(78)-H(78B)	108.0
C(64)-C(63)-C(62)	118.3(6)	Cl(6)-C(79)-Cl(5)	111.6(3)
C(64)-C(63)-H(63)	120.9	Cl(6)-C(79)-H(79A)	109.3
C(62)-C(63)-H(63)	120.9	Cl(5)-C(79)-H(79A)	109.3
C(63)-C(64)-C(59)	125.0(6)	Cl(6)-C(79)-H(79B)	109.3
C(63)-C(64)-H(64)	117.5	Cl(5)-C(79)-H(79B)	109.3
C(59)-C(64)-H(64)	117.5	H(79A)-C(79)-H(79B)	108.0
C(66)-C(65)-C(70)	118.8(5)	C(80)-Cl(7)-Cl(7)#1	95.2(9)
C(66)-C(65)-P(4)	123.1(4)	Cl(8)-C(80)-Cl(7)	111.3(3)
C(70)-C(65)-P(4)	118.1(4)	Cl(8)-C(80)-H(80A)	109.4
C(65)-C(66)-C(67)	120.7(5)	Cl(7)-C(80)-H(80A)	109.4
C(65)-C(66)-H(66)	119.6	Cl(8)-C(80)-H(80B)	109.4
C(67)-C(66)-H(66)	119.6	Cl(7)-C(80)-H(80B)	109.4
C(68)-C(67)-C(66)	120.2(5)	H(80A)-C(80)-H(80B)	108.0
C(68)-C(67)-H(67)	119.9	Cl(10)-C(81)-Cl(9)	111.3(3)
C(66)-C(67)-H(67)	119.9	Cl(10)-C(81)-H(81A)	109.4
C(69)-C(68)-C(67)	120.0(5)	Cl(9)-C(81)-H(81A)	109.4
C(69)-C(68)-H(68)	120.0	Cl(10)-C(81)-H(81B)	109.4
C(67)-C(68)-H(68)	120.0	Cl(9)-C(81)-H(81B)	109.4
C(68)-C(69)-C(70)	120.0(5)	H(81A)-C(81)-H(81B)	108.0
C(68)-C(69)-H(69)	120.0	Cl(12)-C(82)-Cl(11)	111.3(3)
C(70)-C(69)-H(69)	120.0	Cl(12)-C(82)-H(82A)	109.4
C(69)-C(70)-C(65)	120.3(5)	Cl(11)-C(82)-H(82A)	109.4
C(69)-C(70)-H(70)	119.9	Cl(12)-C(82)-H(82B)	109.4
C(65)-C(70)-H(70)	119.9	Cl(11)-C(82)-H(82B)	109.4
C(72)-C(71)-C(76)	117.7(4)	H(82A)-C(82)-H(82B)	108.0
C(72)-C(71)-P(4)	124.1(4)	Cl(14)-C(83)-Cl(13)	111.2(3)
C(76)-C(71)-P(4)	118.2(4)	Cl(14)-C(83)-H(83A)	109.4
C(71)-C(72)-C(73)	119.5(5)	Cl(13)-C(83)-H(83A)	109.4
C(71)-C(72)-H(72)	120.2	Cl(14)-C(83)-H(83B)	109.4

Cl(13)-C(83)-H(83B)	109.4
H(83A)-C(83)-H(83B)	108.0

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

Table 4. Anisotropic displacement parameters (Å^{2}x 10^4) for CCL04 (CCDC 168698). The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	139(2)	143(2)	133(2)	10(2)	41(2)	-6(2)
Pd(2)	145(2)	135(2)	141(2)	2(2)	51(2)	-6(2)
P(1)	148(7)	143(7)	134(7)	-8(5)	45(6)	-21(5)
P(2)	146(7)	140(7)	134(7)	10(5)	45(6)	-12(6)
P(3)	171(8)	143(7)	163(7)	2(6)	58(6)	-13(6)
P(4)	147(7)	149(7)	158(7)	14(6)	49(6)	-4(6)
B(1)	170(30)	160(30)	160(30)	20(20)	80(30)	-20(20)
B(2)	210(30)	130(30)	180(30)	50(30)	40(30)	20(30)
C(1)	190(30)	160(30)	130(30)	20(20)	50(20)	-20(20)
C(2)	150(30)	140(30)	160(30)	-20(20)	40(20)	-10(20)
C(3)	170(30)	150(30)	210(30)	30(20)	70(20)	10(20)
C(4)	150(30)	200(30)	160(30)	60(20)	50(20)	10(20)
C(5)	160(30)	160(30)	110(30)	30(20)	40(20)	0(20)
C(6)	200(30)	220(30)	180(30)	-30(20)	70(20)	0(20)
C(7)	140(30)	260(30)	300(30)	20(30)	40(30)	-20(20)
C(8)	160(30)	260(30)	280(30)	80(30)	100(30)	40(20)
C(9)	290(30)	190(30)	180(30)	10(20)	110(30)	50(20)
C(10)	210(30)	200(30)	150(30)	10(20)	20(20)	-10(20)
C(11)	100(30)	220(30)	150(30)	0(20)	40(20)	20(20)
C(12)	180(30)	240(30)	170(30)	10(20)	50(20)	0(20)
C(13)	260(30)	350(30)	200(30)	-120(30)	110(30)	-30(30)
C(14)	260(30)	290(30)	320(40)	-160(30)	110(30)	-10(30)
C(15)	200(30)	180(30)	390(40)	-40(30)	110(30)	-40(20)
C(16)	180(30)	180(30)	220(30)	-30(20)	60(20)	-20(20)
C(17)	120(30)	170(30)	190(30)	30(20)	60(20)	20(20)
C(18)	190(30)	200(30)	190(30)	0(20)	40(20)	20(20)
C(19)	300(30)	170(30)	260(30)	120(20)	120(30)	10(20)
C(20)	310(30)	160(30)	280(30)	-40(20)	100(30)	-30(20)
C(21)	300(40)	220(30)	260(30)	-30(20)	130(30)	30(20)
C(22)	200(30)	200(30)	230(30)	20(20)	110(20)	10(20)
C(23)	200(30)	100(20)	180(30)	60(20)	70(20)	-20(20)
C(24)	220(30)	190(30)	180(30)	20(20)	60(20)	30(20)
C(25)	140(30)	240(30)	280(30)	80(20)	40(30)	-20(20)
C(26)	180(30)	250(30)	190(30)	60(20)	-30(20)	-50(20)
C(27)	250(30)	190(30)	160(30)	10(20)	50(30)	-20(20)
C(28)	170(30)	160(30)	180(30)	30(20)	50(20)	0(20)
C(29)	190(30)	100(20)	150(30)	30(20)	50(20)	-10(20)
C(30)	200(30)	170(30)	160(30)	10(20)	50(20)	-40(20)
C(31)	200(30)	220(30)	190(30)	30(20)	10(20)	-90(20)
C(32)	350(40)	270(30)	130(30)	-50(20)	90(30)	-150(30)
C(33)	340(40)	260(30)	210(30)	-60(20)	160(30)	-60(30)
C(34)	220(30)	160(30)	190(30)	20(20)	60(20)	-20(20)
C(35)	100(30)	170(30)	140(30)	10(20)	20(20)	10(20)
C(36)	180(30)	190(30)	140(30)	-20(20)	50(20)	10(20)
C(37)	170(30)	160(30)	230(30)	0(20)	50(20)	-30(20)
C(38)	200(30)	220(30)	250(30)	30(20)	130(30)	-20(20)
C(39)	240(30)	210(30)	140(30)	-10(20)	80(20)	20(20)

C(40)	150(30)	140(30)	210(30)	-40(20)	40(20)	0(20)
C(41)	210(30)	160(30)	210(30)	-40(20)	50(20)	20(20)
C(42)	270(30)	280(30)	210(30)	-10(30)	30(30)	-50(30)
C(43)	420(40)	280(30)	190(30)	-10(30)	10(30)	10(30)
C(44)	330(40)	240(30)	250(30)	-110(30)	-70(30)	50(30)
C(45)	210(30)	260(30)	470(40)	-120(30)	10(30)	-50(30)
C(46)	220(30)	290(30)	290(30)	-40(30)	40(30)	-30(30)
C(47)	160(30)	180(30)	190(30)	40(20)	70(20)	-30(20)
C(48)	240(30)	210(30)	210(30)	-20(20)	60(30)	-60(20)
C(49)	290(40)	360(40)	210(30)	-40(30)	110(30)	-60(30)
C(50)	250(40)	470(40)	200(30)	60(30)	80(30)	-80(30)
C(51)	260(40)	290(30)	340(40)	90(30)	90(30)	-40(30)
C(52)	260(30)	220(30)	270(30)	0(20)	90(30)	-30(20)
C(53)	220(30)	170(30)	240(30)	110(20)	40(30)	30(20)
C(54)	240(30)	210(30)	310(30)	80(30)	70(30)	60(30)
C(55)	240(30)	250(30)	470(40)	180(30)	80(30)	80(30)
C(56)	200(40)	340(40)	530(40)	220(30)	180(30)	150(30)
C(57)	410(40)	300(30)	370(40)	110(30)	200(30)	160(30)
C(58)	250(30)	230(30)	270(30)	100(30)	60(30)	50(20)
C(59)	170(30)	230(30)	210(30)	30(20)	0(20)	-30(20)
C(60)	440(40)	320(30)	280(40)	20(30)	120(30)	-140(30)
C(61)	710(50)	400(40)	240(40)	70(30)	120(40)	-180(40)
C(62)	560(50)	400(40)	280(40)	180(30)	10(30)	-190(30)
C(63)	560(50)	220(30)	490(50)	100(30)	150(40)	-20(30)
C(64)	450(40)	290(30)	340(40)	100(30)	140(30)	50(30)
C(65)	180(30)	170(30)	190(30)	50(20)	90(20)	20(20)
C(66)	180(30)	150(30)	280(30)	60(20)	90(30)	10(20)
C(67)	230(30)	230(30)	390(40)	80(30)	170(30)	70(30)
C(68)	380(40)	190(30)	390(40)	50(30)	280(30)	80(30)
C(69)	480(40)	130(30)	210(30)	0(20)	170(30)	10(30)
C(70)	230(30)	140(30)	250(30)	50(20)	120(30)	30(20)
C(71)	140(30)	150(30)	150(30)	10(20)	20(20)	10(20)
C(72)	140(30)	190(30)	160(30)	10(20)	20(20)	-30(20)
C(73)	210(30)	240(30)	250(30)	40(20)	50(30)	-60(20)
C(74)	240(30)	200(30)	180(30)	-10(20)	10(30)	-50(20)
C(75)	240(30)	270(30)	130(30)	-20(20)	60(20)	40(20)
C(76)	220(30)	190(30)	170(30)	30(20)	70(20)	-20(20)
Cl(1)	477(11)	670(12)	455(11)	204(9)	289(9)	101(9)
Cl(2)	450(11)	667(12)	442(11)	161(9)	143(9)	167(9)
C(77)	340(40)	450(40)	350(40)	120(30)	200(30)	50(30)

Table 5. Hydrogen coordinates (x 10[4]) and isotropic displacement parameters ($Å^2$x 10 [3]) for CCL04 (CCDC 168698).

	x	y	z	U_{iso}
H(1A)	2573	2983	5541	19
H(1B)	2350	2247	5433	19
H(2A)	3236	2611	3791	18
H(2B)	3890	2772	4468	18
H(3A)	2184	6280	3834	21
H(3B)	2437	6865	3391	21
H(4A)	2052	5503	1797	21
H(4B)	1319	5466	1670	21
H(6)	730	2160	3696	24
H(7)	-234	2415	3896	29
H(8)	-332	3310	4721	27
H(9)	543	3944	5346	25
H(10)	1495	3716	5107	23
H(12)	2105	2573	2510	23
H(13)	2050	1691	1622	31
H(14)	1774	682	2018	34
H(15)	1602	538	3329	30
H(16)	1679	1409	4246	23
H(18)	3000	1352	5986	23
H(19)	2914	253	5769	28
H(20)	3047	-205	4530	29
H(21)	3313	469	3545	30
H(22)	3387	1579	3753	24
H(24)	4485	2213	5595	24
H(25)	5281	2360	6829	27
H(26)	5053	2767	8051	27
H(27)	4018	2927	8013	25
H(28)	3222	2761	6769	20
H(30)	4407	3792	5588	21
H(31)	4714	4156	7000	25
H(32)	3982	4579	7605	30
H(33)	2932	4621	6830	30
H(34)	2609	4247	5434	23
H(36)	3964	4861	4535	20
H(37)	4637	5315	3825	23
H(38)	4774	4820	2612	25
H(39)	4196	3891	2053	23
H(40)	3548	3423	2758	20
H(42)	3149	5764	5131	31
H(43)	3866	6015	6435	38
H(44)	4750	6618	6473	36
H(45)	4918	6959	5205	40
H(46)	4206	6704	3899	33
H(48)	3431	5396	2045	26
H(49)	3772	5793	916	34
H(50)	3822	6910	716	36
H(51)	3556	7620	1662	36
H(52)	3224	7218	2790	30

H(54)	510	5962	1620	31
H(55)	-426	6108	1930	39
H(56)	-468	6706	3110	41
H(57)	431	7200	3940	41
H(58)	1384	7009	3689	30
H(60)	1939	6210	815	41
H(61)	2040	6996	-149	55
H(62)	1940	8088	153	53
H(63)	1734	8382	1420	51
H(64)	1663	7591	2367	43
H(66)	551	5000	2888	24
H(67)	49	5229	3917	32
H(68)	629	5448	5316	34
H(69)	1709	5402	5700	31
H(70)	2218	5183	4669	24
H(72)	1032	3856	3170	20
H(73)	555	2965	2329	28
H(74)	741	2753	1043	26
H(75)	1423	3412	568	26
H(76)	1905	4281	1366	23
H(77A)	4400	1052	5491	43
H(77B)	4525	349	5167	43
H(78A)	2152	8502	2536	115
H(78B)	2737	8075	2484	115
H(79A)	2951	8363	2457	115
H(79B)	3446	8905	2910	115
H(80A)	437	9496	3571	53
H(80B)	230	10235	3602	53
H(81A)	564	517	4730	53
H(81B)	640	-77	5379	53
H(82A)	991	8481	3695	158
H(82B)	1518	9010	3715	158
H(83A)	153	9223	2465	158
H(83B)	312	8694	1845	158