Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Chemistry Department, X-ray Lab
(Chernoff Hall, Room 107)

X-Ray Crystallographic Analysis Report for

Pd(dimethylbenzyl amine)(pyridine)(chloride) or Pd(dmba)(py)(Cl)

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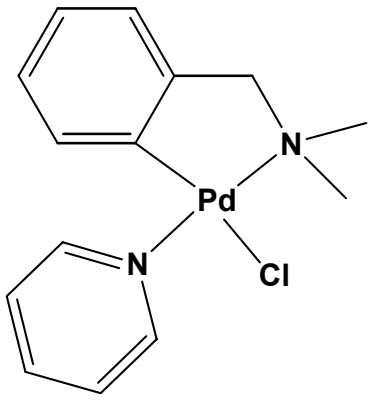
Sample ID: Pd(dmba)(py)(Cl)

User: Zhong-Lin Lu **Supervisor:** Dr. RS Brown

Date: September 28, 2004



A crystal of the compound (colorless, plate-shaped, size 0.5 x 0.2 x 0.1mm) was mounted on a glass fiber with grease very quickly. Data collection was performed on a Bruker SMART CCD 1000 X-ray diffractometer with graphite-monochromated Mo K_α radiation (λ = 0.71073 Å), operating at 50 kV and 30 mA over 2θ ranges of 4.50 ~ 56.56º at -93 °C controlled with Crysostream Controller 700. No significant decay was observed during the data collection.

Data were processed on a Pentium PC using the Bruker AXS Windows NT SHELXTL software package (version 5.10).[1] Neutral atom scattering factors were taken from Cromer and Waber.[2] The raw intensity data were converted (including corrections for scan speed, background, and Lorentz and polarization effects) to structure amplitudes and their esd's using the program SAINT, which corrects for *L*p and decay. Absorption corrections were applied using program SADABS. The crystal is non-merohedrally twinned. GEMINI program (Version 1.0) was applied and only the reflections from the domain component were used to solve the structure. The crystal is orthorhombic space group *P*bca, based on the systematic absences, *E* statistics and successful refinement of the structure. The structure was solved by direct methods. Full-matrix least-square refinements minimizing the function $\sum w (F_o^2 - F_c^2)^2$ were applied to the compound. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were located gradually from difference Fourier map, and their contributions were included in the structure factor calculations.

Convergence to final R_1 = 0.0329 and wR_2 = 0.0595 by using 3377 independent reflections and 231 parameters were achieved,[3] with the largest residual peak and hole to be 0.984 and – 0.630 e/Å^3, respectively. Crystallographic data, atomic coordinates and equivalent isotropic displacement parameters, bond lengths and angles, anisotropic displacement parameters, hydrogen coordinates and isotropic displacement parameters, and torsion angles are given in Table 1 to 6. The molecular structure and the cell packing are shown in Figures 1 and 2.

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[1] Bruker AXS Crystal Structure Analysis Package, Version 5.10 (SMART NT (Version 5.053), SAINT-Plus (Version 6.01), GEMINI (Version 1.02), SHELXTL (Version 5.1)); Bruker AXS Inc.: Madison, WI, 1999.

[2] Cromer, D. T.; Waber, J. T. *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, UK, 1974; Vol. 4, Table 2.2 A.

[3] $R_1 = \sum | |Fo| - |Fc| | / \sum |Fo|$

$wR_2 = \{\sum [w (Fo^2 - Fc^2)^2] / \sum [w(Fo^2)^2]\}^{1/2}$

($w = 1 / [\sigma^2(Fo^2) + (0.030P)^2]$, where $P = [\text{Max} (Fo^2, 0) + 2Fc^2] / 3$)

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Table 1. Crystal data and structure refinement for Pd(dmba)(py)(Cl)

Identification code	Pd(dmba)(py)(Cl)	
Empirical formula	C14 H17 Cl N2 Pd	
Formula weight	355.15	
Temperature	180(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 9.5463(18) Å	α= 90°.
	b = 16.258(4) Å	β= 90°.
	c = 18.117(4) Å	γ = 90°.
Volume	2811.7(11) $Å^3$	
Z	8	
Density (calculated)	1.678 Mg/m^3	
Absorption coefficient	1.493 mm^{-1}	
F(000)	1424	
Crystal size	0.5 x 0.2 x 0.1 mm^3	
Theta range for data collection	2.25 to 28.28°.	
Index ranges	-12<=h<=12, -19<=k<=21, -23<=l<=24	
Reflections collected	18463	
Independent reflections	3377 [R(int) = 0.0478]	
Completeness to theta = 28.28°	96.7 %	
Absorption correction	Empirical (Bruker SADABS)	
Max. and min. transmission	1.0000 and 0.7774	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3377 / 0 / 231	
Goodness-of-fit on F^2	0.928	
Final R indices [I>2sigma(I)]	R1 = 0.0329, wR2 = 0.0595	
R indices (all data)	R1 = 0.0577, wR2 = 0.0641	
Largest diff. peak and hole	0.984 and -0.630 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Pd(dmba)(py)(Cl). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	9018(1)	524(1)	2651(1)	22(1)
Cl(1)	9511(1)	1470(1)	3657(1)	33(1)
N(1)	9308(2)	1403(2)	1830(1)	23(1)
N(2)	8744(2)	-400(2)	3402(1)	24(1)
C(1)	8524(3)	223(2)	1116(2)	25(1)
C(2)	8359(3)	-191(2)	458(2)	31(1)
C(3)	8383(3)	-1046(2)	433(2)	35(1)
C(4)	8598(3)	-1478(2)	1083(2)	32(1)
C(5)	8764(3)	-1059(2)	1750(2)	29(1)
C(6)	8723(3)	-202(2)	1782(2)	24(1)
C(7)	8476(3)	1133(2)	1172(2)	28(1)
C(8)	8927(4)	2259(2)	2025(2)	35(1)
C(9)	10828(3)	1386(2)	1644(2)	32(1)
C(10)	7574(3)	-876(2)	3417(2)	28(1)
C(11)	7390(4)	-1483(2)	3935(2)	34(1)
C(12)	8411(4)	-1610(2)	4460(2)	35(1)
C(13)	9592(4)	-1131(2)	4451(2)	33(1)
C(14)	9726(3)	-539(2)	3918(2)	30(1)

Table 3. Bond lengths [Å] and angles [°] for Pd(dmba)(py)(Cl).

Pd(1)-C(6)	1.987(3)
Pd(1)-N(2)	2.043(2)
Pd(1)-N(1)	2.082(2)
Pd(1)-Cl(1)	2.4312(9)
N(1)-C(8)	1.480(4)
N(1)-C(9)	1.490(4)
N(1)-C(7)	1.497(4)
N(2)-C(14)	1.344(4)
N(2)-C(10)	1.359(4)
C(1)-C(2)	1.378(5)
C(1)-C(6)	1.402(4)
C(1)-C(7)	1.484(5)
C(2)-C(3)	1.391(5)
C(3)-C(4)	1.388(5)
C(4)-C(5)	1.395(5)
C(5)-C(6)	1.395(5)
C(10)-C(11)	1.373(4)
C(11)-C(12)	1.377(5)
C(12)-C(13)	1.370(5)
C(13)-C(14)	1.368(4)
C(6)-Pd(1)-N(2)	94.20(12)
C(6)-Pd(1)-N(1)	81.95(12)
N(2)-Pd(1)-N(1)	176.05(10)
C(6)-Pd(1)-Cl(1)	175.51(9)
N(2)-Pd(1)-Cl(1)	89.51(7)
N(1)-Pd(1)-Cl(1)	94.30(7)
C(8)-N(1)-C(9)	108.1(3)
C(8)-N(1)-C(7)	109.6(3)
C(9)-N(1)-C(7)	109.4(3)
C(8)-N(1)-Pd(1)	116.2(2)
C(9)-N(1)-Pd(1)	106.10(19)
C(7)-N(1)-Pd(1)	107.24(18)
C(14)-N(2)-C(10)	117.6(3)

C(14)-N(2)-Pd(1)	119.9(2)
C(10)-N(2)-Pd(1)	122.5(2)
C(2)-C(1)-C(6)	121.3(3)
C(2)-C(1)-C(7)	122.8(3)
C(6)-C(1)-C(7)	115.9(3)
C(1)-C(2)-C(3)	120.9(3)
C(4)-C(3)-C(2)	118.8(3)
C(3)-C(4)-C(5)	120.2(4)
C(6)-C(5)-C(4)	121.4(3)
C(5)-C(6)-C(1)	117.4(3)
C(5)-C(6)-Pd(1)	128.4(2)
C(1)-C(6)-Pd(1)	114.1(2)
C(1)-C(7)-N(1)	109.3(3)
N(2)-C(10)-C(11)	121.9(3)
C(10)-C(11)-C(12)	119.3(3)
C(13)-C(12)-C(11)	119.2(3)
C(14)-C(13)-C(12)	119.0(3)
N(2)-C(14)-C(13)	123.0(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Pd(dmba)(py)(Cl). 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)	22(1)	21(1)	23(1)	3(1)	-2(1)	-1(1)
Cl(1)	40(1)	28(1)	31(1)	-3(1)	-7(1)	-1(1)
N(1)	22(1)	22(2)	25(2)	2(1)	-1(1)	-1(1)
N(2)	29(1)	21(2)	22(1)	-1(1)	-1(1)	-1(1)
C(1)	21(1)	28(2)	27(2)	5(1)	1(1)	-3(1)
C(2)	34(2)	36(2)	24(2)	6(2)	-1(2)	-2(2)
C(3)	38(2)	44(3)	24(2)	-6(2)	4(2)	-5(2)
C(4)	36(2)	20(2)	38(2)	-5(2)	8(1)	-1(2)
C(5)	28(2)	30(2)	29(2)	6(2)	2(1)	-2(1)
C(6)	19(2)	29(2)	25(2)	0(1)	0(1)	0(1)
C(7)	28(2)	28(2)	27(2)	6(2)	-3(1)	-1(1)
C(8)	45(2)	24(2)	37(2)	4(2)	2(2)	0(2)
C(9)	27(2)	34(2)	35(2)	3(2)	1(2)	-5(2)
C(10)	29(2)	30(2)	24(2)	-1(2)	0(1)	-2(1)
C(11)	36(2)	28(2)	37(2)	0(2)	11(2)	-4(2)
C(12)	54(2)	23(2)	27(2)	7(2)	15(2)	8(2)
C(13)	40(2)	34(2)	24(2)	5(2)	-5(2)	10(2)
C(14)	30(2)	30(2)	29(2)	1(2)	-3(1)	2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Pd(dmba)(py)(Cl).

	x	y	z	U(eq)
H(2)	8220(20)	51(17)	31(15)	10(7)
H(3)	8180(30)	-1330(20)	-9(17)	35(9)
H(4)	8620(30)	-2000(20)	1053(17)	31(10)
H(5)	8920(30)	-1380(20)	2165(17)	29(9)
H(7A)	7500(30)	1312(16)	1241(13)	14(7)
H(7B)	8860(30)	1420(20)	744(17)	33(9)
H(8A)	7970(30)	2260(20)	2169(16)	35(9)
H(8B)	9070(30)	2660(20)	1560(20)	47(10)
H(8C)	9460(40)	2430(30)	2390(20)	51(12)
H(9A)	11390(30)	1500(20)	2047(18)	32(9)
H(9B)	11080(30)	880(20)	1467(17)	29(9)
H(9C)	11050(30)	1850(20)	1268(18)	33(9)
H(10)	6890(30)	-741(16)	3032(14)	14(7)
H(11)	6600(30)	-1740(20)	3911(16)	29(9)
H(12)	8320(30)	-1960(20)	4783(18)	33(10)
H(13)	10220(30)	-1198(18)	4774(16)	23(8)
H(14)	10530(30)	-175(18)	3898(16)	25(8)

Table 6. Torsion angles [°] for Pd(dmba)(py)(Cl).

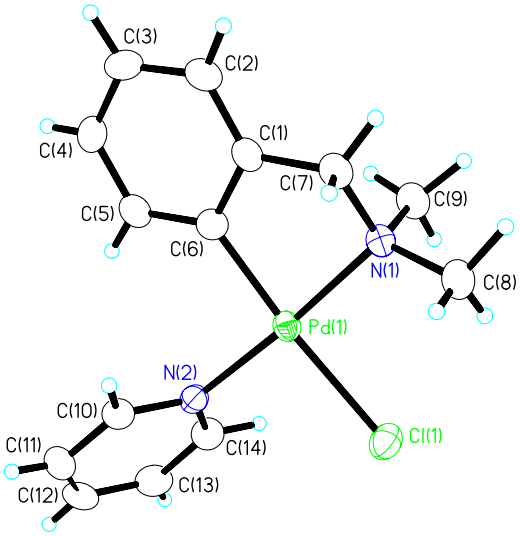
C(6)-Pd(1)-N(1)-C(8)	150.6(2)
N(2)-Pd(1)-N(1)-C(8)	163.7(12)
Cl(1)-Pd(1)-N(1)-C(8)	-31.9(2)
C(6)-Pd(1)-N(1)-C(9)	-89.2(2)
N(2)-Pd(1)-N(1)-C(9)	-76.1(13)
Cl(1)-Pd(1)-N(1)-C(9)	88.3(2)
C(6)-Pd(1)-N(1)-C(7)	27.60(19)
N(2)-Pd(1)-N(1)-C(7)	40.7(13)
Cl(1)-Pd(1)-N(1)-C(7)	-154.89(18)
C(6)-Pd(1)-N(2)-C(14)	126.5(2)
N(1)-Pd(1)-N(2)-C(14)	113.5(12)
Cl(1)-Pd(1)-N(2)-C(14)	-51.0(2)
C(6)-Pd(1)-N(2)-C(10)	-55.4(3)
N(1)-Pd(1)-N(2)-C(10)	-68.5(13)
Cl(1)-Pd(1)-N(2)-C(10)	127.1(2)
C(6)-C(1)-C(2)-C(3)	0.1(5)
C(7)-C(1)-C(2)-C(3)	-178.6(3)
C(1)-C(2)-C(3)-C(4)	-1.0(5)
C(2)-C(3)-C(4)-C(5)	1.0(5)
C(3)-C(4)-C(5)-C(6)	-0.1(5)
C(4)-C(5)-C(6)-C(1)	-0.8(4)
C(4)-C(5)-C(6)-Pd(1)	-176.9(2)
C(2)-C(1)-C(6)-C(5)	0.8(4)
C(7)-C(1)-C(6)-C(5)	179.6(2)
C(2)-C(1)-C(6)-Pd(1)	177.5(2)
C(7)-C(1)-C(6)-Pd(1)	-3.7(3)
N(2)-Pd(1)-C(6)-C(5)	-16.7(3)
N(1)-Pd(1)-C(6)-C(5)	162.4(3)
Cl(1)-Pd(1)-C(6)-C(5)	128.9(10)
N(2)-Pd(1)-C(6)-C(1)	167.0(2)
N(1)-Pd(1)-C(6)-C(1)	-13.9(2)
Cl(1)-Pd(1)-C(6)-C(1)	-47.4(12)
C(2)-C(1)-C(7)-N(1)	-154.0(3)
C(6)-C(1)-C(7)-N(1)	27.2(4)

C(8)-N(1)-C(7)-C(1)	-162.9(3)
C(9)-N(1)-C(7)-C(1)	78.7(3)
Pd(1)-N(1)-C(7)-C(1)	-35.9(3)
C(14)-N(2)-C(10)-C(11)	-0.4(5)
Pd(1)-N(2)-C(10)-C(11)	-178.5(2)
N(2)-C(10)-C(11)-C(12)	0.6(5)
C(10)-C(11)-C(12)-C(13)	-0.3(5)
C(11)-C(12)-C(13)-C(14)	-0.3(5)
C(10)-N(2)-C(14)-C(13)	-0.2(5)
Pd(1)-N(2)-C(14)-C(13)	178.0(3)
C(12)-C(13)-C(14)-N(2)	0.5(5)

Symmetry transformations used to generate equivalent atoms:

Figure 1. Molecular Structure of Pd(dmba)(py)(Cl) (Displacement ellipsoids for non-H atoms are shown at the 50% probability level and H atoms are represented by circles of arbitrary size.)

a)



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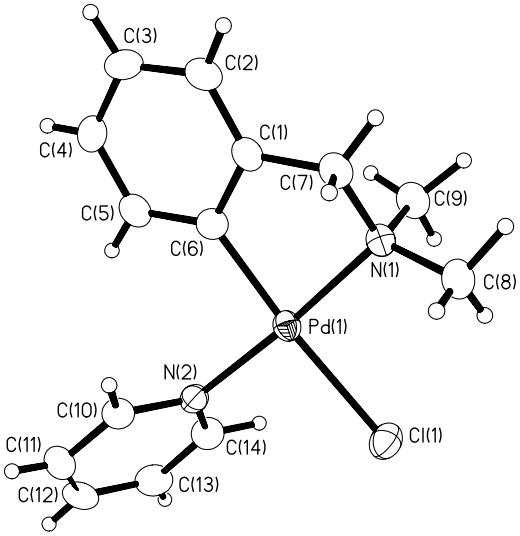

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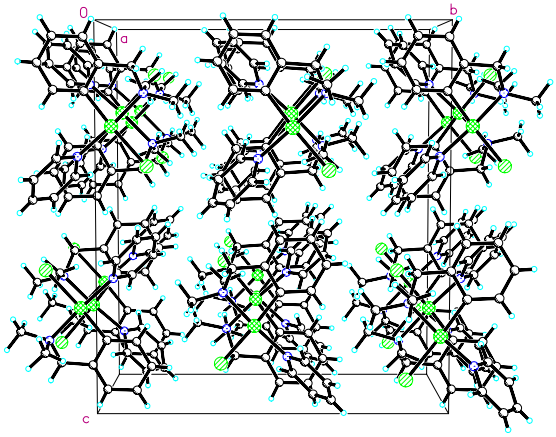
Figure 2. Unit cell packing of Pd(dmba)(py)(Cl)

a)



b)