Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dibromo[2,2′,3,3′-tetrahydro-3,3′-bis(4-methoxybenzyl)-1,1′-methylenedi-1*H*-imidazole-$\kappa^2C^2,C^{2'}$]-palladium(II)

Hon Man Lee* and Pei Ling Chiu

National Changhua University of Education, Department of Chemistry, Changhua, Taiwan 50058

Correspondence e-mail: leehm@cc.ncue.edu.tw

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.014 Å
R factor = 0.052
wR factor = 0.113
Data-to-parameter ratio = 20.7

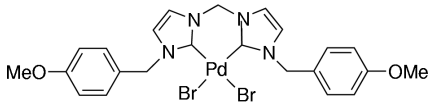
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $[PdBr_2(C_{23}H_{24}N_4O_2)]$, was determined at 273 K. It crystallizes in the non-centrosymmetric trigonal space group $P3_2$. Non-classical hydrogen bonds of types C—H···Br and C—H···O exist in the structure.

Received 26 July 2004
Accepted 28 July 2004
Online 21 August 2004

Comment

N-Heterocyclic carbene (NHC) ligands have been shown to have wide applicability in coordination chemistry and catalysis. The use of chelating bis(NHC) ligands is also receiving much attention because they can provide extra air and moisture stability for the metal centers. For example, palladium complexes of bis(NHC) carbenes have been found to be efficient catalysts in C—C coupling reactions (Herrmann *et al.*, 1998; Zhang & Trudell, 2000; Lee *et al.*, 2004).



(I)

The crystal structures of palladium complexes with bis(NHC) ligands have been reported by Herrmann *et al.* (1999), Schwarz *et al.* (2000), Douthwaite *et al.* (2002), Herdtweck *et al.* (2003), Bonnet *et al.* (2003) and Lee *et al.* (2004). We present here the structure of dibromo[2,2′,3,3′-tetrahydro-3,3′-bis(4-methoxylbenzyl)-1,1′-methylenedi-1*H*-imidazole]palladium(II), (I). The 3-methoxylbenzyl isomer, (II), was reported recently by us (Lee *et al.*, 2004).

The title compound, (I), crystallizes in the non-centrosymmetric trigonal space group $P3_2$. The refined Flack (1983)

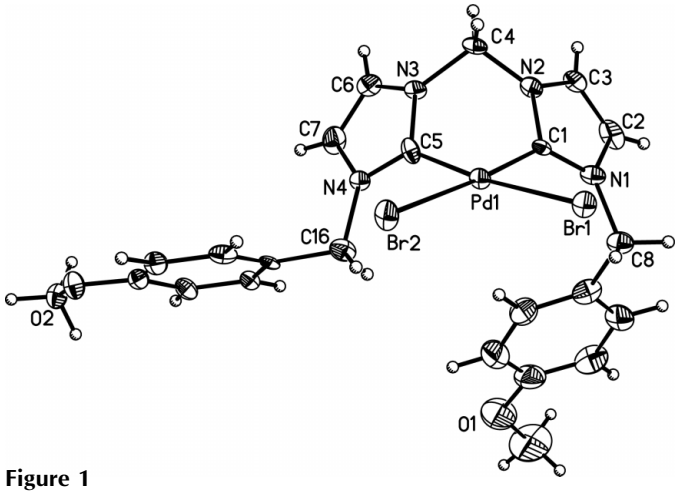


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids for non-H atoms.

parameter is 0.029 (11). The palladium center is in a square-planar geometry (Fig. 1). The dihedral angle between the two methylene-linked imidazole rings is 75.8 (3)°. The molecular dimensions in (I) are similar to those in (II).

Some intermolecular non-classical hydrogen bonds of types C—H···Br and C—H···O are present in the crystal structure (Table 1). Fig. 2 illustrates the honeycomb-like structure, viewed along the *c* axis.

Experimental

The title compound was prepared according to the literature procedure of Lee *et al.* (2004). Suitable crystals were obtained by slow diffusion of diethyl ether into a dimethylformamide solution of the palladium complex at room temperature.

Crystal data

$[PdBr_2(C_{23}H_{24}N_4O_2)]$
M_r = 654.68
Trigonal, $P3_2$
a = 15.41 (3) Å
c = 8.617 (12) Å
V = 1772 (5) Å^3
Z = 3
D_x = 1.840 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3022 reflections
θ = 2.6–28.8°
μ = 4.20 mm^{-1}
T = 273 (2) K
Prism, colorless
0.23 × 0.20 × 0.19 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.401, T_{max} = 0.453
13 263 measured reflections
5870 independent reflections
4809 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 28.4°
$h = -20 \rightarrow 20$
$k = -14 \rightarrow 20$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.113
S = 1.02
5870 reflections
283 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.49 e Å^{-3}
Absolute structure: Flack (1983) 2914 Friedel pairs
Flack parameter = 0.029 (11)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···Br1^{i}	0.93	2.78	3.645 (9)	155
C4—H4B···Br2^{ii}	0.97	2.78	3.751 (9)	177
C23—H23B···Br1^{iii}	0.96	2.81	3.491 (12)	128
C23—H23C···O2^{iv}	0.96	2.45	3.175 (13)	133

Symmetry codes: (i) $x, y, 1+z$; (ii) $-x+y, 1-x, \frac{1}{3}+z$; (iii) $-x+y, -x, \frac{1}{3}+z$; (iv) $1-x+y, 1-x, \frac{1}{3}+z$.

All H atoms were positioned geometrically [C—H = 0.97 (CH_2) and 0.93 Å (other H atoms)] and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for all other H atoms. The highest peak (1.19 e Å^{-3}) is 0.96 Å from Pd1 and the deepest hole (−1.49 e Å^{-3}) is 0.91 Å from Pd1.

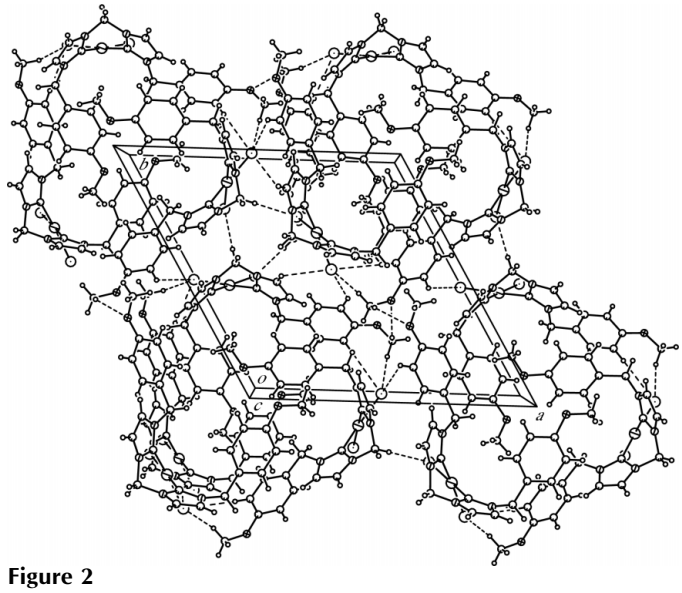


Figure 2
A view of the packing of (I) along the *c* axis; weak interactions are indicated by dashed lines.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We are grateful to the National Science Council of Taiwan for financial support (grant NSC 92-2113-M-018-005).

References

Bruker (2001). *SMART* (Version 5.054) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Bonnet, L. G., Douthwaite, R. E. & Hodgson, R. (2003). *Organometallics*, **22**, 4384–4386.
Douthwaite, R. E., Green, M. L. H., Silcock, P. J. & Gomes, P. T. (2002). *J. Chem. Soc. Dalton Trans.* pp. 1386–1390.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Herdtweck, E., Muehlhofer, M. & Strassner, T. (2003). *Acta Cryst.* E**59**, m970–m971.
Herrmann, W. A., Reisinger, C.-P. & Spiegler, M. (1998). *J. Organomet. Chem.* **557**, 93–96.
Herrmann, W. A., Schwarz, J. & Gardiner, M. G. (1999). *Organometallics*, **18**, 4082–4089.
Lee, H. M., Lu, C. Y., Chen, C. Y., Chen, W. L., Lin, H. C., Chiu, P. L. & Cheng, P. Y. (2004). *Tetrahedron*, **60**, 5807–5825.
Schwarz, J., Bohm, V. P. W., Gardiner, M. G., Grosche, M., Herrmann, W. A., Hieringer, W. & Raudaschl-Sieber, G. (2000). *Chem. Eur. J.* **6**, 1773–1780.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2002). *SADABS*. Verion 2.04. University of Göttingen, Germany.
Zhang, C. & Trudell, M. L. (2000). *Tetrahedron Lett.* **41**, 595–598.