Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(*R*)-Bis[1,3-bis(1-phenylethyl)imidazolin-2-ylidene]-dichloropalladium(II) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.006 Å
R factor = 0.034
wR factor = 0.083
Data-to-parameter ratio = 17.3

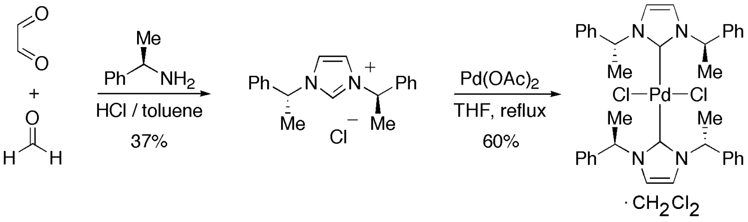
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[PdCl_2(C_{19}H_{20}N_2)_2]\cdot CH_2Cl_2$, contains a Pd^{II} atom in a square-planar geometry and is isolated as the *trans* isomer.

Received 22 April 2004
Accepted 29 April 2004
Online 8 May 2004

Comment

Recently, *N*-heterocyclic carbenes (NHC) have emerged as powerful ligands for numerous transition metals and main group elements (Herrmann, 2002). The strong coordination ability of NHCs stabilizes and activates the metal center to perform a variety of synthetic transformations, including metathesis and cross-coupling reactions (Yong & Nolan, 2003). Among NHC complexes, a number of chiral NHC transition metal complexes have been reported for asymmetric catalysis (Perry & Burgess, 2003). Our interest in ligands suitable for inducing enantioselectivity in transition-metal-catalysed processes prompted us to prepare a series of chiral palladium complexes, including the title compound, (I) (Grasa *et al.*, 2002; Charette *et al.*, 2003). Although similar complexes have previously been prepared (Enders *et al.*, 1996; Herrmann *et al.*, 2001), no X-ray crystal structure has been reported for the dichloropalladium(II) complex.



(I)

The title compound, (I), features a four-coordinate Pd atom in a slightly distorted square-planar geometry. The Pd—C distances are not significantly different and are consistent with Pd—C single bonds. Both chiral heterocyclic carbene ligands coordinate the palladium through their C2 atom, an important feature as recent results show that this procedure may lead to unusual C5 imidazolium coordination (Lebel *et al.*, 2004). The orientation of the four phenylethyl groups is also of interest. In all cases, both the methyl and phenyl groups are directed away from the metal center, probably to avoid steric interaction.

Experimental

The title palladium complex was synthesized according to the literature procedure of Herrmann *et al.* (1995). Under an argon atmosphere, a flask was charged with *N*,*N*′-bis(1-phenylethyl)-imidazolium chloride (566 mg, 1.87 mmol) and palladium acetate (200 mg, 0.89 mmol). The flask was then fitted with a condenser and

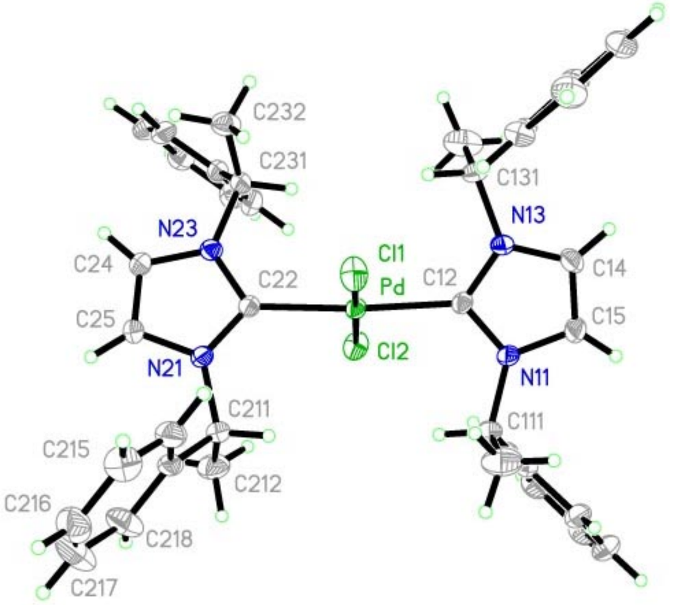


Figure 1
The structure of the title molecule. Displacement ellipsoids are drawn at the 30% probability level. The solvent molecule is not shown.

tetrahydrofuran (20 ml) was added *via* a syringe. The resulting brown mixture was refluxed for 12 h. Volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (30% ethyl acetate in hexanes) to afford the title compound as a yellow solid (390 mg, 60%). Suitable crystals were obtained by slow diffusion of pentane into a saturated solution of the complex in dichloromethane. Spectroscopic analysis, ^{1}H NMR ($CDCl_3$, δ): 7.60 (*d*, 8H, J = 7 Hz), 7.39–7.29 (*m*, 12H), 6.89 (*q*, 4H, J = 7 Hz), 6.56 (*s*, 4H), 1.93 (*d*, 12H, J = 7 Hz); ^{13}C NMR ($CDCl_3$, δ): 171.2, 140.8, 129.1, 128.3, 128.1, 118.4, 58.8, 21.0; analysis calculated for $C_{38}H_{40}Cl_2N_4Pd$: C 62.51, H 5.52, N 7.67%; found: C 62.29, H 5.61, N 7.70%.

Crystal data

$[PdCl_2(C_{19}H_{20}N_2)_2]\cdot CH_2Cl_2$
$M_r = 814.97$
Orthorhombic, $P2_12_12_1$
$a = 11.6567\ (1)$ Å
$b = 13.6627\ (1)$ Å
$c = 24.1997\ (2)$ Å
$V = 3854.09\ (5)$ Å^3
$Z = 4$
$D_x = 1.405$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 6850 reflections
$\theta = 3.7$–$72.9°$
$\mu = 6.68$ mm^{-1}
$T = 220\ (2)$ K
Plate, colorless
0.41 × 0.30 × 0.05 mm

Data collection

Bruker SMART 2K/Platform diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996*a*)
$T_{min} = 0.135$, $T_{max} = 0.715$
46 333 measured reflections
7556 independent reflections
6917 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 72.9°$
$h = -13 \rightarrow 11$
$k = -16 \rightarrow 16$
$l = -29 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.083$
$S = 1.04$
7556 reflections
437 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Absolute structure: Flack (1983), 3255 Friedel Pairs
Flack parameter = −0.023 (7)

Table 1
Selected geometric parameters (Å, °).

Pd—C12	2.038 (3)	N13—C131	1.486 (4)
Pd—C22	2.039 (3)	C14—C15	1.334 (6)
Pd—Cl1	2.3050 (9)	N21—C22	1.331 (4)
Pd—Cl2	2.3119 (9)	N21—C25	1.396 (4)
N11—C12	1.354 (4)	N21—C211	1.472 (4)
N11—C15	1.385 (5)	C22—N23	1.342 (4)
N11—C111	1.493 (4)	N23—C24	1.379 (4)
C12—N13	1.344 (4)	N23—C231	1.486 (4)
N13—C14	1.380 (5)	C24—C25	1.333 (5)
C12—Pd—C22	175.64 (14)	C14—N13—C131	123.8 (3)
C12—Pd—Cl1	90.74 (9)	C15—C14—N13	106.7 (4)
C22—Pd—Cl1	88.91 (9)	C14—C15—N11	106.9 (4)
C12—Pd—Cl2	90.29 (9)	C22—N21—C25	110.8 (3)
C22—Pd—Cl2	90.32 (9)	C22—N21—C211	125.7 (3)
Cl1—Pd—Cl2	176.55 (4)	C25—N21—C211	123.2 (3)
C12—N11—C15	110.5 (3)	N21—C22—N23	105.1 (3)
C12—N11—C111	124.1 (3)	N21—C22—Pd	130.0 (3)
C15—N11—C111	124.8 (3)	N23—C22—Pd	124.8 (2)
N13—C12—N11	104.6 (3)	C22—N23—C24	111.0 (3)
N13—C12—Pd	127.3 (2)	C22—N23—C231	124.8 (3)
N11—C12—Pd	128.1 (2)	C24—N23—C231	124.1 (3)
C12—N13—C14	111.2 (3)	C25—C24—N23	106.7 (3)
C12—N13—C131	124.9 (3)	C24—C25—N21	106.4 (3)
C15—N11—C12—N13	−0.6 (4)	C25—N21—C22—N23	0.3 (4)
C111—N11—C12—N13	171.1 (3)	C211—N21—C22—N23	173.1 (3)
C15—N11—C12—Pd	176.6 (3)	C25—N21—C22—Pd	178.2 (3)
C111—N11—C12—Pd	−11.6 (5)	C211—N21—C22—Pd	−9.0 (5)
Cl1—Pd—C12—N13	−70.1 (3)	Cl1—Pd—C22—N21	−88.6 (3)
Cl2—Pd—C12—N13	113.2 (3)	Cl2—Pd—C22—N21	88.0 (3)
Cl1—Pd—C12—N11	113.2 (3)	Cl1—Pd—C22—N23	88.9 (3)
Cl2—Pd—C12—N11	−63.5 (3)	Cl2—Pd—C22—N23	−94.5 (3)
N11—C12—N13—C14	1.0 (4)	N21—C22—N23—C24	0.2 (4)
Pd—C12—N13—C14	−176.3 (3)	Pd—C22—N23—C24	−177.8 (2)
N11—C12—N13—C131	179.3 (3)	N21—C22—N23—C231	−178.1 (3)
Pd—C12—N13—C131	2.0 (5)	Pd—C22—N23—C231	3.8 (5)
C12—N13—C14—C15	−1.0 (5)	C22—N23—C24—C25	−0.7 (4)
C131—N13—C14—C15	−179.3 (4)	C231—N23—C24—C25	177.7 (3)
N13—C14—C15—N11	0.6 (5)	N23—C24—C25—N21	0.8 (4)
C12—N11—C15—C14	0.0 (5)	C22—N21—C25—C24	−0.7 (4)
C111—N11—C15—C14	−171.7 (4)	C211—N21—C25—C24	−173.8 (3)

H atoms were constrained to the parent site using a riding model (C—H = 0.94–0.98 Å); $U_{iso}(H) = 1.5U_{eq}(C)$ in methyl groups and $1.2U_{eq}(C)$ for the other H atoms. A final verification of possible voids was performed using the VOID routine of the *PLATON* program (Spek, 1995).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*b*); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL*96 (Sheldrick, 1996*b*).

This research was supported by the Natural Sciences and Engineering Research Council of Canada, the Fonds FCAR du Ministère de l'Éducation du Québec, the Foundation for Innovation and the Université de Montréal.

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