Chloro{1-[(1-hydroxymethyl-2-methylpropyl)-iminomethyl]ferrocenyl-$\kappa^2 C,N$}(triphenylphosphine-κP)palladium(II) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.011 Å
R factor = 0.062
wR factor = 0.146
Data-to-parameter ratio = 15.8

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

The asymmetric unit of the title compound, [FePd-$(C_5H_5)(C_{11}H_{15}NO)Cl(C_{18}H_{15}P)]\cdot CH_2Cl_2$, contains two molecular palladium complexes and two dichloromethane solvent molecules. The Pd atoms have approximately square planar configurations. Intermolecular O—H···Cl hydrogen bonds stabilize the crystal structure.

Comment

Cyclopalladated complexes have been widely studied in different areas as a result of their important applications as precursors for organic or organometallic synthesis (Vicente *et al.*, 1995), catalytic activity (Gruber *et al.*,2000; Bose & Saha, 1989; Santra & Saha, 1987), and the determination of enantiomeric excess of chiral reagents (Albert *et al.*, 2000). In addition, palladated compounds have proven to be efficient catalysts in Heck reactions (Ziegler & Heck, 1978). The *ortho*-cyclopalladated compounds have been studied extensively in the past decade (Zhao *et al.*, 1997; Lopez *et al.*, 2005) and we report here the crystal structure of the title compound (I) as its dichloromethane solvate

(I)
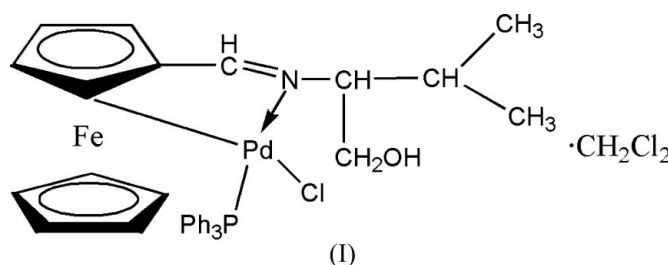
The two mononuclear palladium complexes in the asymmetric unit each adopt an approximately square planar configuration defined by the atoms C6, P1, N1 and Cl1 or C41, P2, N2 and Cl4 (Fig. 1 and Table 1). The imine groups adopt an *anti* conformation, as reflected by the C10—C11—N1—C12 [−179.3 (7)°] and C45—C46—N2—C47 [176.4 (6)°] torsion angles. The C=N bond lengths are similar to those reported for other monocyclopalladated complexes (Bosque *et al.*, 1994; Lopez *et al.*, 1994).

The coordinated triphenylphosphine is *trans* to the imino N atom. The two Cp rings are almost parallel, with a dihedral angle of 0.8 (5) and 1.6 (5)° and the two rings involved in the bicyclic system formed by fusion of the palladacycle with the substituted Cp ring are almost coplanar, the relevant dihedral angles being 4.2 (4) and 7.8 (4)°.

In the crystal structure of (I), molecules are linked by O—H···Cl hydrogen bonds which help to stablize the molecular structure.
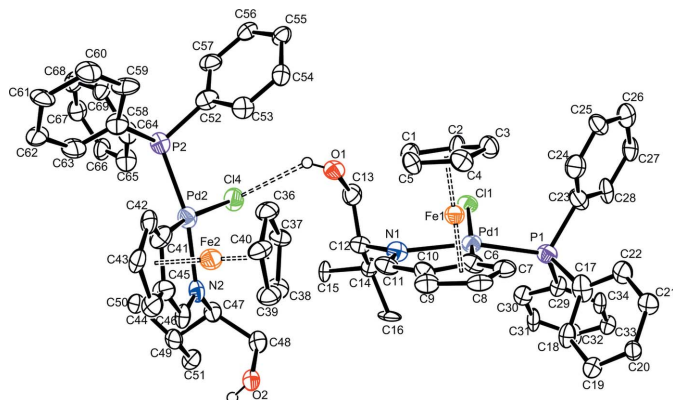
Figure 1
The structures of the palladium complex molecules in the asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. Hydrogen bonds are indicated by dashed lines.

Experimental

A mixture of equimolar ferrocenylaldimine (1 mmol), sodium acetate (1 mmol) and lithium palladium tetrachloride (10 ml, 0.1 mol l^{-1}) in dry methanol (15 ml) was stirred for 20 h at room temperature. The mixture was evaporated to dryness under reduced pressure. Triphenylphosphine (1.5 mmol) was added to a dichloromethane solution (10 ml) of this product and the mixture stirred at room temperature for 2 h. The mixture was purified by chromatography on a silica gel developed by the elute (dichloromethane) in a 85% yield and recrystallized from dichloromethane–hexane (2:1). Orange single crystals of (I) were obtained after 3 d. Analysis calculated for $C_{35}H_{37}Cl_3FeNOPPd$: C 53.37, H 4.70, N 1.78%; found: C 53.68, H 4.70, N 1.79%.

Crystal data

[FePd(C$_5$H$_5$)(C$_{11}$H$_{15}$NO)Cl-(C$_{18}$H$_{15}$P)]·CH$_2$Cl$_2$	$V = 3549.1$ (12) Å^3
$M_r = 787.23$	$Z = 4$
Monoclinic, $P2_1$	$D_x = 1.473$ Mg m^{-3}
$a = 10.229$ (3) Å	Mo $K\alpha$ radiation
$b = 32.4690$ (18) Å	$\mu = 1.21$ mm^{-1}
$c = 10.6860$ (16) Å	$T = 293$ (2) K
$\beta = 90.130$ (13)°	Block, orange
	0.32 × 0.25 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	18654 measured reflections
	12232 independent reflections
φ and ω scans	10975 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{int} = 0.033$
$T_{min} = 0.70$, $T_{max} = 0.77$	$\theta_{max} = 26.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.09P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.062$	$+ 1.99P]$
$wR(F^2) = 0.146$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
12232 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
773 parameters	$\Delta\rho_{min} = -0.96$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Absolute structure: Flack (1983), 5117 Friedel pairs
	Flack parameter: 0.05 (3)

Table 1
Selected geometric parameters (Å, °).

C11—N1	1.266 (10)	N2—Pd2	2.124 (7)
C12—N1	1.468 (10)	P1—Pd1	2.232 (2)
C46—N2	1.278 (10)	P2—Pd2	2.247 (2)
N1—Pd1	2.181 (7)		
C13—O1—H1C	107 (9)	N1—Pd1—Cl1	98.50 (18)
C48—O2—H2C	105 (8)	N2—Pd2—Cl4	94.48 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2C$\cdots$Cl1^i	0.96 (14)	2.60 (13)	3.178 (6)	119 (9)
O1—H1C$\cdots$Cl4	0.92 (14)	2.65 (14)	3.382 (6)	136 (11)

Symmetry code: (i) $x + 1, y, z$.

The O—H atoms were located in a Fourier difference map and refined with isotropic displacement parameters of 0.08 Å^2. All other H atoms were placed in calculated positions, C—H = 0.93–0.98 Å, and refined using a riding model, with $U_{iso} = 1.2$ or 1.5 times U_{eq}(C).

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

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