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Key indicators

Single-crystal X-ray study
 $T = 110\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.033
 wR factor = 0.077
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**{1,1'-Bis[bis(5-methyl-2-furyl)phosphino]-
ferrocene- κ^2P,P' }dichloroplatinum(II)
dichloromethane hemisolvate**

The synthesis of the title compound, $[\text{FePtCl}_2(\text{C}_{15}\text{H}_{14}\text{O}_2\text{P})_2] \cdot 0.5\text{CH}_2\text{Cl}_2$, has been achieved by reacting $\text{PtCl}_2(\text{PhCN})_2$ with the 1,1'-bis[bis(5-methyl-2-furyl)phosphino]ferrocene chelating ligand. The environment at the Pt atom can be described as distorted square-planar, with two *cis*-Cl atoms and two *cis*-P atoms.

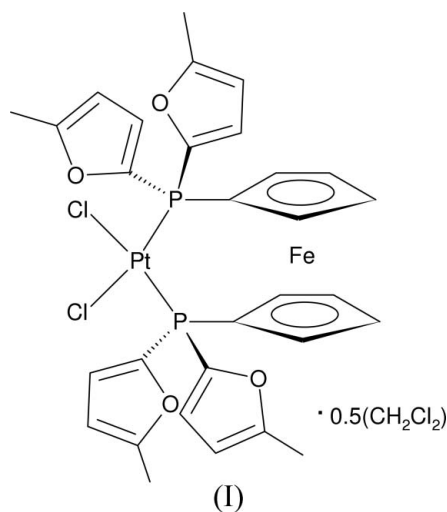
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Comment

For three decades, the 1,1'-bis(diphenylphosphino)ferrocene (dppf) complexes of precious metals have been used as homogeneous catalysts (Gan & Hor, 1995; Cullen & Woolins, 1981; Hayashi *et al.*, 1984; Brown & Cooley, 1990; Itoh *et al.*, 1992). The use of dppf complexes of platinum in carbon-carbon and carbon-heteroatom coupling reactions has been reviewed (Colacot, 2001). Among other noticeable applications of $\text{PtCl}_2(\text{dppf})$ is its use in supramolecular chemistry for the self-assembly of macrocyclic squares incorporating Pt atoms at the corner (Stang *et al.*, 1996). Our group has recently reported the synthesis and X-ray characterization of the palladium complex analogous to the title compound (Fihri *et al.*, 2005). The catalytic activity we found in nucleophilic allylic amination reactions (Tsuji-Trost allylic substitutions) for the system combining 1,1'-bis[di(5-methyl-2-furyl)phosphino]ferrocene with palladium was unprecedented in terms of high turnover frequencies (Hierso *et al.*, 2005). We report here the preparation and crystal structure of the related platinum complex, (I), which is readily obtained from the reaction of 1,1'-bis[di(5-methyl-2-furyl)phosphino]ferrocene with $\text{PtCl}_2(\text{PhCN})_2$.



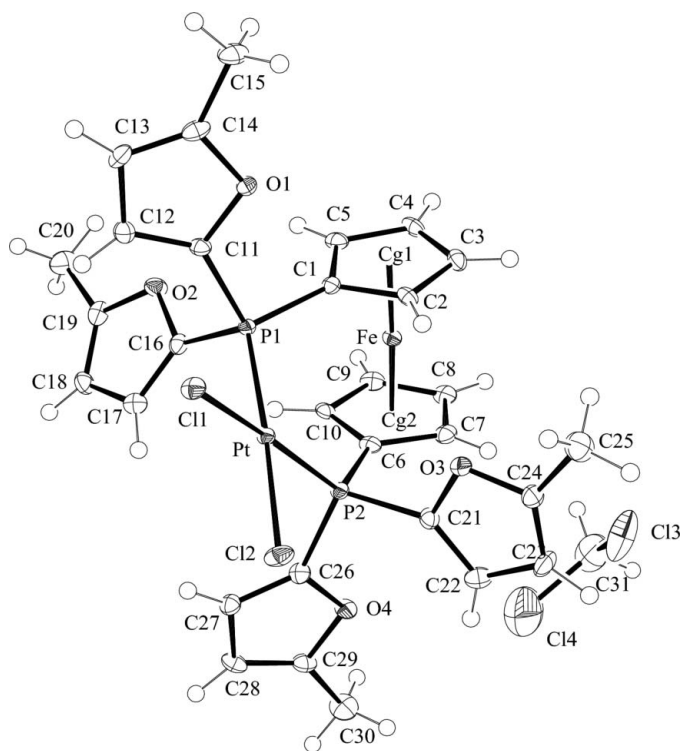


Figure 1
A view of (I), with 50% probability displacement ellipsoids.

The molecular structure of (I) shows that the chelating ferrocenylphosphine ligand adopts a staggered conformation for the cyclopentadienyl rings. The cyclopentadienyl rings are nearly parallel, with a dihedral angle of $7.1(1)^\circ$. The environment at the Pt atom can be described as distorted square-planar, with two *cis*-Cl atoms and two *cis*-P atoms. The P—Pt—P angle of $97.35(4)^\circ$ and the Cl—Pt—Cl angle of $87.61(4)^\circ$ agree well with values reported for structures of the type $[P_2PtX_2]$ (see, for example, De Lima & Filguerias, 1995). The Pt—P bond length of $2.241(1) \text{ \AA}$ is slightly shorter than the corresponding bond length in $PtCl_2(dppf)$ (2.260 \AA). The P1—Cg1—Cg2—P2 torsion angle is $-38.2(2)^\circ$ (Cg1 and Cg2 are the centroids of the cyclopentadienyl rings C1—C5 and C6—C10, respectively). Structural data on such chelated complexes remain of interest, especially in relation to catalytic studies, or in comparison with polydentate ligands of higher degree [tri- and tetraphosphines; see, for example, Hierso *et al.* (2003)].

Experimental

All experiments were conducted under an inert atmosphere of argon using conventional vacuum-line glasswork techniques. The solvents were dried and distilled prior to use. A solution of 1,1'-bis[di(5-methyl-2-furyl)phosphino]ferrocene (240 mg, 0.42 mmol) (Hierso *et al.*, 2005) in CH_2Cl_2 (20 ml) was added to a solution of dichlorobis(benzonitrile)platinum(II) (200 mg, 0.42 mmol) in CH_2Cl_2 (25 ml). After stirring the solution at room temperature for 20 min, the yellow mixture obtained was filtered twice through SiO_2 (SDC 60 35–70 μm) and dried *in vacuo* to give 160 mg (0.19 mmol) of the title compound. An analytically pure sample was obtained by recrystallization from a

dichloromethane–hexane mixture. Analysis, calculated for $C_{30}H_{28}FeP_2O_4PtCl_2 \cdot 0.5CH_2Cl_2$: C 48.20, H 3.81%; found: C 48.98, H 3.47%. Spectroscopic analysis: 1H NMR (300.13 MHz, $CDCl_3$, δ , p.p.m.): Cp is cyclopentadienyl and Fu is furyl): 2.27 (s, 12H, ^{Me}Fu), 4.26 and 4.32 (m, 8H, Cp), 5.22 and 6.00 (m, 8H, $^H Fu$); ^{31}P NMR (121.49 MHz, $CDCl_3$, δ , p.p.m.): -23.42 (singlet, $^1J_{Pt-P} = 3763.7$ Hz); $^{13}C\{^1H\}$ NMR (75.47 MHz, $CDCl_3$, δ , p.p.m.): 13.10 (s, 4C, ^{Me}Fu), 70 (d, 2C, $^1J_{CP} = 111$ Hz, Cp), 72.70 and 74.21 (m, 8C, CpCH), 107 [m, 4C, sp^2 CH=C(O)Me], 125 [m, 4C, sp^2 CH=C(O)], 139.70 [dd, 4C, $^1J_{CP} = 251$ Hz, P—C(O)=C], 157.52 [m, 4C, Me—C(O)=C].

Crystal data

$[FePtCl_2(C_{15}H_{14}O_2P)_2] \cdot 0.5CH_2Cl_2$
 $M_r = 878.77$
 Triclinic, $P\bar{1}$
 $a = 10.6903(2) \text{ \AA}$
 $b = 10.7487(3) \text{ \AA}$
 $c = 14.9344(4) \text{ \AA}$
 $\alpha = 80.926(1)^\circ$
 $\beta = 78.379(1)^\circ$
 $\gamma = 68.302(1)^\circ$
 $V = 1555.24(7) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.877 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 3814 reflections
 $\theta = 1-27.5^\circ$
 $\mu = 5.36 \text{ mm}^{-1}$
 $T = 110(2) \text{ K}$
 Plate, orange
 $0.25 \times 0.25 \times 0.08 \text{ mm}$

Data collection

Nonius KappaCCD area-detector diffractometer
 φ scans ($\kappa = 0$) + additional ω scans
 Absorption correction: multi-scan (Blessing, 1995)
 $T_{min} = 0.162$, $T_{max} = 0.335$
 8983 measured reflections

6868 independent reflections
 6137 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.034$
 $\theta_{max} = 27.6^\circ$
 $h = -13 \rightarrow 13$
 $k = -13 \rightarrow 13$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.077$
 $S = 1.03$
 6868 reflections
 393 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 4.3017P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 1.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -2.24 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cg1 and Cg2 are the centroids of the cyclopentadienyl rings C1—C5 and C6—C10, respectively			
Fe—Cg1	1.644 (5)	Pt—Cl1	2.3385 (10)
Fe—Cg2	1.648 (5)	Pt—Cl2	2.3475 (11)
Pt—P2	2.2416 (10)		
Cg1—Fe—Cg2	177.4 (2)	P1—Pt—Cl2	176.78 (4)
P1—Pt—Cl1	90.44 (4)	P2—Pt—Cl2	84.76 (4)
P2—Pt—Cl1	171.47 (4)		

The CH_2Cl_2 solvent molecule is located close to an inversion centre and was therefore refined with an occupancy factor of 0.5. H atoms were located in difference maps and treated as riding atoms, with C—H distances in the range $0.93-0.97 \text{ \AA}$ and with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl groups.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP III*

(Johnson & Burnett, 1996); software used to prepare material for publication: *SHELXL97* and *WinGX* (Farrugia, 1999).

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