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Chloro(2-{[*N*-(4-methoxybenzyl)methylamino]-methyl}ferrocene-κ^2N,C^1)(triphenylphosphine-κP)-palladium(II)

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.032
wR factor = 0.076
Data-to-parameter ratio = 17.1

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

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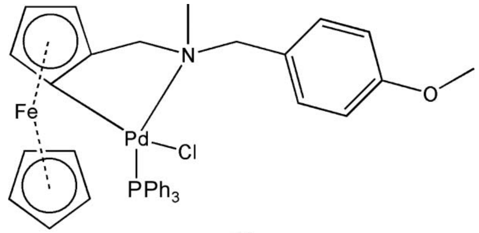
In the title compound, [PdCl{Fe(C_5H_5)($C_{15}H_{17}NO$)}-($C_{18}H_{15}P$)] or [FePd(C_5H_5)($C_{15}H_{17}NO$)Cl($C_{18}H_{15}P$)], the Pd^{II} atom is in a slightly distorted square-planar environment. The dihedral angle between the two cyclopentadienyl rings of the ferrocenyl group is 6.4 (1)°.

Comment

Cyclopalladation of *N*-donor ligands, especially those bearing a ferrocenylimino group, have been extensively studied due to their applications in organic synthesis such as in the Heck reaction (Iyer & Ramesh, 2000) and the Suzuki coupling reaction (Weissmann & Milstein, 1999), *etc*. As a part of our ongoing investigations of cyclometallation of *N*-methyl-*N*-ferrocenylmethylbenzyamines (Wang *et al.*, 2006), a new compound, (I), has been prepared and we report its crystal structure here.



(I)

In the compound (I), atom Pd1 is in a slightly distorted square-planar environment (Fig. 1 and Table 1). Atoms Pd1, P1, N1, Cl1 and C6 deviate from the mean plane through them by 0.0096 (7), −0.1110 (9), −0.1251 (10), 0.0963 (9), 0.1303 (11) Å, respectively. The dihedral angle between the two cyclopentadienyl rings of the ferrocenyl group is 6.4 (1)°. The substituted cyclopentadienyl plane forms a dihedral angle of 82.3 (1)° with the C14–C19 benzene ring. Except for an intramolecular C—H···Cl interaction (Table 2), no hydrogen bonds are observed in the crystal structure.

Experimental

A solution of sodium tetrachloropalladate(II) (290 mg, 1 mmol) in methanol (15 ml) was added dropwise to a stirred solution of {[(*N*-methyl-*N*-4-methoxybenzyl)amino]methyl}ferrocene (350 mg, 1 mmol) and sodium acetate (82 mg, 1 mmol) in methanol (30 ml). The mixture was stirred at room temperature for 4 h. Then triphenylphosphine (410 mg, 1.5 mmol) was added and the mixture was stirred for another 30 min. The solvent was removed *in vacuo* and the residue was purified by column chromatography (silica gel, eluant: ethyl acetate / petroleum ether (333–363 K), 1:3) to give compound (I) (Yield: 80%). Analysis calculated for $C_{38}H_{37}ClFeNOPPd$: C 60.66, H 4.96, N 1.86%; found: C 60.38, H 4.89, N 1.82%.

Crystal data

[FePd(C_5H_5)($C_{15}H_{17}NO$)-Cl($C_{18}H_{15}P$)]
M_r = 752.36
Monoclinic, $P2_1/c$
a = 17.6945 (19) Å
b = 16.1753 (17) Å
c = 12.1585 (13) Å
β = 106.128 (2)°
V = 3343.0 (6) Å^3
Z = 4
D_x = 1.495 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6333 reflections
θ = 2.4–26.3°
μ = 1.13 mm^{-1}
T = 294 (2) K
Prism, red
0.30 × 0.26 × 0.20 mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.670, $T_{\max}$ = 0.798
18615 measured reflections
6828 independent reflections
5059 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036
$\theta_{\max}$ = 26.4°
$h = -22 \rightarrow 20$
$k = -9 \rightarrow 20$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.076
S = 1.02
6828 reflections
399 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.4688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.37 e Å^{-3}
$\Delta\rho_{\min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pd1—C6	1.995 (3)	N1—C12	1.484 (3)
Pd1—N1	2.184 (2)	N1—C13	1.493 (3)
Pd1—P1	2.2264 (8)	N1—C11	1.499 (4)
Pd1—Cl1	2.3889 (8)		
C6—Pd1—N1	82.70 (10)	C6—Pd1—Cl1	171.93 (8)
C6—Pd1—P1	91.36 (8)	N1—Pd1—Cl1	91.80 (7)
N1—Pd1—P1	170.80 (6)	P1—Pd1—Cl1	94.79 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···Cl1	0.97	2.75	3.387 (3)	124

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The U^{ij} components of the atoms C23 and C24 were restrained to approximately isotropic behaviour.

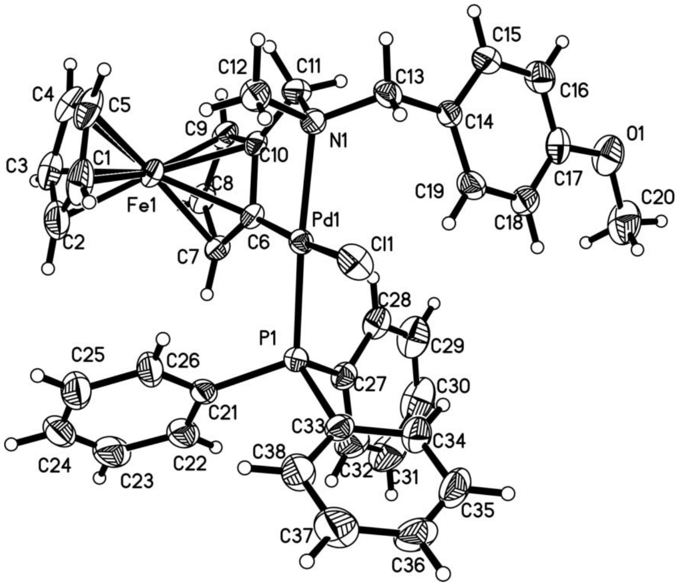


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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