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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}—\text{C}) = 0.005\text{ Å}$
 R factor = 0.029
 wR factor = 0.068
Data-to-parameter ratio = 14.4

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

Chloro[2-{1-[*N*-methyl-*N*-(4-methylbenzyl)-amino]ethyl}ferrocenyl- $\kappa^2\text{N},\text{C}^1$](triphenylphosphine- κP)palladium(II)

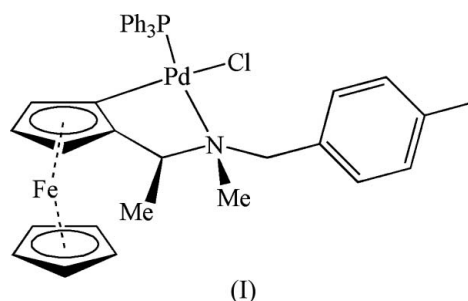
In the title compound, $[\text{FePd}(\text{C}_5\text{H}_5)(\text{C}_{16}\text{H}_{19}\text{N})\text{Cl}(\text{C}_{18}\text{H}_{15}\text{P})]$, the Pd^{II} atom is in a slightly distorted square-planar environment. The dihedral angle between the cyclopentadienyl rings of the ferrocenyl group is $0.6(1)^\circ$.

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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 part of our ongoing investigations of cyclometallations on tertiary ferrocenylamines (Wang *et al.*, 2006), the title new compound, (I), has been synthesized and we report its crystal structure here.



In compound (I), atom Pt1 is in a slightly distorted square-planar environment, with a maximum deviation of $0.1490(2)\text{ Å}$ for atom N1 from the five-atom mean plane (defined by atoms Pd1, N1, Cl1, C1 and P1). The maximum deviation from the mean plane of the five-membered palladocycle is $0.1421(2)\text{ Å}$ for atom N1. The dihedral angle between the two above-mentioned planes is $5.1(2)^\circ$.

Experimental

A solution of sodium tetrachloropalladate(II) (150 mg, 0.5 mmol) in methanol (15 ml) was added dropwise to a stirred solution of [1-[[*N*-methyl-*N*-(4-methylbenzyl)]amino]ethyl]ferrocene (175 mg, 0.5 mmol) and sodium acetate (41 mg, 0.5 mmol) in methanol (20 ml). The mixture was stirred at room temperature for 4 h. Triphenylphosphine (220 mg, 0.8 mmol) was then added and the mixture was stirred for another 30 min. The solvent was removed *in vacuo* and the solid residue was recrystallized from anhydrous ethanol to obtain compound (I) (yield 68%). Crystals of (I) were grown by slow evaporation of a dichloromethane solution at room temperature over a period of one week. Analysis, calculated for $\text{C}_{39}\text{H}_{39}\text{ClFeNPPd}$: C 62.42, H 5.24, N 1.87%; found: C 62.67, H 4.98, N 1.99%.

Crystal data

[FePd(C₅H₅)(C₁₆H₁₉N)Cl-
(C₁₈H₁₅P)]

$M_r = 750.38$

Triclinic, $P\bar{1}$

$a = 10.305$ (3) Å

$b = 10.681$ (4) Å

$c = 17.584$ (6) Å

$\alpha = 92.012$ (5)°

$\beta = 105.451$ (4)°

$\gamma = 115.978$ (4)°

$V = 1651.0$ (10) Å³

$Z = 2$

$D_x = 1.509$ Mg m⁻³

Mo $K\alpha$ radiation

$\mu = 1.14$ mm⁻¹

$T = 293$ (2) K

Block, red

$0.28 \times 0.22 \times 0.20$ mm

Data collection

Bruker APEXII CCD area-detector
diffractometer

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.72$, $T_{\max} = 0.80$

9065 measured reflections

5765 independent reflections

4565 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.068$

$S = 1.02$

5765 reflections

400 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.40$ e Å⁻³

$\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pd1—C1	1.990 (3)	N1—C11	1.506 (4)
Pd1—N1	2.244 (2)	C1—C5	1.435 (4)
Pd1—P1	2.2499 (9)	C5—C11	1.503 (4)
Pd1—Cl1	2.3858 (10)		
C1—Pd1—N1	81.22 (10)	P1—Pd1—Cl1	94.31 (4)
C1—Pd1—P1	92.93 (8)	C13—N1—Pd1	97.28 (16)
N1—Pd1—P1	166.55 (6)	C14—N1—Pd1	119.33 (18)
C1—Pd1—Cl1	172.75 (8)	C11—N1—Pd1	110.66 (15)
N1—Pd1—Cl1	91.60 (7)		
C1—Pd1—N1—C13	−98.44 (18)	Cl1—Pd1—N1—C13	82.60 (17)
P1—Pd1—N1—C13	−33.5 (4)	P1—Pd1—N1—C11	82.4 (3)

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 $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, 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 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

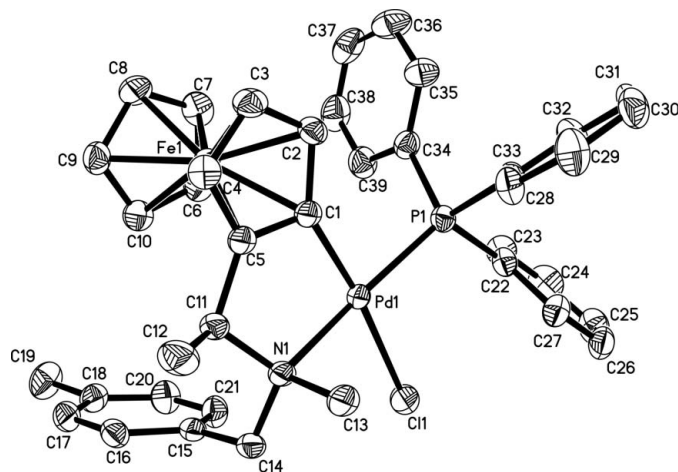


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms have been omitted for clarity.

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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