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Key indicators

Single-crystal X-ray study
 $T = 273$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.024
 wR factor = 0.061
 Data-to-parameter ratio = 15.2

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

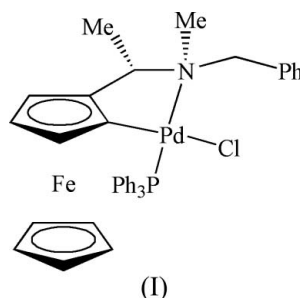
{2-[1-(*N*-Benzyl-*N*-methylamino)ethyl]ferrocenyl- $\kappa^2\text{N},\text{C}^1$ }chloro(triphenylphosphine- κP)palladium(II)

The title compound, $[\text{FePd}(\text{C}_5\text{H}_5)(\text{C}_{15}\text{H}_{17}\text{N})\text{Cl}(\text{C}_{18}\text{H}_{15}\text{P})]$, a new cyclopalladated complex, has been synthesized and structurally characterized. The Pd^{II} atom is in a slightly distorted square-planar environment. The dihedral angle between the cyclopentadienyl rings of the ferrocenyl group is 0.7 (2)°.

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Comment

Cycloplatination reactions have attracted much attention in recent decades due to their applications in organic syntheses (Fossy & Richards, 1999). However, only a few examples of cyclopalladated complexes with *N*-donor ligands have been reported (Ryabov *et al.*, 2002). As part of our ongoing investigation of the cyclometallation of tertiary ferrocenylamines (Wang *et al.*, 2006), the title compound, (I), has been synthesized and we report its crystal structure.



In compound (I), the Pd atom is in a slightly distorted square-planar environment. The coordination plane is defined by the atoms Pd1/N1/Cl1/C1/P3, and the deviations of these atoms from this plane are -0.077 (2), 0.121 (1), -0.070 (2), -0.085 (1) and 0.111 (2) Å, respectively. The dihedral angle between the cyclopentadienyl rings of the ferrocenyl group is 0.7 (2)°.

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*-benzyl)amino]ethyl}ferrocene (167 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 63%). Crystals of (I) were grown by slow evaporation of an ethyl acetate–petroleum ether solution (3:1) at room temperature over a period of one week. Analysis, calculated for $\text{C}_{38}\text{H}_{37}\text{ClFeNPPd}$: C 61.98, H 5.06, N 1.90%; found: C 61.77, H 5.00, N 1.89%.

Crystal data

[Pd(C₅H₅)(C₁₅H₁₇N)Cl(C₁₈H₁₅P)]
M_r = 736.36
 Orthorhombic, *Pbca*
a = 16.878 (4) Å
b = 16.488 (4) Å
c = 24.107 (5) Å
V = 6709 (2) Å³

Z = 8
D_x = 1.458 Mg m⁻³
 Mo *K*α radiation
 μ = 1.12 mm⁻¹
T = 273 (2) K
 Block, red
 0.32 × 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} = 0.701, *T_{max}* = 0.801

34744 measured reflections
 5924 independent reflections
 4851 reflections with *I* > 2σ(*I*)
R_{int} = 0.033
 θ_{max} = 25.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.024
wR(*F*²) = 0.061
S = 1.02
 5924 reflections
 390 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 4.0356P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Pd1—C1	1.993 (2)	N1—C11	1.512 (3)
Pd1—N1	2.1962 (19)	C1—C5	1.437 (3)
Pd1—P3	2.2452 (7)	C5—C11	1.497 (4)
Pd1—Cl1	2.4107 (8)		
C1—Pd1—N1	81.93 (8)	C1—Pd1—Cl1	174.71 (7)
C1—Pd1—P3	98.56 (7)	N1—Pd1—Cl1	92.81 (6)
N1—Pd1—P3	169.99 (6)	P3—Pd1—Cl1	86.71 (2)

All H atoms were initially located in difference Fourier maps. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and *U_{iso}*(H) = 1.5*U_{eq}*(C), but each group was allowed to rotate freely about its C—C bond. The 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_{iso}*(H) = 1.2*U_{eq}*(C). There are solvent-accessible voids of 53 Å³ in the structure. However, the highest peak of the residual density is located 1.32 Å from the Pd atom.

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve

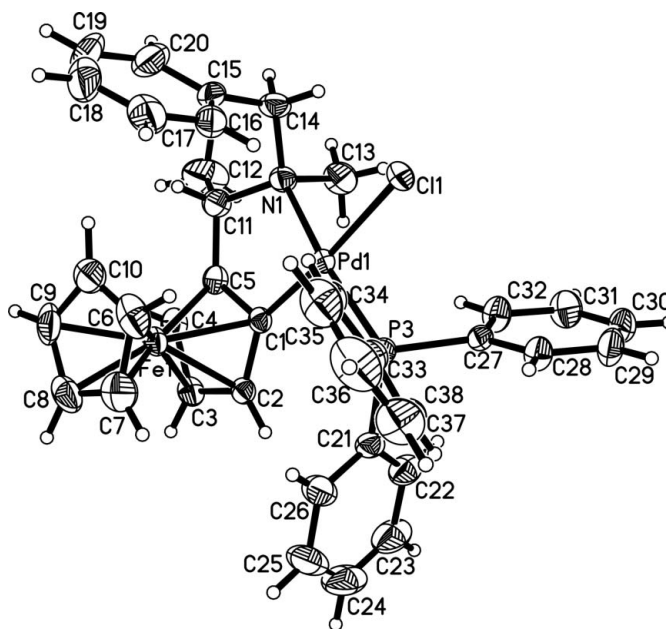


Figure 1

A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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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References

- Bruker (1998). SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2003). SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
- Fossy, J. S. & Richards, C. J. (1999). Organometallics, **18**, 2116–2124.
- Ryabov, A. D., Panyashkina, I. M., Polyakov, V. A. & Fischer, A. (2002). Organometallics, **21**, 1633–1636.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXL97 and SHELXS97. University of Göttingen, Germany.
- Wang, H.-X., Wu, H.-F., Geng, F.-Y., Gao, R.-Q. & Zhou, H.-C. (2006). Acta Cryst. E62, m14–m15.