

Xiao-Lin Zhang, Ren-Yun Kuang,
Qian-Yong Cao* and Lai-Tao LuoDepartment of Chemistry, Nanchang University,
Nanchang 330047, People's Republic of China

Correspondence e-mail: cqyong@ncu.edu.cn

Key indicators

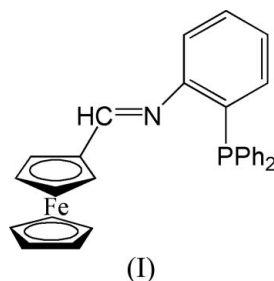
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.012$ Å
 R factor = 0.079
 wR factor = 0.192
Data-to-parameter ratio = 15.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.[2-(Diphenylphosphino)phenyliminomethyl]-
ferrocene

The title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{24}\text{H}_{19}\text{NP})]$, a new Schiff base containing a ferrocenyl (Fc) group, has been synthesized and characterized structurally. The compound is a *trans* isomer with the $\text{C}_{\text{Fc}}-\text{C}-\text{N}-\text{C}$ linkage almost planar.

Received 27 July 2006
Accepted 31 August 2006

Comment

Ferrocene and its derivatives have attracted much attention for their potential applications as molecular sensors, as magnetic and optical materials, and in homogeneous catalysis (Togni & Hayashi, 1995; Long, 1995; Beer *et al.*, 1999). Schiff bases derived from ferrocenecarbaldehyde and their metal complexes are quite attractive because of their rather simple syntheses. Several studies on this class of ferrocene-containing molecules have been reported recently (Wu *et al.*, 2001; López *et al.*, 2005). We have synthesized such a compound, (I), and present its structure here.



The molecular structure of (I) is in a *trans* configuration of the benzene ring and the cyclopentadienyl (Cp) ring about the $\text{N}=\text{C}19$ bond; the dihedral angle between the planes $\text{C}19-\text{C}24$ and $\text{N}/\text{C}13-\text{C}18$ is $4.47(5)^\circ$. In the Fc group, the $\text{Fe}-\text{C}$ bond lengths (Table 1) are within the normal range (Huo *et al.*, 1995). The $\text{Fe}\cdots\text{C}g1$ and $\text{Fe}\cdots\text{C}g2$ distances are 1.637 and 1.645 Å, respectively, and the $\text{C}g1\cdots\text{Fe}\cdots\text{C}g2$ angle is 177.5° , where $\text{C}g1$ and $\text{C}g2$ are the centroids of the substituted and unsubstituted Cp rings, respectively. The two Cp rings are almost eclipsed, with a dihedral angle of $2.37(5)^\circ$, and the largest torsion angle $\text{C}21\cdots\text{C}g1\cdots\text{C}g2\cdots\text{C}27$ is 2.9° . In the triphenylphosphine group, the P atom adopts a slightly distorted pyramidal geometry.

Experimental

The raw material 2-(diphenylphosphino)aniline was prepared by a literature method (Cooper *et al.*, 1992), and formylferrocene was commercially obtained. Formylferrocene (1.024 g, 4.78 mmol) was added to a solution of 2-(diphenylphosphino)aniline (1.325 g, 4.78 mmol) in benzene (50 ml). The reaction flask was connected to a

condenser and a Dean–Stark apparatus. The mixture was refluxed until *ca* 15 ml of benzene–water azeotrope had condensed on the Dean–Stark apparatus. The reaction mixture was then concentrated on a rotary evaporator to about 10 ml. Slow evaporation of the solvent afforded the red title compound (1.833 g, yield 81%). M.p. 400–401 K. Elemental analysis calculated for C₂₉H₂₄FeNP: C 73.59, H 5.11, N 2.96%; found C 73.67, H 5.05, N 2.82%. Single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of a CH₃CN solution at 293 K.

Crystal data

[Fe(C ₅ H ₅)(C ₂₄ H ₁₉ NP)]	Z = 8
M _r = 473.31	D _x = 1.353 Mg m ⁻³
Orthorhombic, <i>Pbca</i>	Mo K α radiation
a = 13.902 (3) Å	μ = 0.73 mm ⁻¹
b = 9.3370 (19) Å	T = 293 (2) K
c = 35.815 (7) Å	Needle, red
V = 4648.9 (16) Å ³	0.40 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	4553 independent reflections
$\omega/2\theta$ scans	1939 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North <i>et al.</i> ; 1968)	$\theta_{\max} = 26.0^\circ$
$T_{\min} = 0.758$, $T_{\max} = 0.930$	3 standard reflections
4553 measured reflections	every 200 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 4.5P]$
$R[F^2 > 2\sigma(F^2)] = 0.079$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.192$	$(\Delta/\sigma)_{\max} < 0.001$
S = 0.95	$\Delta\rho_{\max} = 0.27 \text{ e \AA}^{-3}$
4553 reflections	$\Delta\rho_{\min} = -0.31 \text{ e \AA}^{-3}$
289 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Fe–C21	2.012 (7)	Fe–C20	2.039 (7)
Fe–C24	2.021 (8)	Fe–C28	2.039 (8)
Fe–C22	2.022 (7)	Fe–C23	2.040 (8)
Fe–C27	2.033 (8)	P–C7	1.818 (7)
Fe–C29	2.033 (8)	P–C13	1.826 (7)
Fe–C25	2.035 (7)	P–C1	1.843 (7)
Fe–C26	2.035 (8)	N–C19	1.260 (8)
C7–P–C13	102.8 (3)	C13–P–C1	102.5 (3)
C7–P–C1	101.0 (3)		

All H atoms were positioned geometrically and treated as riding (C–H = 0.98 Å for cyclopentadienyl rings and 0.93 Å for other H atoms); $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

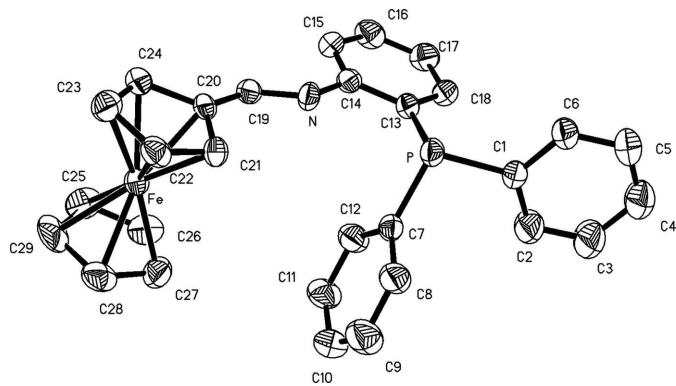


Figure 1

Perspective structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and all H atoms have been omitted for clarity.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

This work was supported by the Bureau of Education of Jiangxi Province (2006, No. 29). We are also grateful to the Center of Analysis and Testing, Nanchang University, for financial support (Foundation No. 2006020).

References

- Beer, P. D., Gale, P. A. & Chen, G. Z. (1999). *Coord. Chem. Rev.* **185–186**, 3–36.
- Bruker (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cooper, M. K., Downes, J. M., Duckworth, P. A. & Tiekink, E. R. (1992). *Aust. J. Chem.* **45**, 595–609.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Huo, S. Q., Zhu, Y. & Wu, Y. J. (1995). *J. Organomet. Chem.* **490**, 243–247.
- Long, N. J. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 21–38.
- López, C., Pérez, S. & Font-Bardia, M. (2005). *J. Organomet. Chem.* **690**, 228–243.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Togni, A. & Hayashi, T. (1995). *Ferrocenes – Homogeneous Catalysis, Organic Synthesis, Materials Science*. Weinheim: VCH.
- Wu, Y., Huo, S., Gong, J., Cui, X., Ding, L., Ding, K., Du, C., Liu, Y. & Song, M. (2001). *J. Organomet. Chem.* **637–639**, 27–46.