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# 1,1′-Bis{α-[(4-methylbenzyl)imino]benzyl}ferrocene

**Hong-Xing Wang,* Ren-Qing Gao, Hong-Fei Wu, Feng-Ying Geng and Xiao-Li Yang**

Department of Chemistry, College of Sciences, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: hongxing_wang@hotmail.com

**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.038
*wR* factor = 0.104
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.



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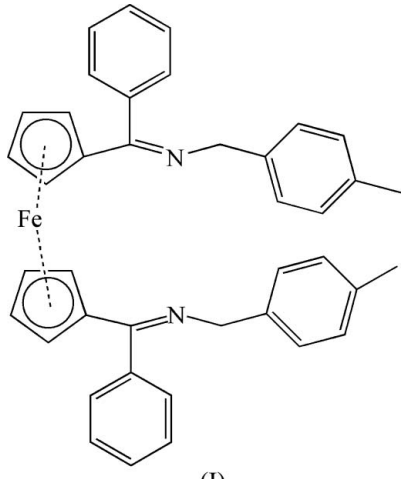
The title compound, $[Fe(C_{20}H_{18}N)_2]$, a new 1,1′-disubstituted ferrocenylketimine derivative, has been synthesized and characterized. All the bond lengths are within normal range.

## Comment

The cyclometallation reaction of ferrocenyl derivatives, especially those bearing *N*-donor ligands, is one of the most advanced research areas of modern organometallic chemistry, because of the wide-ranging application in organic synthesis, such as in the Heck reaction and the Suzuki cross-coupling reaction (Cassol *et al.*, 2005). In the course of our investigation of the cyclometalation on tertiary ferrocenylamines, we observed that the title compound, (I), was an excellent candidate for cyclometallation. Its crystal structure is reported here (Fig. 1).




(I)

In the compound, all the bond lengths are within normal range (Allen *et al.*, 1987). The N1—C6 and N2—C26 bond lengths (Table 1) confirm that they are C=N double bonds. The torsion angles C33—N2—C26—C27 and C13—N1—C6—C7 are 0.3 (4) and 8.0 (4)°, respectively.

## Experimental

A solution of 1,1′-dibenzoylferrocene (3.94 g, 10 mmol), 4-methylbenzylamine (10 ml, 100 mmol) and *p*-toluenesulfonic acid (100 mg) in methanol (100 ml) was heated under reflux for 12 h with azeotropic removal of water. The mixture was then concentrated on a rotary evaporator and the solid residue was recrystallized from absolute ethanol. A red solid product was obtained. Analysis calculated for $C_{40}H_{36}FeN_2$: C 80.00, H 6.04, N 4.66%; found: C 79.95, H 6.22, N 4.79%.

*Crystal data*

$[Fe(C_{20}H_{18}N)_2]$
$M_r = 600.56$
Monoclinic, $P2_1/n$
$a = 10.827\ (5)$ Å
$b = 10.818\ (5)$ Å
$c = 27.055\ (12)$ Å
$\beta = 91.947\ (8)^\circ$
$V = 3167\ (3)$ Å$^3$
$Z = 4$
$D_x = 1.259$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.51$ mm$^{-1}$
$T = 293\ (2)$ K
Block, red
0.26 × 0.24 × 0.20 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.781$, $T_{max} = 1.000$
(expected range = 0.706–0.904)
15923 measured reflections
5575 independent reflections
3997 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0^\circ$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.104$
$S = 1.02$
5575 reflections
388 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.6967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| N1—C6 | 1.282 (3) | N2—C26 | 1.278 (3) |
| N1—C13 | 1.470 (3) | N2—C33 | 1.467 (3) |
| | | | |
| N1—C6—C5 | 116.9 (2) | N2—C26—C25 | 118.4 (2) |
| N1—C6—C7 | 125.2 (2) | N2—C26—C27 | 124.7 (2) |
| N1—C13—C14 | 107.7 (2) | N2—C33—C34 | 113.3 (2) |
| | | | |
| C13—N1—C6—C7 | 8.0 (4) | C33—N2—C26—C27 | 0.3 (4) |

All H atoms were initially located in a difference Fourier map. All H atoms were then constrained to an ideal geometry, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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*.

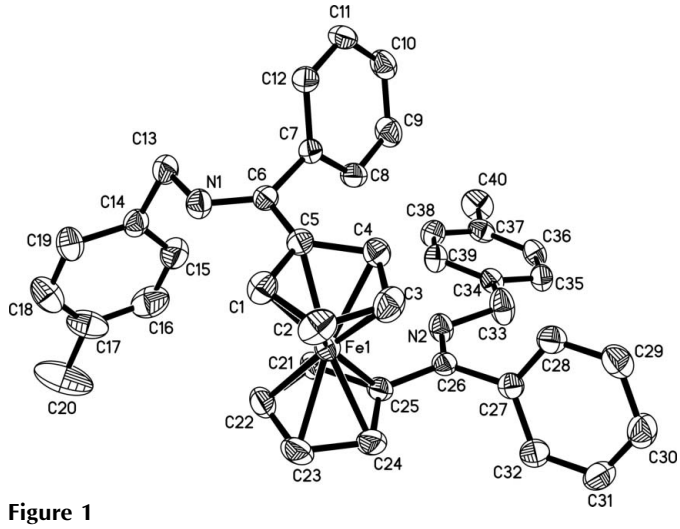



**Figure 1**
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms have been omitted.

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## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cassol, C. C., Umpierre, A. P., Machado, G., Wolke, S. I. & Dupont, J. (2005). *J. Am. Chem. Soc.* **127**, 3298–3299.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.