

1-Ferrocenyl-*N*-(1-phenylethyl)ethylamineHeng-Yu Qian,^{a*} Xiu-Ling Cui^b
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Key indicators

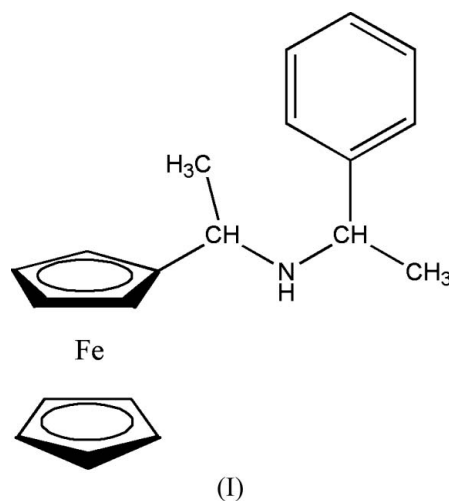
Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$
R factor = 0.056
wR factor = 0.127
Data-to-parameter ratio = 16.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{15}\text{H}_{18}\text{N})]$, has been synthesized as a prochiral agent by refluxing equimolar mixtures of acetylferrocene and 1-phenylethylamine followed by reduction with sodium borohydride. The crystal structure exhibits normal geometrical parameters. The C—N—C angle is $118.6(4)^\circ$ due to the steric effect of the benzene ring and the substituted ferrocene ring.

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Comment

Functionalized ferrocene derivatives are widely used ligands in many fields, such as asymmetric catalysis (Hyshi *et al.*, 1988; Pastor & Togni, 1989) and coupling reactions (Trost & Vranken, 1996). The synthesis of (*R,R*)-*N*-(1-phenylethyl)-1-ferrocenylethylamine has been reported by David *et al.* (1990), while the structure of α -ferrocenyl-*N*-(1-phenylethyl)benzylamine (Yin & Qian, 2005) has been reported by our group. We report here the results of a single-crystal X-ray diffraction analysis of the title compound, (I). The molecular structure of (I) is shown in Fig. 1. Selected bond lengths and angles are given in Table 1. The cyclopentadienyl rings of the ferrocene fragment are planar and parallel. The C10—C11—C12 and C14—C13—C20 angles are $113.9(4)^\circ$ and $110.8(4)^\circ$ respectively. In the crystal structure, molecules are linked by C—H $\cdots\pi$ interactions (Table 2, Fig. 2). C—H $\cdots\pi$ interactions are formed between C4 and C17, which act as the hydrogen-bond donors, and the phenyl ring (C14—C19) and the ferrocene ring (C1—C5), respectively, which act as acceptors (Steiner *et al.*, 1995).



Experimental

Compound (I) was prepared according to a literature method (David *et al.*, 1990). Acetylferrocene was converted to the ferrocenylimine in

87% yield by treatment with 1-phenylethylamine in dry toluene solvent for 20 h followed by reduction with sodium borohydride in methanol solvent at 273 K for 20 h. The product was separated by flash chromatography on silica gel using chloroform:ethyl acetate (4:1) as eluant and crystallized from a dichloromethane-hexane solution in 72% yield by slow evaporation of the solvent. Spectroscopic analysis: IR (KBr, ν , cm^{-1}): 3431, 3079, 2959, 1604, 1488, 1447, 1130, 1007, 834; $^1\text{H NMR}$ (CDCl_3 , δ , p.p.m.): 7.26–7.37 (*m*, 5H), 4.10–4.16 (*m*, 4H), 4.07 (*s*, 5H), 3.81 (*d* \times *d*, 1H, $J=6.4$ Hz and $J=12.8$ Hz) 3.33 (*d* \times *d*, 1H, $J=6.8$ Hz and $J=13.4$ Hz) 1.41 (*d*, 3H, $J=6.8$ Hz), 1.24 (*d*, 3H, $J=6.8$ Hz). Analysis: calculated for $\text{C}_{20}\text{H}_{23}\text{FeN}$: C 72.07, H 6.91, N 4.20%; found: C 71.85, H 7.02, N 4.53%.

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{15}\text{H}_{18}\text{N})]$
 $M_r = 333.24$
 Orthorhombic, $P2_12_12_1$
 $a = 7.3401$ (15) Å
 $b = 11.688$ (2) Å
 $c = 19.841$ (4) Å
 $V = 1702.1$ (6) Å³
 $Z = 4$
 $D_x = 1.300$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 1335 reflections
 $\theta = 2.2$ – 25.3°
 $\mu = 0.88$ mm⁻¹
 $T = 291$ (2) K
 Block, orange
 $0.27 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{\text{min}} = 0.797$, $T_{\text{max}} = 0.857$
 7239 measured reflections

3222 independent reflections
 2629 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -8 \rightarrow 9$
 $k = -14 \rightarrow 14$
 $l = -24 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.127$
 $S = 1.01$
 3222 reflections
 200 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.66P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.56$ e Å⁻³
 Absolute structure: Flack (1983), 1291 Friedel pairs
 Flack parameter: 0.08 (3)

Table 1

Selected geometric parameters (Å, °).

C10–C11	1.481 (7)	C13–C14	1.510 (7)
C11–C12	1.545 (6)	C13–C20	1.523 (7)
N1–C13–C14	115.0 (5)	C15–C14–C13	119.4 (5)
N1–C13–C20	107.8 (4)	C16–C15–C14	121.8 (5)
C14–C13–C20	110.8 (4)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C4–H4A...Cg3 ⁱ	0.98	2.99	3.819 (6)	143
C17–H17A...Cg1 ⁱⁱ	0.93	3.05	3.932 (6)	158

Symmetry codes: (i) $-x + \frac{3}{2}, -y, z - \frac{1}{2}$; (ii) $-x + \frac{5}{2}, -y, z + \frac{1}{2}$. Cg1 and Cg3 are the centroids of rings C1–C5 and C14–C19, respectively

All H atoms were placed in calculated positions and were refined, using a riding model, with C–H = 0.93–0.98 Å, N–H = 0.86 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

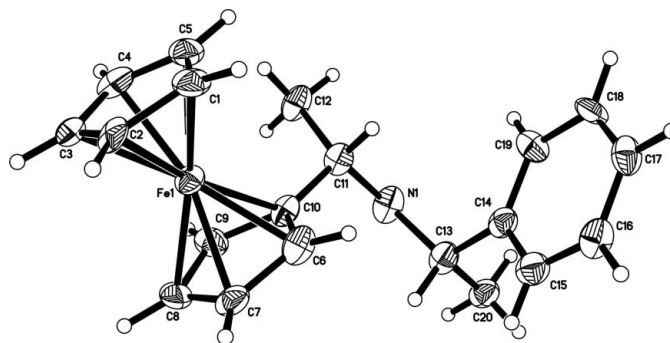


Figure 1 The structure of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

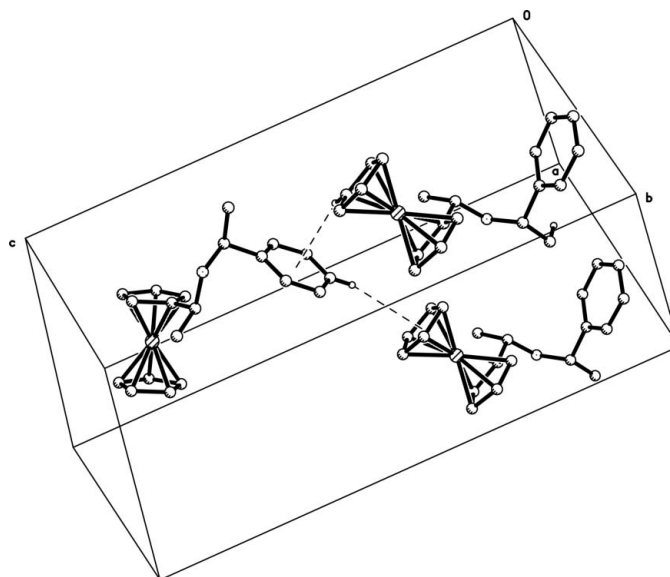


Figure 2 Packing of (I). Dashed lines indicate C–H... π interactions.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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