α-Ferrocenyl-N-(1-phenylethyl)benzylamine

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Key indicators

Single-crystal X-ray study

$T = 291$ K

Mean σ(C–C) = 0.007 Å

R factor = 0.058

wR factor = 0.113

Data-to-parameter ratio = 15.8

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

The title compound, [Fe(C_5H_5)($C_{20}H_{20}N$)], possesses normal geometrical parameters. A network of C—H···π interactions, one of which is bifurcated, helps to consolidate the crystal packing.

Comment

Functionalized ferrocene derivatives have recently been the subject of renewed interest owing to their promise in applications such as homogeneous catalysis (Sawamura & Ito,1992; Togni, 1996; Borman, 1996) and the construction of carbon–carbon or carbon–heteroatom bonds (Trost *et al.*, 1996; Sesay *et al.*, 1998). We report here the synthesis and crystal structure of the title compound, (I) (Fig. 1).
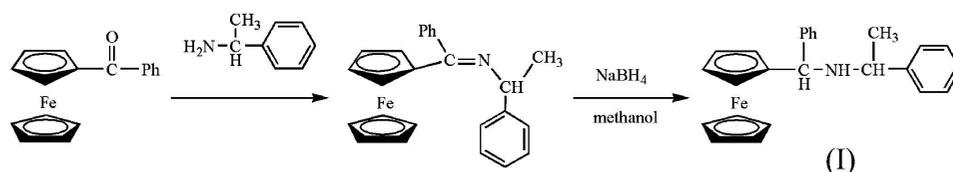
All geometrical values for (I) are normal. The two ferrocene rings are almost parallel [dihedral angle = 2.7 (3)°]. Conversely, the dihedral angle between the two side-chain phenyl rings is 75.2 (4)°.

In the crystal structure of (I), the molecules are linked by C—H···π interactions (Table 1), which involve both ferrocene rings (C1–C5 and C6–C10) and the phenyl ring (C12–C17) as acceptors (Steiner *et al.*, 1995). These bonds result in the formation of a two-dimensional network (Fig. 2).
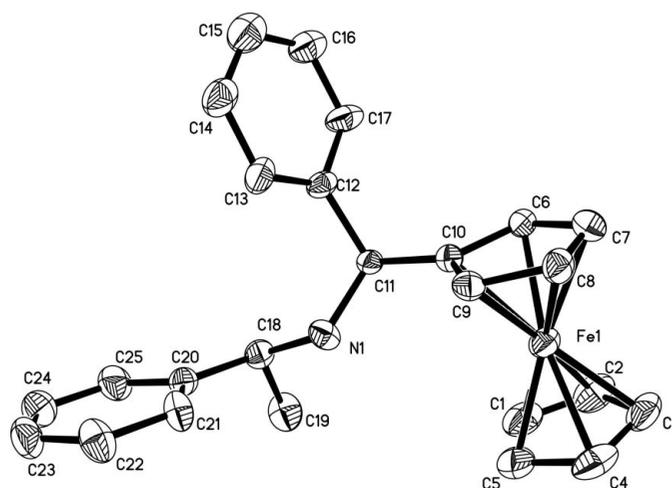
Figure 1

The structure of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

metal-organic papers
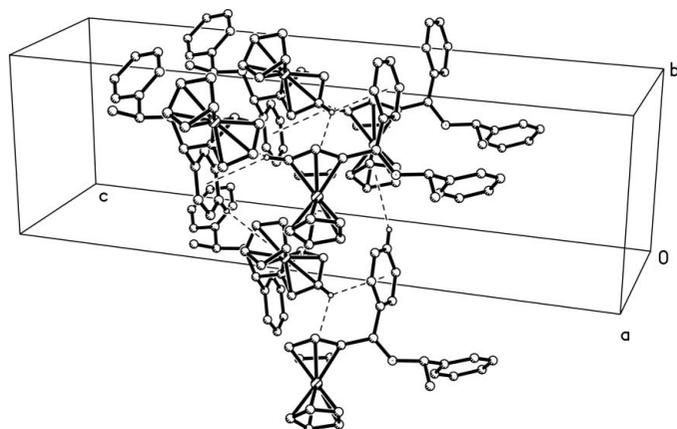
Figure 2
Packing of (I), with dashed lines indicating C—H···π interactions. All H atoms except, H7A and H15A, have been omitted for clarity.

Experimental

The method of David *et al.* (1990) was followed; an equimolar mixture of benzoylferrocene and 1-phenylethylamine in dry toluene was refluxed for 24 h and afforded a crude mixture in 85% yield. The mixture was reduced by sodium borohydride in methanol at 273 K for 20 h (see scheme). The product was purified by chromatography on silica gel (petroleum ether/dichloromethane, 2:1) to give a 65% yield and recrystallized from dichloromethane/hexane to yield orange blocks of (I). IR (KBr, cm^{-1}): ν 3325, 3022, 2962, 1742, 1599, 1449, 1102, 1021, 699; ^{1}H NMR (CDCl$_3$): δ 7.2–7.36 (*m*, 10H), 4.57 (*s*, 1H), 4.18 (*s*, 1H), 4.08–4.10 (*m*, 3H), 4.01 (*s*, 5H), 3.76 (*d* × *d*, 1H, *J* = 6.2 and 8.8 Hz), 1.93 (*s*, 1H), 1.39 (*d*, 3H, *J* = 6.6 Hz). Analysis calculated for C$_{25}$H$_{25}$FeN: C 75.95, H 6.33, N 3.54%; found: C 75.46, H 6.58, N 3.02%.

Crystal data

[Fe(C$_5$H$_5$)(C$_{20}$H$_{20}$N)]	Mo *K*α radiation
$M_r = 395.31$	Cell parameters from 1875
Orthorhombic, $P2_12_12_1$	reflections
$a = 8.0109$ (16) Å	$\theta = 2.1$–$24.6°$
$b = 8.7198$ (17) Å	$\mu = 0.76$ mm^{-1}
$c = 28.718$ (6) Å	$T = 291$ (2) K
$V = 2006.0$ (7) Å^3	Block, orange
$Z = 4$	$0.20 \times 0.19 \times 0.18$ mm
$D_x = 1.309$ Mg m^{-3}	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3866 independent reflections
	2675 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.043$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 26.0°$
	$h = -9 \rightarrow 9$
$T_{min} = 0.863$, $T_{max} = 0.875$	$k = -10 \rightarrow 10$
9952 measured reflections	$l = -35 \rightarrow 35$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.99$	$\Delta\rho_{max} = 0.61$ e Å^{-3}
3866 reflections	$\Delta\rho_{min} = -0.42$ e Å^{-3}
244 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	1578 Friedel pairs
	Flack parameter: 0.08 (3)

Table 1
Hydrogen-bond geometry (Å, °).

$Cg1$, $Cg2$ and $Cg3$ are the centroids of the C1–C5, C6–C10 and C12–C17 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···$Cg2^{i}$	0.98	3.20	3.895 (5)	129
C7—H7A···$Cg3^{i}$	0.98	3.00	3.749 (5)	134
C15—H15A···$Cg1^{ii}$	0.93	2.85	3.744 (7)	161

Symmetry codes: (i) $-x-1, y+\frac{1}{2}, -z+\frac{5}{2}$; (ii) $x+1, y-1, z$.

All H atoms were placed in calculated positions (N—H = 0.86 Å and C—H = 0.93–0.98 Å) and were refined as riding, with U_{iso}(H) = $1.2U_{eq}$(carrier).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*.

References

Borman, S. (1996). *Chem. Eng. News*, **22**, 38–40.
Bruker (2000). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
David, D. M., Kane-Maguire, A. P. & Pyne, S. G. (1990). *J. Chem. Soc. Chem. Commun.* pp. 889–990.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sawamura, M. & Ito, Y. (1992). *Chem. Rev.* **92**, 857–871.
Sesay, S. J., Williams, J. M. J. (1998). *Adv. Asymm. Synth.* **3**, 235–271.
Steiner, T., Starikov, E. B., Amado, A. M., Teixeira-Dias, J. J. C. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 1321–1326.
Togni, A. (1996). *Angew. Chem. Int. Ed. Engl.* **35**, 1475–1477.
Trost, B. M., Vranken, D. L. (1996). *Chem. Rev.* **96**, 395–422.