

Hong-Xing Wang,^{a,b*} Ying-Jie Li,^a Hong-Fei Wu,^a Hui-Chao Zhou,^a Feng-Ying Geng^a and Ren-Qing Gao^a

^aDepartment of Chemistry, College of Sciences, Tianjin University, Tianjin 300072, People's Republic of China, and ^bState Key Laboratory of Elemento-organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail:
hongxing_wang@hotmail.com

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.028
 wR factor = 0.081
Data-to-parameter ratio = 14.9

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

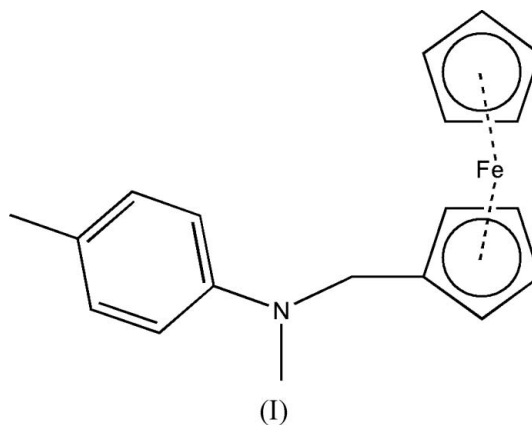
1-[[*N*-Methyl-*N*-(4-methylphenyl)amino]methyl]-ferrocene

In the title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{14}\text{H}_{16}\text{N})]$, the substituted cyclopentadienyl ring is essentially perpendicular to the plane of the benzene ring [dihedral angle = $90.7(2)^\circ$]. There are no important intermolecular interactions.

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Comment

Recently, we have reported a series of tertiary ferrocenylamines (Li *et al.*, 2005; Wang, Li & Hou, 2005; Wang, Li, Wu *et al.*, 2005). As an extension of our work on the structural characterization of tertiary amines, the title compound, (I), is reported here.



In the compound, all the bond lengths are within normal ranges (Allen *et al.*, 1987). Atom N1 and the benzene ring are almost coplanar, with a mean deviation of 0.0085 Å. The dihedral angle between the benzene ring and the plane through atoms C11, N1 and C12 is 27.7° . Owing to the steric effect between the ferrocenyl and benzene groups, the C10—C11—N1 angle is widened to $113.59(18)^\circ$. No obvious intermolecular interactions are observed.

Experimental

To a stirred solution of *N*-(*p*-methylphenyl)aminomethylferrocene (1.525 g, 5 mmol) and 37% aqueous formaldehyde (4 ml, 50 mmol) in acetonitrile (30 ml) was added sodium cyanoborohydride (0.95 g, 15 mmol). A dark residue separated. The reaction mixture was stirred for 30 min; glacial acetic acid was added dropwise until the solution tested neutral on wet pH paper. Stirring was continued for another 1 h. The reaction mixture was poured into diethyl ether (80 ml) and then washed with 1 *N* KOH and saturated brine. The ether solution was dried with K_2CO_3 and evaporated *in vacuo* (yield 82%). Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution at room temperature over a period of a week. ^1H NMR (CDCl_3 , p.p.m.): δ 7.03 (*d*, 2H), 6.71 (*d*,

2H), 4.22 (s, 2H), 4.14 (s, 5H), 4.13 (s, 2H), 4.07 (s, 2H), 2.81 (s, 3H), 2.25 (s, 3H). Analysis calculated for $C_{19}H_{21}FeN$: C 71.49, H 6.33, N 4.39%; found: C 71.39, H 6.91, N 4.60%.

Crystal data

$[Fe(C_5H_5)(C_{14}H_{16}N)]$
 $M_r = 319.22$
 Monoclinic, $P2_1/c$
 $a = 9.587$ (5) Å
 $b = 12.569$ (7) Å
 $c = 13.950$ (7) Å
 $\beta = 105.311$ (6)°
 $V = 1621.3$ (15) Å³
 $Z = 4$

$D_x = 1.308$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3742 reflections
 $\theta = 2.7$ – 26.5°
 $\mu = 0.92$ mm⁻¹
 $T = 293$ (2) K
 Prism, yellow
 $0.38 \times 0.32 \times 0.22$ mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.681$, $T_{max} = 0.816$
 8512 measured reflections

2862 independent reflections
 2447 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$
 $\theta_{max} = 25.0^\circ$
 $h = -9 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.081$
 $S = 1.05$
 2862 reflections
 192 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.3894P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.19$ e Å⁻³
 $\Delta\rho_{min} = -0.22$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0118 (10)

Table 1

Selected geometric parameters (Å, °).

N1–C13	1.398 (3)	C10–C11	1.508 (3)
N1–C12	1.448 (3)	C16–C17	1.516 (4)
N1–C11	1.458 (3)		
C13–N1–C12	118.8 (2)	C12–N1–C11	114.1 (2)
C13–N1–C11	119.65 (18)	N1–C11–C10	113.59 (18)
C7–C6–C10–C11	−178.97 (19)	C11–N1–C13–C14	−153.5 (2)
C12–N1–C13–C14	−5.3 (3)		

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with a C–H distance of 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$; the group was

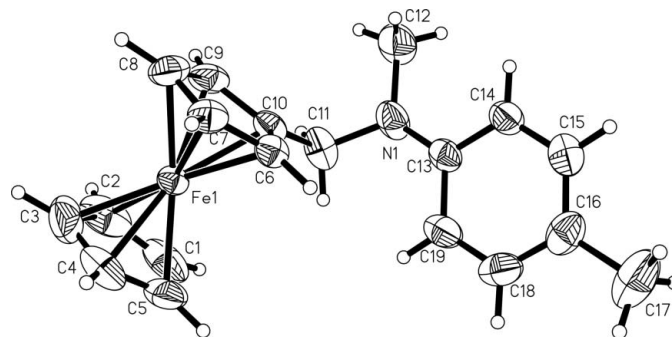


Figure 1

View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.

allowed to rotate freely about the C–C bond. All 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 $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); 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, 1997); software used to prepare material for publication: SHELXTL.

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