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N-Methyl-*N*-(ferrocenylmethyl)aniline

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.100
Data-to-parameter ratio = 17.0

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

The title compound, $[Fe(C_5H_5)(C_{13}H_{14}N)]$, a tertiary arylamine bearing a ferrocenyl group was synthesized by reductive methylation of amines. The result reveals that the cyclopentadiene ring carrying the methylaniline residue is nearly perpendicular to the benzene plane [dihedral angle = 92.1 (3)°].

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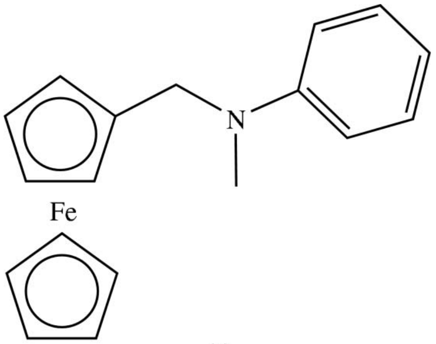
Comment

Ferrocene-containing compounds have been widely studied because of their potential application in catalysis, materials science, hydrometallurgy and as molecular devices (Beer *et al.*, 1997). As a part of our ongoing investigations on cyclometalation of tertiary amines, the title compound, (I), has been prepared and we present here its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The methyl C atom (C12) and the methylene C atom (C11) are displaced from the plane of the phenyl ring by 0.1309 (3) and

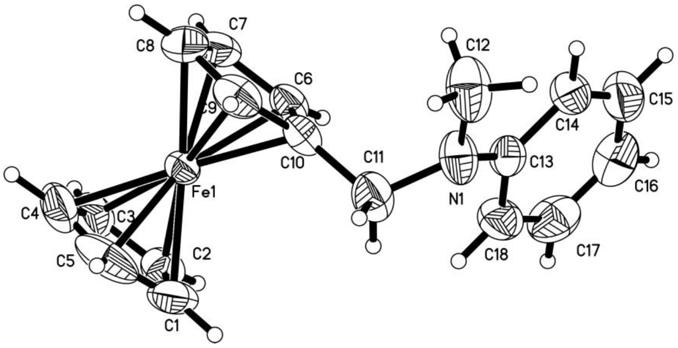


Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms are shown as small spheres of arbitrary radii.

0.3913 (2) Å, respectively. The cyclopentadiene ring carrying the methylaniline residue is nearly perpendicular to the benzene plane [dihedral angle = 92.1 (3) °].

Experimental

A solution of sodium cyanoborohydride (378 mg, 6 mmol) in acetonitrile (20 ml) was added to a strongly stirred solution of *N*-(ferrocenylmethylidene)aniline (582 mg, 2 mmol) and 37% aqueous formaldehyde (2 ml, 25 mmol) in acetonitrile (30 ml). The reaction mixture was stirred for 30 min. Glacial acetic acid was then added dropwise until the solution was neutral. Stirring was continued for a further 4 h. During this period, glacial acetic acid was added occasionally to maintain the pH at nearly neutral. The reaction was monitored by thin-layer chromatography to establish when it was complete. The reaction was quenched by pouring it into diethyl ether (80 ml) and the organic layer was washed with 1 *M* KOH and saturated aqueous sodium chloride (50 ml). The diethyl ether solution was dried with K_2CO_3 and the solvent was removed *in vacuo*. The organic solid was purified by silica-gel column chromatography using ethyl acetate–hexane (3:1) giving a yield of 78%. A yellow single-crystal suitable for X-ray analysis was obtained by slow evaporation of a dichloromethane and petroleum solution at room temperature over a period of a week. Analysis calculated for $C_{18}H_{19}FeN$: C 70.84, H 6.27, N 4.59%. found: C 63.65, H 5.34, N 4.12%.

Crystal data

$[Fe(C_5H_5)(C_{13}H_{14}N)]$
$M_r = 305.19$
Monoclinic, $P2_1/c$
$a = 9.626$ (2) Å
$b = 11.675$ (2) Å
$c = 14.062$ (3) Å
$\beta = 104.762$ (4)°
$V = 1528.1$ (5) Å^3
$Z = 4$
$D_x = 1.327$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2691 reflections
$\theta = 2.2$–24.4°
$\mu = 0.98$ mm^{-1}
$T = 294$ K
Block, yellow
0.34 × 0.30 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.704$, $T_{\max} = 0.807$
8371 measured reflections
3089 independent reflections
2166 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.3°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 13$
$l = -10 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.100$
$S = 1.05$
3089 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.4801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C13	1.388 (4)	C13—C14	1.401 (4)
N1—C12	1.451 (4)	C14—C15	1.371 (5)
N1—C11	1.459 (4)	C15—C16	1.367 (5)
C10—C11	1.516 (4)	C16—C17	1.370 (5)
C13—C18	1.401 (4)	C17—C18	1.378 (5)
C13—N1—C12	119.7 (3)	C6—C10—C9	106.7 (3)
C13—N1—C11	120.4 (3)	C6—C10—C11	126.5 (3)
C12—N1—C11	113.6 (3)	C9—C10—C11	126.8 (3)
C2—C1—C5	107.1 (4)	N1—C11—C10	113.4 (3)
C3—C2—C1	109.4 (3)	N1—C13—C18	122.3 (3)
C4—C3—C2	107.9 (4)	N1—C13—C14	121.0 (3)
C3—C4—C5	109.1 (4)	C18—C13—C14	116.6 (3)
C4—C5—C1	106.5 (3)	C15—C14—C13	120.9 (3)
C7—C6—C10	109.0 (3)	C16—C15—C14	121.9 (4)
C8—C7—C6	108.0 (3)	C15—C16—C17	118.1 (4)
C7—C8—C9	108.1 (3)	C16—C17—C18	121.5 (4)
C8—C9—C10	108.2 (3)	C17—C18—C13	120.9 (3)
C13—N1—C11—C10	−86.0 (4)	C11—N1—C13—C18	−29.5 (5)
C12—N1—C11—C10	66.1 (4)	C12—N1—C13—C14	4.0 (5)
C6—C10—C11—N1	71.4 (4)	C11—N1—C13—C14	154.5 (3)
C9—C10—C11—N1	−107.5 (3)	N1—C13—C14—C15	173.9 (3)
C12—N1—C13—C18	−180.0 (3)	N1—C13—C18—C17	−174.6 (3)

All H atoms were located in a difference Fourier map. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but each group was allowed to rotate freely about its 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 0.93–0.97 Å and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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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