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3-Chloro-*N*-ferrocenylmethyl-*N*-methylaniline

Ying-Jie Li,[a] Hong-Xing Wang[a,b]* and Hong-Fei Wu[a]

[a]Department of Chemistry, College of Sciences, Tianjin University, Tianjin 300072, People's Republic of China, and [b]State Key Laboratory of Elemento-organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: liwenxuemei@hotmail.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.072
Data-to-parameter ratio = 13.8

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

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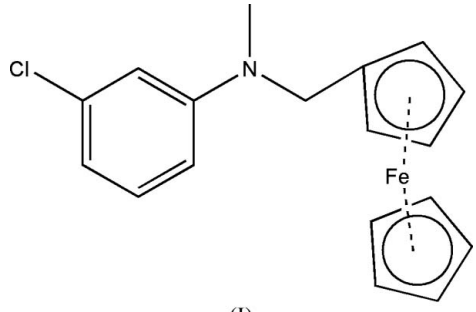
The title tertiary arylamine, $[Fe(C_5H_5)(C_{13}H_{13}ClN)]$, incorporating a ferrocenyl group, was synthesized by reductive methylation of the amine. The cyclopentadienyl plane is nearly perpendicular to the $C_{methylene}-N-C_{methyl}$ plane [dihedral angle = 96.8 (3)°]

Comment

Ferrocene-containing compounds have been widely studied owing to their potential application in catalysis, materials science, molecular devices and hydrometallurgy (Beer *et al.*, 1997). In addition, the chemistry of cyclometallated compounds, especially those bearing *N*-donor ligands, is one of the advanced areas of organometallic chemistry. As part of our ongoing investigation of the cyclometallation of tertiary amines, the title compound, (I), has recently been prepared in our laboratory. We present here the crystal structure of (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The N1—C13 bond distance is significantly shorter then the other two N—C bonds in (I) (Table 1). Methylene atom C11 is coplanar with the benzene plane [deviation = 0.0163 (4) Å], whereas the methyl atom C12 is out of the benzene plane by

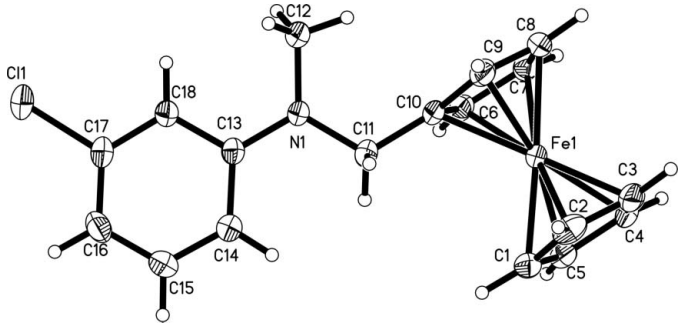


Figure 1
The structure of (I) with 35% probability displacement ellipsoids (arbitrary spheres for H atoms).

0.1969 (3) Å. The C10-containing cyclopentadienyl plane is nearly perpendicular to the C11/C12/N11 plane [dihedral angle = 96.8 (3)°] to minimize the repulsion between the cyclopentadienyl and the neighbouring methyl group.

Experimental

An acetonitrile solution (15 ml) of sodium cyanoborohydride (0.19 g, 3 mmol) was mixed with an acetonitrile solution (30 ml) of 3-chloro-*N*-(ferrocenylmethylene)aniline (0.65 g, 2 mmol) and 37% aqueous formaldehyde (2 ml, 25 mmol). The reaction mixture was stirred for 30 min at room temperature, and then glacial acetic acid was added dropwise until the pH was 7. The neutral solution was stirred for a further 1 h and then poured into diethyl ether (80 ml). The ether layer was washed with 1 *M* KOH aqueous solution and saturated NaCl aqueous solution (50 ml). The ether solution was dried with K_2CO_3 and the solvent was removed *in vacuo*. The organic oil was purified by silica-gel column chromatography using 3:1 ethyl acetate–hexane as eluant (yield 76%). Yellow single crystals of (I) were obtained from a dichloromethane and petroleum ether mixture after a week. Elemental analysis calculated for $C_{18}H_{18}ClFeN$: C 63.65, H 5.34, N 4.12%; found: C 63.39, H 5.71, N 4.36%.

Crystal data

$[Fe(C_5H_5)(C_{13}H_{13}ClN)]$	$Z = 2$
$M_r = 339.63$	$D_x = 1.496$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.243$ (2) Å	Cell parameters from 3182 reflections
$b = 10.250$ (3) Å	$\theta = 2.2$–$27.7°$
$c = 10.655$ (3) Å	$\mu = 1.17$ mm^{-1}
$\alpha = 116.643$ (3)°	$T = 293$ (2) K
$\beta = 92.658$ (3)°	Block, yellow
$\gamma = 106.771$ (3)°	0.38 × 0.30 × 0.22 mm
$V = 754.0$ (4) Å^3	

Data collection

Bruker APEX-II CCD area-detector diffractometer	2629 independent reflections
φ and ω scans	2421 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.012$
$T_{min} = 0.501$, $T_{max} = 0.773$	$\theta_{max} = 25.0°$
4102 measured reflections	$h = -9 \rightarrow 9$
	$k = -10 \rightarrow 12$
	$l = -12 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.2296P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.026$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.072$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.35$ e Å^{-3}
2629 reflections	
191 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C11	1.462 (2)	N1—C13	1.381 (2)
N1—C12	1.448 (2)		
C11—N1—C12	118.18 (15)	C12—N1—C13	119.61 (15)
C11—N1—C13	119.21 (15)		

Methyl H atoms were initially placed in calculated positions, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 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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References

Beer, P. D., Szemes, F., Balzani, V., Sala, C. M., Drew, M. G. B., Dent, S. W. & Maestri, M. (1997). *J. Am. Chem. Soc.* **119**, 11864–11875.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*, University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.