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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
 R factor = 0.047
 wR factor = 0.147
Data-to-parameter ratio = 14.5

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

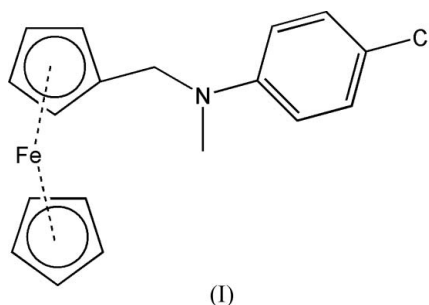
{[*N*-(4-Chlorophenyl)-*N*-methylamino]methyl}-ferrocene

In the title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{13}\text{H}_{13}\text{ClN})]$, the dihedral angle between the substituted cyclopentadienyl ring and the plane of the chlorophenylamino group is $77.4(2)^\circ$.

Received 18 May 2006
Accepted 3 June 2006

Comment

As a part of our ongoing project on the synthesis and structural characterization of tertiary ferrocenylamines [Wang, Li & Hou, 2005; Wang, Li, Wu *et al.*, 2005], we report here the structure of the title compound, (I) (Fig. 1). In (I), the ferrocenyl unit adopts a *cis* conformation with respect to the phenyl ring. The chlorophenyl plane, which also contains the amino N1 atom, with a mean deviation of $0.012(8)$ Å, makes an angle of $77.4(2)^\circ$ with the substituted cyclopentadienyl ring (Table 1). The planar cyclopentadienyl rings of the ferrocenyl unit are nearly parallel to each other.



Experimental

Sodium cyanoborohydride (0.95 g, 15 mmol) was added to a stirred solution of *N*-(*p*-chlorophenyl)aminomethylferrocene (1.628 g, 5 mmol) and 37% aqueous formaldehyde (4 ml, 50 mmol) in aceto-

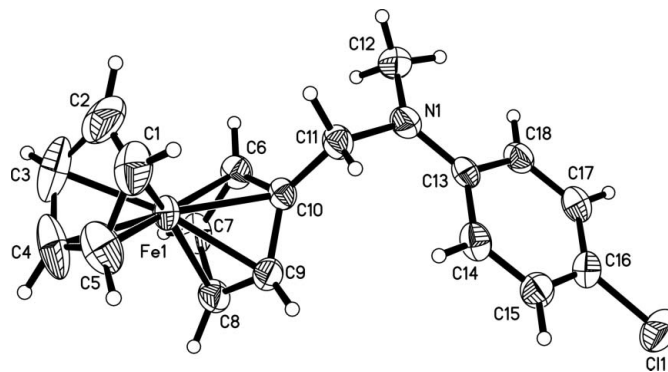


Figure 1
View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by spheres of arbitrary size.

nitrile (30 ml). A dark residue separated. The reaction mixture was stirred for 40 min, and glacial acetic acid was then 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 (60 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 73%). 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 one week. Analysis calculated for $C_{18}H_{18}ClFeN$: C 63.65, H 5.34, N 4.12%; found: C 63.61, H 5.57, N 4.33%.

Crystal data

$[Fe(C_5H_5)(C_{13}H_{13}ClN)]$	$Z = 8$
$M_r = 339.63$	$D_x = 1.444 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 10.093 (2) \text{ \AA}$	$\mu = 1.13 \text{ mm}^{-1}$
$b = 8.8080 (19) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 35.159 (8) \text{ \AA}$	Block, yellow
$V = 3125.5 (12) \text{ \AA}^3$	$0.20 \times 0.18 \times 0.12 \text{ mm}$

Data collection

Bruker CCD area-detector diffractometer	14959 measured reflections
φ and ω scans	2752 independent reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	1756 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.709$, $T_{\max} = 0.901$	$R_{\text{int}} = 0.050$
	$\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 2.6017P]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.147$	$(\Delta/\sigma)_{\max} = 0.002$
$S = 1.11$	$\Delta\rho_{\max} = 0.37 \text{ e \AA}^{-3}$
2752 reflections	$\Delta\rho_{\min} = -0.61 \text{ e \AA}^{-3}$
190 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—C13	1.381 (6)	N1—C11	1.460 (5)
N1—C12	1.452 (6)		
C13—N1—C12	118.6 (4)	N1—C11—C10	113.6 (3)
C13—N1—C11	121.8 (4)		
C12—N1—C13—C14	-171.0 (4)	C11—N1—C13—C14	-11.2 (6)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, 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 0.93–0.98 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; 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*.

We are indebted to the Natural Science Foundation of Tianjin City, People's Republic of China, for financial support (grant No. 033609011).

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