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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.027
 wR factor = 0.078
Data-to-parameter ratio = 12.0

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

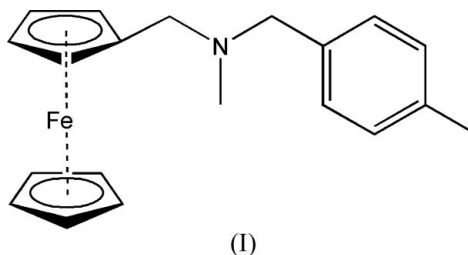
1-[*N*-Methyl-*N*-(4-methylbenzyl)amino-methyl]ferrocene

The title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{15}\text{H}_{18}\text{N})]$, has been synthesized as a precursor of ferrocenylaminophosphine and its crystal structure determined. The substituted cyclopentadienyl plane is nearly perpendicular to the plane of the benzene ring [dihedral angle = $84.9(2)^\circ$].

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Comment

Cyclometallated compounds are widely used in organic synthesis, such as the Heck and Suzuki reactions (Dupont *et al.* 2005). As a part of our ongoing investigations of cyclometallation and its applications in the asymmetric catalysis of ferrocenylamines (Wang *et al.*, 2005), a new compound, (I), has been prepared and we report its crystal structure here.



The deviation of atom N1 from the C11/C12/C13 plane is 0.4580 (1) Å. Methylene atom C11 is coplanar with the benzene plane [deviation 0.0204 (3) Å], whereas methyl atom C12 lies out of the benzene plane by 1.8453 (2) Å. The substituted cyclopentadienyl plane is nearly perpendicular to the plane of the benzene ring [dihedral angle $84.9(2)^\circ$].

Experimental

To a vigorously stirred solution of *N*-(4-methylbenzyl) aminomethylferrocene (319 mg, 1 mmol) and 37% aqueous formaldehyde (1 ml, 12.5 mmol) in acetonitrile (20 ml) was added sodium cyanoborohydride (190 mg, 3 mmol). After 30 min, glacial acetic acid was added dropwise until the pH was 7. The mixture was stirred at room temperature for 6 h. The solvent was removed and 2 *N* sodium hydroxide (20 ml) was then added to the residue. The mixture was extracted with diethyl ether. The organic layer was dried over anhydrous sodium sulfate and the solvent was removed to give the crude product, which was purified by column chromatography (silica gel, ethyl acetate–hexane 1:5) to afford (I) (yield 70%). Red single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane–petroleum ether (333–363 K) solution (3:1) at room temperature over a period of a week. Analysis, calculated for $\text{C}_{20}\text{H}_{23}\text{FeN}$: C 72.08, H 6.96, N 4.20%; found: C 72.11, H 6.71, N 4.36%.

Crystal data

[Fe(C₅H₅)(C₁₅H₁₈N)]
M_r = 333.24
 Monoclinic, *P*2₁/*n*
a = 14.8570 (14) Å
b = 5.9690 (5) Å
c = 19.3667 (18) Å
 β = 102.5300 (10)°
V = 1676.6 (3) Å³
Z = 4

D_x = 1.320 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3999
 reflections
 θ = 2.2–27.6°
 μ = 0.90 mm⁻¹
T = 293 (2) K
 Block, red
 0.38 × 0.26 × 0.20 mm

Data collection

Bruker APEX-II CCD area-
 detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
T_{min} = 0.635, *T_{max}* = 0.836
 8678 measured reflections

2950 independent reflections
 2436 reflections with *I* > 2σ(*I*)
R_{int} = 0.017
 θ_{max} = 25.0°
h = -17 → 14
k = -7 → 5
l = -21 → 23

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.027
wR(*F*²) = 0.078
S = 1.06
 2950 reflections
 246 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.3349P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 $\Delta\rho_{\text{max}}$ = 0.18 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.31 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Fe1—C1	2.0334 (17)	N1—C11	1.462 (2)
N1—C13	1.453 (2)	C1—C2	1.413 (2)
N1—C12	1.460 (2)	C13—C14	1.509 (2)
C13—N1—C12	110.91 (14)	N1—C11—C1	112.68 (14)
C13—N1—C11	111.59 (13)	N1—C13—C14	114.25 (14)
C12—N1—C11	109.34 (14)		
C12—N1—C11—C1	177.47 (15)	C11—N1—C13—C14	166.52 (14)
C12—N1—C13—C14	-71.31 (19)		

The C6-containing cyclopentadienyl ring is disordered over two positions; one position is eclipsed and the site-occupancy factor is 0.75, while the other position is *gauche* and the site-occupancy factor is 0.25 (torsion angle = 36°). The disordered ferrocenyl ring was refined as a regular pentagon. All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained

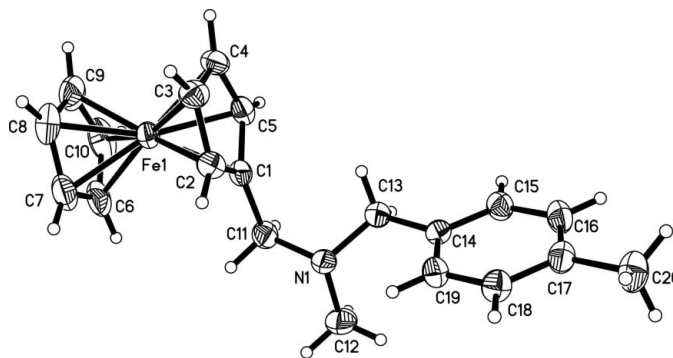


Figure 1

The molecular structure of (I), shown with 35% probability displacement ellipsoids (arbitrary spheres for H atoms). The minor component of the disordered ferrocenyl ring has been omitted for clarity.

to an ideal geometry with C—H distances of 0.96 Å 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 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); 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, 1998); software used to prepare material for publication: *SHELXTL*.

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