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1-[(*N*-Benzyl-*N*-methylamino)methyl]ferrocene

Hong-Fei Wu, Hong-Xing Wang,* Xiao-Li Yang, Li Wan and Nan Ma

Department of Chemistry, College of Sciences, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: hongxing_wang@hotmail.com

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Key indicators

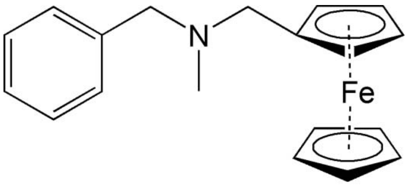
Single-crystal X-ray study
T = 294 K
Mean *σ*(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.032
wR factor = 0.082
Data-to-parameter ratio = 13.9

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)($C_{14}H_{16}N$)], has been synthesized to act as a precursor to ferrocenylaminophosphines. The dihedral angle between the substituted cyclopentadienyl plane and the plane of the phenyl ring is 65.0 (2)°.

Comment

Tertiary amines are one type of important nitrogen-containing ligands in organic synthesis (Gruter *et al.*, 1995). Their cyclometallated compounds are widely used in the Heck reaction, the Suzuki reaction and so on (Dupont *et al.*, 2005). As a part of our ongoing investigations on cyclometallation and applications in asymmetric catalysis of ferrocenylamines (Wang *et al.*, 2006), the new compound (I) has been prepared and we report here its crystal structure.



(I)

The deviation of atom N1 from the C11/C12/C13 plane is 0.2505 (2) Å. Methylene atom C11 is coplanar with the phenyl plane [deviation = 0.0797 (3) Å], whereas the methyl atom C12 lies out of the phenyl plane by 1.939 (5) Å. The dihedral angle between the substituted cyclopentadienyl plane and the plane of the phenyl ring is 65.0 (2)°. The two pentagonal rings of the ferrocenyl group are planar and nearly parallel [the interplanar angle is 1.8 (1)°].

Experimental

To a vigorously stirred solution of *N*-benzylaminomethylferrocene (305 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 1 h, glacial acetic acid was added dropwise until the pH was 7. The mixture was stirred at room temperature for 5 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 evaporated to give a crude product, which was purified by column chromatography (silica gel, ethyl acetate–hexane 1:5) to afford (I) (yield 66%). Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane–petroleum ether (333–363 K) solution (1:1) at room temperature over a period of two weeks. Analysis calculated for $C_{19}H_{21}FeN$: C 71.49, H 6.63, N 4.39%; found: C 71.51, H 6.72, N 4.26%.

Crystal data

[$Fe(C_5H_5)(C_{14}H_{16}N)$]
$M_r = 319.22$
Triclinic, $P\overline{1}$
$a = 5.9691$ (13) Å
$b = 10.245$ (2) Å
$c = 13.717$ (3) Å
$\alpha = 93.916$ (3)°
$\beta = 99.948$ (3)°
$\gamma = 105.071$ (3)°
$V = 792.2$ (3) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.95$ mm^{-1}
$T = 294$ (2) K
0.26 × 0.24 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.791$, $T_{\max} = 0.848$
4475 measured reflections
3191 independent reflections
2763 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.082$
$S = 1.07$
3191 reflections
230 parameters
30 restraints
H-atom parameters constrained
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Atoms C13–C19 and their attached H atoms are disordered over two positions; site-occupancy factors were refined and converged to 0.673 (5) and 0.327 (5), respectively. The phenyl ring was constrained to be a regular hexagon. 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 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, 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 $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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

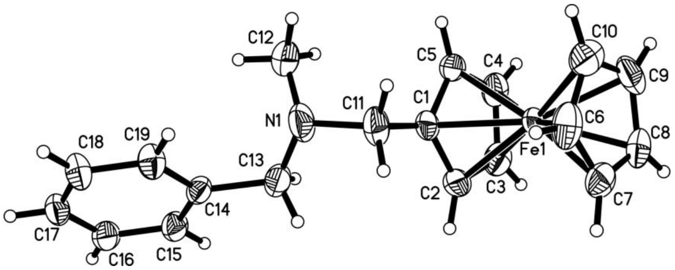


Figure 1
The molecular structure of (I), shown with 35% probability displacement ellipsoids (arbitrary spheres for H atoms). The minor disorder component has been omitted for clarity.

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