Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

[*N*-Methyl-*N*-(3-nitrophenyl)aminomethyl]-ferrocene

Hong-Xing Wang,[a,b]* Ying-Jie Li,[a] Hong-Fei Wu,[a] Hui-Chao Zhou,[a] Ren-Qing Gao[a] and Feng-Ying Geng[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:
hongxing_wang@hotmail.com

Received 23 August 2005
Accepted 25 August 2005
Online 31 August 2005

Key indicators

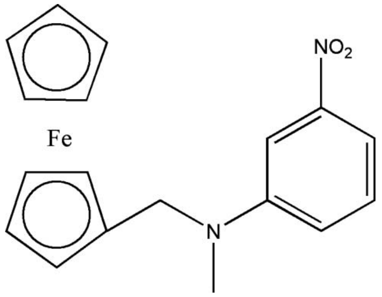
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 13.1

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)($C_{13}H_{13}N_2O_2$)], a tertiary ferrocenylamine, was synthesized by reductive methylation of the corresponding secondary amine. The substituted cyclopentadienyl ring is nearly perpendicular to the plane of the benzene ring [dihedral angle = 87.8 (3)°].

Comment

Ferrocene-containing compounds have been widely studied because of their potential application in catalysis, in materials science, in hydrometallurgy and as molecular devices (Beer *et al.*, 1997). As a part of our ongoing investigations on cyclometallation 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 (C7) and the methylene C atom (C8) are displaced from the plane of the benzene ring by 0.1379 (2) and 0.4934 (1) Å, respectively. The substituted cyclopentadienyl ring is nearly perpendicular to the plane of the benzene ring [dihedral angle = 87.8 (3)°].

Experimental

To a vigorously stirred solution of *N*-(3-nitrophenyl)amnioferrocene (700 mg, 2 mmol) and 37% aqueous formaldehyde (2 ml, 25 mmol) in acetonitrile (30 ml), a solution of sodium cyanoborohydride (378 mg, 6 mmol) in acetonitrile (20 ml) was added. 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 neutral pH. The reaction was monitored by thin-layer chromatography in order to find out when it was complete. The reaction was quenched by pouring the mixture 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, giving a yield of 83%. Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane–petroleum ether solution (3:1 *v*/*v*) at room temperature over a period of one week. Analysis calculated for $C_{18}H_{18}FeN_2O_2$: C 61.74, H 5.18, N 8.00%; found: C 61.69, H 5.31, N 8.28%.

Crystal data

$[Fe(C_5H_5)(C_{13}H_{13}N_2O_2)]$
$M_r = 350.19$
Triclinic, $P\overline{1}$
$a = 8.2896\ (14)$ Å
$b = 9.7588\ (16)$ Å
$c = 10.5669\ (17)$ Å
$\alpha = 90.153\ (3)^\circ$
$\beta = 109.629\ (2)^\circ$
$\gamma = 101.455\ (2)^\circ$
$V = 786.9\ (2)$ Å^3
$Z = 2$
$D_x = 1.478$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2812 reflections
$\theta = 2.7$–26.5°
$\mu = 0.97$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
$0.28 \times 0.20 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.719$, $T_{max} = 0.890$
4000 measured reflections
2751 independent reflections
2491 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0^\circ$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 11$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.03$
2751 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.1823P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.064 (3)

Table 1
Selected geometric parameters (Å, °).

N2—C1	1.395 (3)	C1—C2	1.398 (3)
N2—C7	1.457 (3)	C1—C6	1.410 (3)
N2—C8	1.474 (2)	C8—C9	1.504 (3)
C1—N2—C7	118.00 (17)	N2—C1—C6	121.23 (19)
C1—N2—C8	119.43 (15)	N2—C8—C9	112.40 (15)
C7—N2—C8	113.76 (16)	C13—C9—C8	126.17 (17)
N2—C1—C2	121.90 (17)	C10—C9—C8	126.84 (17)
C7—N2—C1—C2	−172.91 (17)	C1—N2—C8—C9	−78.8 (2)
C8—N2—C1—C2	−27.5 (3)	C7—N2—C8—C9	68.1 (2)
C7—N2—C1—C6	10.1 (3)	N2—C8—C9—C13	76.8 (2)
C8—N2—C1—C6	155.54 (18)	N2—C8—C9—C10	−99.0 (2)
N2—C1—C2—C3	−178.38 (16)	N2—C8—C9—Fe1	168.48 (13)
N2—C1—C6—C5	177.3 (2)	C8—C9—C13—C12	−176.50 (17)

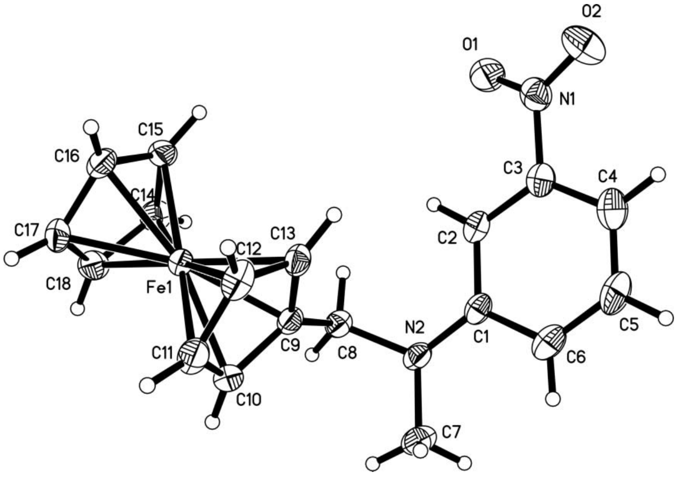


Figure 1
View of the molecule of (I). showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

All H atoms were located in a difference Fourier map but 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 $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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 (project No. 033609011).

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. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

m1872 Wang *et al.* • $[Fe(C_5H_5)(C_{13}H_{13}N_2O_2)]$ *Acta Cryst.* (2005). E**61**, m1871–m1872