[4](1)(2,3-Diazabuta-1,3-diene)ferrocenophane

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean σ(C–C) = 0.007 Å

R factor = 0.059

wR factor = 0.136

Data-to-parameter ratio = 17.4

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

The molecular structure of the title compound, [Fe$_2$(C$_5$H$_5$)$_2$(C$_{12}$H$_{10}$N$_2$)], is centrosymmetric, with bond parameters similar to its analogue [4](1)(1,4-dimethyl-2,3-diazabuta-1,3-diene)-ferrocenophane. The cyclopentadienyl rings of the ferrocene moieties are eclipsed and their average dihedral angle with the Schiff base C=N—N=C fragment is 8.5 (4)°.

Comment

Ferrocenophane derivatives are interesting compounds, both from the point of view electrochemical properties and also for their applications, such as molecular wires (Heo & Lee, 1999). The molecular structure of the title compound, (I), is similar to its [4](1)(1,4-dimethyl-2,3-diazabuta-1,3-diene)ferroceno-phane analogue, (II) (Osborne *et al.*, 1996). The molecule is centrosymmetric about the mid-point of the N—N bond and the cyclopentadienyl rings of both ferrocene moieties are eclipsed. The bond lengths and angles are in the normal ranges for ferrocene compounds (Roberts *et al.*, 1988) and in agreement with those in (II) (Osborne *et al.*, 1996), although the N1—N1^i bond length of 1.436 (7) Å (see Table 1 for symmetry code) is slightly longer than that in (II) [1.406 (6) Å]. The C11=N1—N1^i angle of 110.4 (5)° is smaller than that in (II) [113.5 (4)°]. The C11=N1—N1^i=C11^i moiety is planar and makes an average dihedral angle of 8.5 (4)° with the cyclopentadienyl rings.

(I)
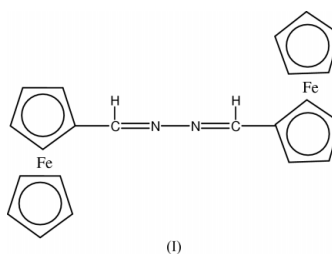
Experimental

Ferrocenecarboxaldehyde (0.5 g, 0.0022 mol) was added to 10 ml methanol and stirred at room temperature until completely dissolved. A stoichiometric amount of hydrazine (0.11 g, 0.0022 mol) and 0.5 ml of glacial acetic acid were then poured into the solution in a two-neck round-bottomed flask. The reaction mixture was refluxed for 1 h. After this period, the reaction mixture was filtered and the filtrate was allowed to cool to room temperature. Slow evaporation of the solvent produced a precipitate, which were filtered off and washed with cold methanol and air-dried. Recrystallization from dichloro-methane and hexane produced dark orange crystals of (I) suitable for X-ray analysis.

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Crystal data

$[Fe_2(C_5H_5)_2(C_{12}H_{10}N_2)]$
$M_r = 424.10$
Monoclinic, $P2_1/c$
$a = 7.5844\,(11)$ Å
$b = 10.0804\,(14)$ Å
$c = 12.1698\,(16)$ Å
$\beta = 105.726\,(3)°$
$V = 895.6\,(2)$ Å^3
$Z = 2$

$D_x = 1.573$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 812
 reflections
$\theta = 2.7–27.5°$
$\mu = 1.63$ mm^{-1}
$T = 293\,(2)$ K
Slab, dark orange
$0.24 \times 0.16 \times 0.02$ mm

Data collection

Bruker SMART APEX CCD area-
 detector diffractometer
ω scans
Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{min} = 0.696$, $T_{max} = 0.968$
5939 measured reflections

2066 independent reflections
1439 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 12$
$l = -15 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.136$
$S = 1.07$
2066 reflections
119 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C11	1.271 (6)	C1—C11	1.451 (6)
N1—N1^i	1.436 (7)		
C11—N1—N1^i	110.4 (5)	N1—C11—C1	122.0 (4)

Symmetry code: (i) $1 - x, -y, 2 - z$.

After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms, with C—H = 0.97 Å and N—H = 0.89 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); molecular graphics:
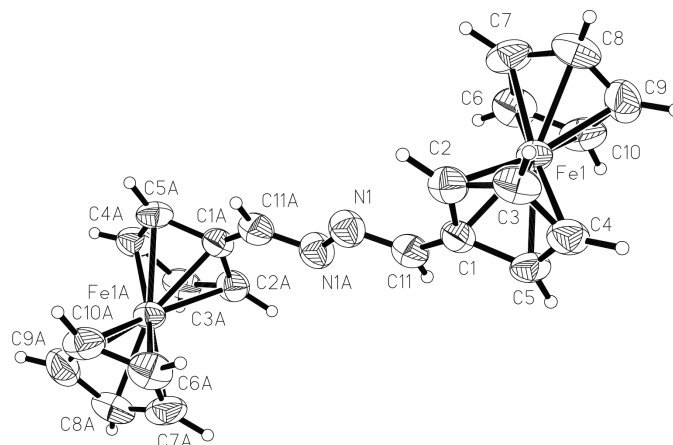

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids for non-H atoms.

SHELXTL; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

References

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