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[(*N*,*N*-Dimethylamino)methylferrocenyl]-methyl(*N*-methylanilino)phenylsilane

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Key indicators

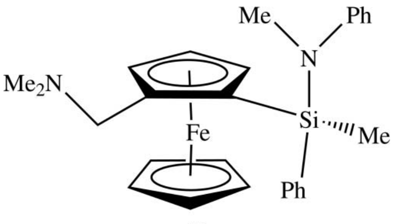
Single-crystal X-ray study
T = 93 K
Mean *σ*(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.030
wR factor = 0.087
Data-to-parameter ratio = 18.9

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

The title compound, $[Fe(C_5H_5)(C_{22}H_{27}N_2Si)]$, is a diastereomeric *N*,*N*-dimethylaminomethylferrocenylsilane having chirality at the ferrocenyl unit and at the Si atom. The compound crystallizes in a centrosymmetric space group containing two out of the possible four isomers.

Comment

Much attention has been focused on the chemistry of *N*,*N*-dimethylaminomethylferrocenes, mainly for the preparation of heterobimetallic complexes (Jacob & Edelmann, 1998). Only a few *N*,*N*-dimethylaminomethylferrocenylsilanes have been structurally characterized so far (Beyer *et al.* 2002; Jacob *et al.*, 2004; Lorenz *et al.*, 2002; Palitzsch *et al.*, 1998, 1999), none of them having chirality at the Si atom.



(I)

In this paper, we describe the structure of the title compound, (I), which was prepared by reaction of lithium *N*-methylanilide with chloro(*N*,*N*-dimethylaminomethylferrocenyl)methylphenylsilane in 1,2-dimethoxyethane.

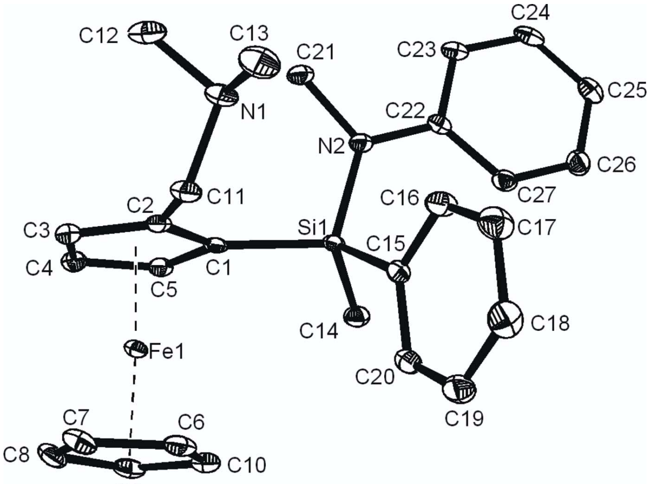


Figure 1
The molecular structure of (I), drawn with 50% probability displacement ellipsoids. H atoms and the minor disorder component have been omitted for clarity.

Some basic features of the ferrocenyl group should be mentioned. The cyclopentadienyl rings of the ferrocene unit have a slightly staggered conformation, as can be seen from the torsion angle C1—Cg1—Cg2—C10 of −20.1° (Cg1 and Cg2 are the centroids of the C1–C5 and C6–C10 rings, respectively). The distances from the Fe atom to Cg1 and Cg2 are 1.649 and 1.651 Å, respectively. The cyclopentadienyl rings are nearly coplanar, forming a dihedral angle of 4.09 (8)°. Atom N1 of the dimethylaminomethyl group is not bonded to the Si atom; the N1···Si1 distance is 3.831 (1) Å. Atom Si1 is tetrahedrally coordinated by the ferrocenyl group, a methyl group, a phenyl group and the *N*-methylanilide. Four different substituents at the Si atom make this atom chiral. Furthermore, chiral planarity is introduced into this molecule by the 1,2-substitution of the *N*,*N*-dimethylaminomethylferrocenyl group. The crystal under investigation showed a centrosymmetric space group containing two out of the possible four diastereomers.

Experimental

Preparative work was performed in Schlenk tubes under argon with dry and air-free solvents. Lithium *N*-methylanilide (1.3 g, 0.0116 mol) in 1,2-dimethoxyethane (40ml) was added slowly to a cooled solution (195 K) of chloro(*N*,*N*-dimethylaminomethylferrocenyl)methylphenylsilane (4.63 g, 0.0116 mol) in 1,2-dimethoxyethane (70 ml). The cooling bath was removed and the solution stirred for 70 h at room temperature. The solvent was removed *in vacuo* and the residue taken up in pentane (120 ml). The solution was filtered into a Schlenk tube and concentrated. Orange crystals (3.9 g, 72%) were obtained by cooling the solution for some weeks to 278 K. These crystals were used for the X-ray study without recrystallization.

Crystal data

$[\mathrm{Fe}(\mathrm{C_5H_5})(\mathrm{C_{22}H_{27}N_2Si})]$
$M_r = 468.49$
Monoclinic, $P2_1/c$
$a = 15.5905\ (11)$ Å
$b = 8.6684\ (6)$ Å
$c = 21.2931\ (11)$ Å
$\beta = 125.320\ (4)°$
$V = 2348.0\ (3)$ Å^3

$Z = 4$
$D_x = 1.325$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.71$ mm^{-1}
$T = 93\ (2)$ K
Prism, orange
0.33 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick 1996)
$T_{\min} = 0.800$, $T_{\max} = 0.907$

47177 measured reflections
5389 independent reflections
4601 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.040$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.087$
$S = 1.09$
5389 reflections
285 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.9734P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.95$ e Å^{-3}
$\Delta\rho_{\min} = -0.66$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—N2	1.756 (1)	Si1—C14	1.868 (2)
Si1—C1	1.857 (2)	Si1—C15	1.880 (2)
N2—Si1—C1	107.80 (6)	N2—Si1—C15	108.84 (6)
N2—Si1—C14	110.50 (7)	C1—Si1—C15	111.41 (6)
C1—Si1—C14	107.72 (7)	C14—Si1—C15	110.54 (7)

All H atoms were positioned geometrically, with C—H distances ranging from 0.95 to 0.99 Å and U_{iso}(H) values of $1.2U_{\text{eq}}$(C) or $1.5U_{\text{eq}}$(methyl C). The C14 methyl group is disordered over two positions, with site-occupation factors of 0.49 (2) and 0.51 (2). All methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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