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Dimethyl[1-(pentadienylidene)ethyl]amine

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Key indicators

Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.004 Å
R factor = 0.073
wR factor = 0.155
Data-to-parameter ratio = 14.8

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

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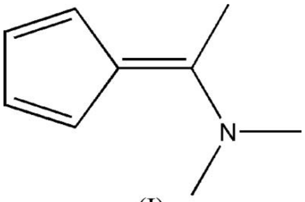
In the title molecule, $C_9H_{13}N$, all bond lengths and angles show normal values. In the crystal structure, weak C—H$\cdots\pi$ interactions link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by van der Waals forces.

Comment

Fulvenes have been extensively studied as catalysts for olefin polymerization (Wang *et al.*, 2005; Sabine *et al.*, 2003; Bai *et al.*,1999), and as important substrates in the synthesis of many polycyclic systems, natural products and anticancer drugs (Manikandan *et al.*, 2002; Sweeney *et al.*, 2005). In our work with 6,6-dimethylamino-6-methylfulvene, (I), first prepared in 1964 (Hafner *et al.*, 1964), we obtained a single crystal. Here we report the crystal structure of (I).



(I)

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987). Some elongation of the exocyclic C═C bond in (I) [1.412 (4) Å *versus* 1.386 Å observed in 6-(*N*,*N*-dimethylamino)pentafulvene] may be attributed to the concerted effect of electronic reorganization and steric interaction (Ammon & Wheeler, 1975). In the crystal structure, weak C—H$\cdots\pi$ interactions (Table 1; *Cg* is the centroid of C1–C5) link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by van der Waals forces.

Experimental

Dimethyl sulfate (4.8 ml, 6.4 g, 0.05 mol) was slowly added at 323–333 K to *N*,*N*-dimethylacetamide (4.7 ml, 4.4 g, 0.05 mol). After heating the reaction mixture for 2 h at 343–353 K, it was cooled to room temperature. The resulting reaction mixture was added at 263 K to a stirred solution of sodium cyclopentadienide (4.4 g, 0.05 mol) in 40 ml of THF. After the resulting sodium methyl sulfate was filtered off, the solvent was removed in vacuum and the brown–yellow residue was extracted with diethyl ether. After removal of the solvent, the yellow product, (I) was obtained in 68% yield. The product was dissolved in diethyl ether, and allowed to evaporate slowly at room temperature over several days to yield light-yellow crystals suitable for X-ray diffraction.

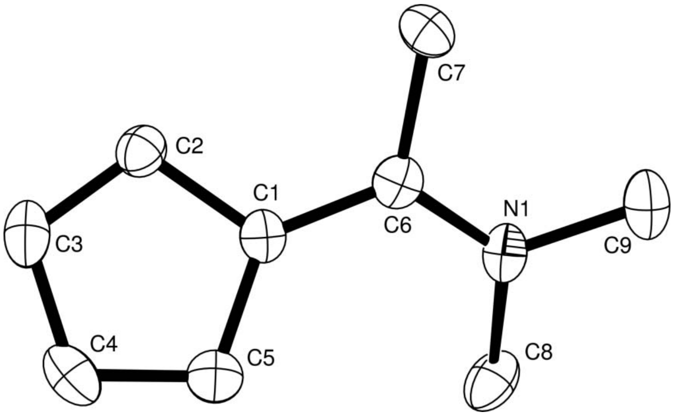


Figure 1
The molecular structure of (I), showing the atomic numbering and 30% probability displacement ellipsoids. H atoms are not shown.

Crystal data

$C_9H_{13}N$
$M_r = 135.20$
Monoclinic, $P2_1/c$
$a = 7.167$ (3) Å
$b = 9.505$ (4) Å
$c = 11.647$ (4) Å
$\beta = 90.423$ (5)°
$V = 793.4$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 213$ (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.980$, $T_{\max} = 0.987$
3179 measured reflections
1392 independent reflections
1191 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.155$
$S = 1.21$
1392 reflections
94 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···$Cg^{\rm i}$	0.96	2.68	3.615 (3)	166

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

All H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) values set to 1.2–1.5$U_{\rm eq}$(parent atom).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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