Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

4,4-Difluoro-8-methyl-4-bora-3a,4a-diaza-*s*-indacene

Dong-Chuan Wang, Hong-Ping Wang, Shang Gao, Tong-Yan Zhang and Xiao-Jun Peng*

State Key Laboratory of Fine Chemicals, Dalian University of Technology, 158 Zhongshan Rd, Dalian 116012, People's Republic of China

Correspondence e-mail: pengxj@dlut.edu.cn

Received 24 March 2007
Accepted 28 March 2007

Key indicators

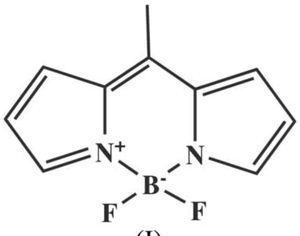
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.114
Data-to-parameter ratio = 12.4

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

In the title compound, $C_{10}H_9BF_2N_2$, a boron–dipyrromethene (BODIPY) dye, a network of C—H···F interactions help to establish the crystal packing. The asymmetric unit consists of two molecules.

Comment

As part of our studies of boron–dipyrromethene (BODIPY) dyes, which are well known fluorescence labels and biomolecular sensors (e.g. Trieflinger *et al.*, 2005; Dost *et al.*, 2006), we now report the synthesis and crystal structure of the title compound, (I) (Fig. 1).



(I)

There are two molecules in the asymmetric unit, with essentially identical geometries. The B—N bond lengths are indistinguishable in both molecules (Table 1), indicating the expected delocalization of the positive charge. The C and N atoms of the three fused heterocyclic rings in each molecule are almost planar, with r.m.s. deviations from the mean plane of 0.041 and 0.054 Å for the molecules including atoms B1 and B2, respectively. However, the B atoms are displaced from the mean planes, by 0.241 (2) Å for B1 and 0.291 (2) Å for B2.

A number of C—H···F interactions (Table 2) help to establish the crystal packing in (I).

Experimental

Pyrrole (8 mmol, 0.6 ml) was injected into a 500 ml flask with dichloromethane (DCM, 250 ml), then acetyl chloride (4 mmol, 0.4 ml) was added immediately. After being refluxed with stirring for 10 h under a nitrogen atmosphere, DCM (200 ml) was evaporated. Triethylamine (37 mmol, 4 ml) was then added to the brown residue, followed by boron trifluoride diethyl ether complex (37.4 mmol, 6 ml). After stirring for 3 h, the solution was washed with an aqueous solution of $NaHCO_3$, the organic layer was dried over $MgSO_4$, and the solvent was removed. Chromatography on a silica column was carried out and eluted with an ethyl acetate/petroleum ether (1:6 *v*/*v*) mixture. The collected red fraction was subsequently recrystallized from chloroform/hexane (1:8 *v*/*v*) to obtain the target compound in 33% yield. Red blocks of (I) were obtained from hexane–dichloromethane (1:1 *v*/*v*) solution by slow evaporation at room temperature after 35 d.

doi:10.1107/S1600536807015243

Crystal data

$C_{10}H_9BF_2N_2$
$M_r = 206.00$
Triclinic, $P\overline{1}$
$a = 7.7156$ (4) Å
$b = 7.7329$ (4) Å
$c = 16.6895$ (8) Å
$\alpha = 102.952$ (3)°
$\beta = 93.604$ (3)°
$\gamma = 92.362$ (4)°
$V = 966.94$ (8) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 298$ (2) K
$0.60 \times 0.55 \times 0.44$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.936$, $T_{\max} = 0.953$
7399 measured reflections
3372 independent reflections
2780 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.114$
$S = 1.08$
3372 reflections
272 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected bond lengths (Å).

B1—N1	1.538 (2)	B2—N3	1.541 (2)
B1—N2	1.532 (2)	B2—N4	1.547 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···F2$^{\text{i}}$	0.93	2.40	3.242 (2)	150
C5—H5A···F1$^{\text{ii}}$	0.93	2.50	3.417 (2)	170
C11—H11A···F1$^{\text{ii}}$	0.93	2.55	3.384 (2)	149
C6—H6C···F1$^{\text{iii}}$	0.96	2.48	3.315 (2)	146
C6—H6A···F2$^{\text{iv}}$	0.96	2.54	3.323 (2)	139
C17—H17A···F3$^{\text{v}}$	0.96	2.52	3.375 (2)	149
C17—H17C···F4$^{\text{vi}}$	0.96	2.50	3.343 (2)	147
C6—H6B···F2$^{\text{vii}}$	0.96	2.60	3.475 (2)	152

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x, -y, -z$; (iii) $-x, -y+1, -z$; (iv) $x, y+1, z$; (v) $x-1, y, z$; (vi) $-x+1, -y, -z+1$; (vii) $-x+1, -y+1, -z$.

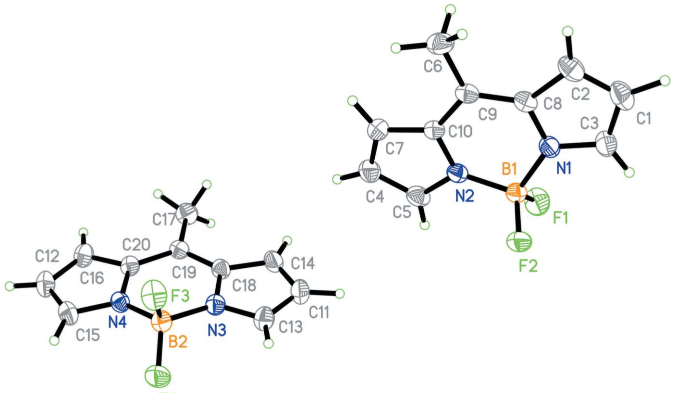


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level and H atoms shown as small spheres of arbitrary radius.

The H atoms were placed in geometrically calculated positions (C—H = 0.93–0.96 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}$(methyl C).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work was financially supported by the Education Ministry of China and the National Natural Science Foundation of China (project No. 20472012).

References

Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1997). *SMART* (Version 5.611), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Dost, Z., Atilgan, S. & Akkaya, E. U. (2006). *Tetrahedron*, **62**, 8484–8488.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Trieflinger, C., Rurack, K. & Daub, J. (2005). *Angew. Chem. Int. Ed.* **44**, 2288–2291.