organic papers

4,4-Difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

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The structure of the title compound (TM-BODIPY), $C_{13}H_{15}BF_2N_2$, which can be used to synthesize molecular probes for labelling DNA and proteins, has been determined using a very small single crystal employing Cu $K\alpha$ radiation and a two-dimensional multi-wire proportional detector. The molecules stack perpendicular to $(90\bar{4})$, forming a corrugated sheet-like structure when viewed along (3,0,10).

Comment

Since the discovery of BODIPY (BODIPY = dipyrrometheneboron difluoride) as fluorophores, TM-BODIPY (the title compound) derivatives have proved to be important structures because they can be used to synthesize molecular probes for labelling DNA (Ihara et al., 2002) and proteins (Karolin et al., 1994). Although the crystal structures of several of the substituted TM-BODIPY derivatives have been investigated, the structure of the title compound, TM-BODIPY, (I), has not been reported. In view of its importance, we were interested in studying its crystal structure.

(I)
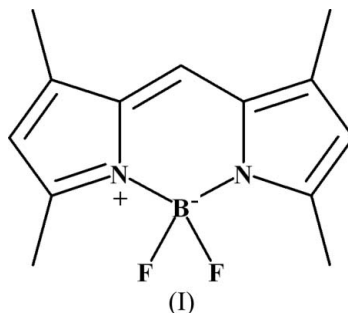
Compound (I) (Fig. 1) was obtained from the corresponding pyrroles by stepwise formation of aldehydes, Vilsmeier–Haack reaction (Silverstein *et al.*, 1956) followed by condensation with another equivalent of pyrrole (Treibs *et al.*, 1968; Vos de Wael *et al.*, 1977).

The structure of (I) is essentially planar (for non-H atoms), the average deviation being 0.0244 Å. This molecular plane intersects the plane formed by atoms B1, F1 and F2 at 89.8 (1)°. The molecules stack perpendicular to $(90\bar{4})$, forming a corrugated sheet-like structure when viewed along (3,0,10) (Fig. 2).

Experimental

The title compound was obtained by a literature method (Vos de Wael *et al.*, 1977). Small single crystals were obtained by slow evaporation of TM-BODIPY in diethyl ether.

Crystal data

C$_{13}$H$_{15}$BF$_2$N$_2$
$M_r = 248.08$
Monoclinic, $P2_1/n$
$a = 7.6932$ (13) Å
$b = 14.379$ (2) Å
$c = 11.5295$ (16) Å
$\beta = 107.245$ (8)°
$V = 1218.0$ (3) Å^3

$Z = 4$
$D_x = 1.353$ Mg m^{-3}
Cu $K\alpha$ radiation
$\mu = 0.84$ mm^{-1}
$T = 110$ (2) K
Block, red
$0.08 \times 0.03 \times 0.01$ mm

Data collection

Bruker GADDS D8 Discover
  diffractometer
φ and ω scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2004)
  $T_{min} = 0.936$, $T_{max} = 0.992$

7569 measured reflections
1736 independent reflections
1035 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$
$\theta_{max} = 59.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.119$
$S = 1.06$
1736 reflections
167 parameters
H-atom parameters constrained

$w = [\exp\{2(\sin\theta/\lambda)^2\}]/[\sigma^2(F_o^2) + (0.061P)^2]$
  where $P = 0.333F_o^2 + 0.667F_c^2$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

    All H atoms atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

    Data collection: *FRAMBO* (Bruker–Nonius, 2003); cell refinement: *Cell_Now* (Sheldrick, 2003) and *SAINT* (Bruker–Nonius, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2001); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2001); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenbourg, 2001); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek 2003) as incorporated in *WinGX* (Farrugia, 1999)'.

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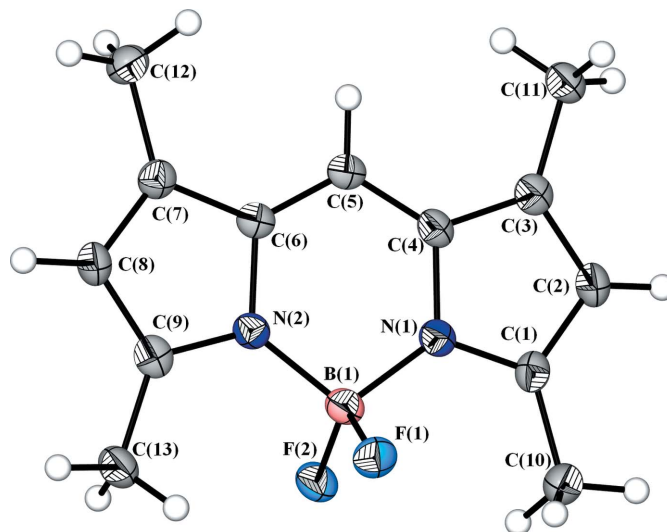
Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.
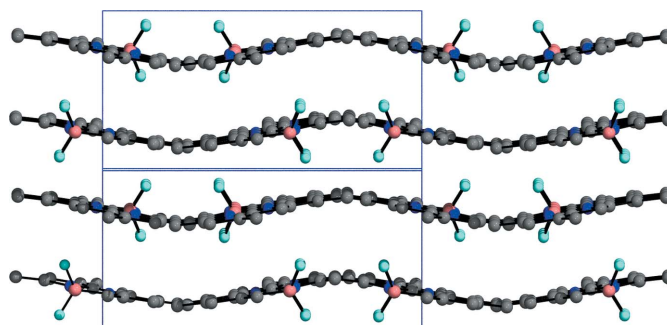
Figure 2
The crystal structure of (I), viewed along (3,0,10), showing the corrugated sheet-like packing in which molecules are held together by weak van der Waals interactions.

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