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4,4-Difluoro-3-[(4-methoxyphenyl)styryl]-1,5,7,8-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.132
Data-to-parameter ratio = 13.1

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

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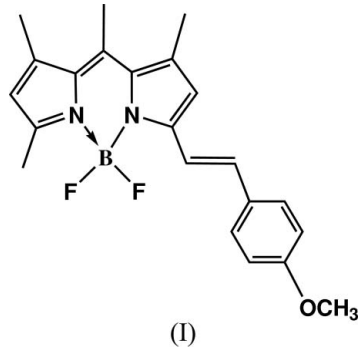
In the title compound, $C_{22}H_{23}BF_2N_2O$, the styrene and boron-dipyrromethene (BODIPY) fused-ring fragments are almost coplanar. Short C—C bond lengths suggest some electronic conjugation between the two systems.

Comment

Boron-dipyrromethene (BODIPY) dyes are well known and have attracted much interest in the design of fluorescence labels and biomolecular sensors (Bergström *et al.*, 2002; Trieflinger *et al.*, 2005; Dost *et al.*, 2006). Detailed information on their molecular and crystal structures is necessary to understand their photophysical and photochemical properties (Euler *et al.*, 2002). As part of our own work on BODIPY compounds, we report here the synthesis and crystal structure of the title compound, (I) (Fig. 1).



(I)

The main skeleton of the molecule, which is formed from three fused heterocyclic rings, is close to planar with an r.m.s. deviation of 0.006 Å. The maximum deviations from the mean plane are 0.011 (2) Å for N2 and 0.011 (3) Å for B. The styrene part of the molecule (C6 benzene ring plus C10 and C13) is close to being planar, with an r.m.s. deviation for the fitted atoms of 0.008 Å. The dihedral angle between the two fragments is 1.36 (6)°. This near coplanarity of the two delocalized systems, and the shortened C13—C20 [1.444 (2) Å] and C10—C17 [1.452 (2) Å] bond lengths in the bridging chain, suggests a significant degree of conjugation between the two fragments.

Experimental

Compound (I) was prepared from 4,4-difluoro-1,3,5,7,8-pentamethyl-4-bora-3a,4a-diaza-*s*-indacene (methyl-BODIPY) and *p*-methoxybenzaldehyde in chlorobenzene. Methyl-BODIPY (1 mmol) and *p*-methoxybenzaldehyde (2.5 mmol) were dissolved in chlorobenzene (15 ml) with absolute acetic acid (0.5 ml) and anhydrous piperidine

(0.6 ml) as catalysts under an argon atmosphere. After being stired for 24 h at 398 K, the cooled mixture was purified by silica column chromatography and elution with a dichloromethane/petroleum ether (1:3) mixture. The collected dark-blue fraction was subsequently recrystallized from chloroform/hexane (1:4) to obtain the target compound with a 23% yield. Suitable single crystals of (I) were obtained by recrystallization from a hexane/dichloromethane (1:2) solution at room temperature.

Crystal data

$C_{22}H_{23}BF_2N_2O$
M_r = 380.23
Triclinic, $P\overline{1}$
a = 8.6442 (2) Å
b = 11.0246 (3) Å
c = 12.0034 (3) Å
α = 64.565 (1)°
β = 73.311 (2)°
γ = 68.007 (1)°
V = 946.90 (4) Å^3
Z = 2
D_x = 1.334 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
Cube, dark-blue
0.60 × 0.60 × 0.60 mm

Data collection

Bruker SMART APEX-II CCD diffractometer
φ and ω scans
Absorption correction: none
11471 measured reflections
3314 independent reflections
2915 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.132
S = 1.07
3314 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0798P)^2 + 0.1878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

The H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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*.

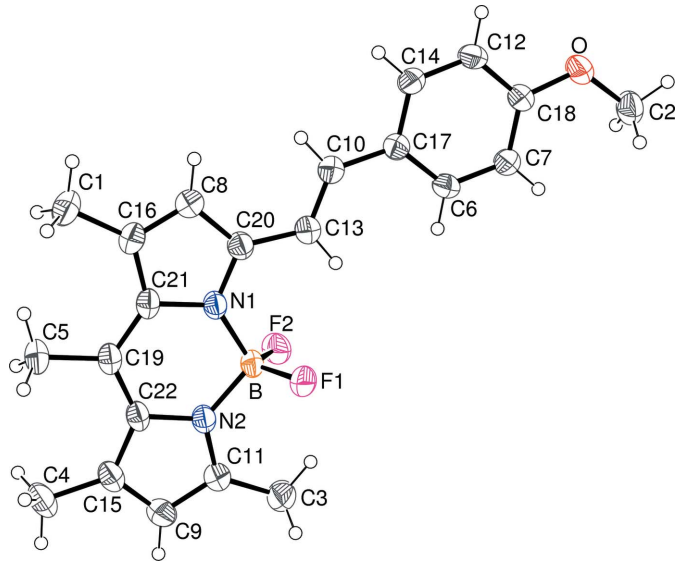


Figure 1
The molecular structure of (I), with 40% probability displacement ellipsoids (arbitrary spheres for the H atoms).

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