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# 8-[3,4-Bis(ethoxycarbonylmethoxy)-phenyl]-4,4-difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

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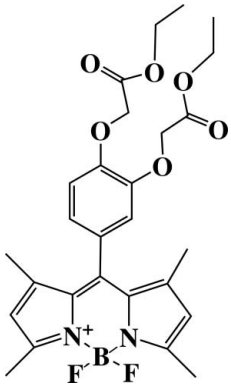
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.056; $wR$ factor = 0.152; data-to-parameter ratio = 13.6.

In the title compound, $C_{27}H_{31}BF_2N_2O_6$, the benzene ring is almost perpendicular to the fused-ring system [the dihedral angle is 82.3 (1)°]. The B—N distances in the central ring are almost identical, indicating delocalization of the formal positive charge over the two N atoms.

## Related literature

For background, see: Wu *et al.* (2005); Trieflinger *et al.* (2005); Dost *et al.* (2006); Qin *et al.* (2005); Yu *et al.* (2007).



## Experimental

### Crystal data

$C_{27}H_{31}BF_2N_2O_6$
$M_r$ = 528.35
Monoclinic, $P2_1/n$
$a$ = 9.6068 (6) Å
$b$ = 7.5418 (6) Å
$c$ = 36.896 (2) Å
$\beta$ = 93.231 (5)°
$V$ = 2669.0 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 298 (2) K
0.62 × 0.21 × 0.08 mm

### Data collection

Bruker SMART APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min}$ = 0.940, $T_{max}$ = 0.992
12387 measured reflections
4681 independent reflections
2283 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.079

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.152
$S$ = 0.93
4681 reflections
343 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| B—N1 | 1.533 (5) | B—N2 | 1.537 (5) |

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2001); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2443).

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# supplementary materials

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# 8-[3,4-Bis(ethoxycarbonylmethoxy)phenyl]-4,4-difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

**D.-C. Wang, Z.-Z. Hu, H.-J. Chi, Z.-Q. Zhang and X.-J. Peng**

## Comment

Boron-dipyrromethene (BODIPY) dyes are well known as fluorescence labels and biomolecule sensors (Wu *et al.*, 2005; Trieflinger *et al.*, 2005; Dost *et al.*, 2006). So As part of our ongoing studies of BOPIPY species (Qin *et al.*, 2005; Yu *et al.*, 2007) the structure of the title compound, (I), (Fig. 1) is now reported.

The BODIPY skeleton formed by the three conjugated heterocyclic rings is almost coplanar, with an r.m.s. deviation of 0.061 (3) Å; the maximum deviations from the mean plane are 0.113 (3) Å for B and 0.094 (3) Å for C4. Perhaps due to the presence of the C3 and C10 methyl groups, the benzene ring is almost perpendicular to the BODIPY mean plane, with the dihedral angle is 82.3 (1)°. The B—N distances in the central ring are almost identical, indicating delocaization of the formal positive charge on one of the N atoms (Table 1).

## Experimental

8 mmol (0.8 ml) of 2,4-dimethyl-pyrrole and 4 mmol (1240 mg) (2-ethoxycarbonylmethoxy-4-formyl-phenoxy)-acetic acid ethyl ester were dissolved in 350 ml absolute DCM and 0.15 ml trifluoroacetic acid was added immediately under an nitrogen atmosphere. After being stirred at room temperature for 8 h, 320 ml DCM was evaporated, a solution of 2,3-dichloro-5,6-dicyano-benzoquinone (4 mmol) in DCM (30 ml) was added and stirred for half an hour, followed by injecting 74 mmol triethylamine (8 ml) into the dark residue. Then 75 mmol (12 ml) boron trifluoride ethyl ether complex was injected slowly. After stirring for 3 h, the solution was washed with an aqueous solution of $NaHCO_3$, the organic layer was dried over $Na_2SO_4$, and the solvent was evaporated by reduced pressure.

Chromatography on a silica column was carried out and eluted with ethyl acetate/petroleum ether (1:4 *v*/*v*) mitxture. The collected red fraction was subsequently recrystallized from chloroform/hexane (1:4 *v*/*v*) to acquire 507 mg (yield 24%) of the title compound.

Red slabs of (I) were acquired after 7 weeks by slow evaporation of a DCM/hexane (1:2 *v*/*v*) solution.

## Refinement

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

## Figures

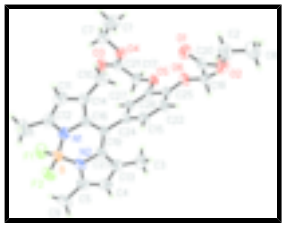


Fig. 1.

### 8-[3,4-Bis(ethoxycarbonylmethoxy)phenyl]- 4,4-difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-s-indacene

*Crystal data*

| | |
|---|---|
| $C_{27}H_{31}BF_2N_2O_6$ | $F_{000}$ = 1112 |
| $M_r$ = 528.35 | $D_x$ = 1.315 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Melting point: 392K K |
| Hall symbol: -P 2yn | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 9.6068 (6) Å | Cell parameters from 6052 reflections |
| $b$ = 7.5418 (6) Å | θ = 2.2–27.5º |
| $c$ = 36.896 (2) Å | μ = 0.10 mm$^{-1}$ |
| β = 93.231 (5)º | $T$ = 298 (2) K |
| $V$ = 2669.0 (3) Å$^3$ | Slab, red |
| $Z$ = 4 | 0.62 × 0.21 × 0.08 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX II CCD diffractometer | 4681 independent reflections |
| Radiation source: fine-focus sealed tube | 2283 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.079 |
| Detector resolution: 3.53 pixels mm$^{-1}$ | $\theta_{max}$ = 25.0º |
| $T$ = 298(2) K | $\theta_{min}$ = 2.2º |
| ω scans | $h$ = −11→11 |
| Absorption correction: multi-scan (SADABS; Bruker, 1997) | $k$ = −8→8 |
| $T_{min}$ = 0.940, $T_{max}$ = 0.992 | $l$ = −43→43 |
| 12387 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.056 | H-atom parameters constrained |
| $wR(F^2)$ = 0.152 | $w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|---|---|
| $S$ = 0.93 | $(\Delta/\sigma)_{max} < 0.001$ |
| 4681 reflections | $\Delta\rho_{max} = 0.25$ e Å$^{-3}$ |
| 343 parameters | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. Mp.: 392–393 K(uncorrected);

$^{1}$H NMR ($CDCl_3$, 400 MHz): δ 6.961 (d, 1H, Ar—H, J = 8.4 Hz), 6.840 (dd, 1H, Ar—H, J = 8.0 Hz), 6.746 (d, 1H, Ar—H, J = 1.2 Hz), 5.962 (s, 2H, pyrrole-H), 4.763 (s, 2H, methoxy-H), 4.698 (s, 2H, methoxy-H), 4.287–4.234 (m, 2H, ethoxy-H), 4.222–4.169 (m, 2H, ethoxy-H), 2.530 (s, 6H, pyrrole-$CH_3$), 1.430 (s, 6H, pyrrole-$CH_3$), 1.278 (t, 3H, ethyl ester-$CH_3$), 1.242 (t, 3H, ethyl ester-$CH_3$);

$^{13}$C NMR ($CDCl_3$, 400 MHz): δ 168.667, 168.464, 155.704, 148.751, 148.432, 143.270, 140.728, 131.649, 128.818, 121.834, 121.341, 115.443, 114.494, 66.679, 66.292, 61.616, 61.555, 14.720, 14.500, 14.280, 14.211;

HRMS(TOF MS EI$^{+}$): Found: 528.2246, calculated: 528.2243.

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on $F^2$, conventional *R*-factors *R* are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O6 | −0.1822 (2) | 0.8666 (3) | 0.06347 (5) | 0.0579 (6) |
| O5 | −0.00951 (19) | 1.0920 (3) | 0.04268 (5) | 0.0507 (6) |
| O4 | 0.2203 (2) | 0.9416 (3) | 0.02309 (5) | 0.0577 (6) |
| F2 | 0.5394 (2) | 1.1742 (3) | 0.24401 (5) | 0.0815 (7) |
| C27 | 0.1234 (3) | 1.0922 (4) | 0.10128 (8) | 0.0458 (8) |
| H27A | 0.1871 | 1.1750 | 0.0936 | 0.055* |
| O3 | 0.3274 (2) | 1.1992 (4) | 0.01361 (6) | 0.0743 (8) |
| C26 | 0.0153 (3) | 1.0365 (4) | 0.07791 (7) | 0.0424 (8) |
| N2 | 0.3670 (3) | 1.2976 (4) | 0.20215 (7) | 0.0535 (7) |
| C25 | −0.0805 (3) | 0.9139 (4) | 0.08945 (8) | 0.0467 (8) |
| N1 | 0.4885 (3) | 1.0196 (4) | 0.18797 (6) | 0.0534 (7) |
| F1 | 0.61150 (19) | 1.2953 (3) | 0.19255 (6) | 0.0860 (7) |
| O2 | −0.3397 (2) | 0.4649 (3) | 0.08974 (7) | 0.0765 (8) |
| C24 | 0.1374 (3) | 1.0247 (4) | 0.13646 (8) | 0.0452 (8) |
| O1 | −0.1254 (2) | 0.5049 (4) | 0.07078 (7) | 0.0823 (8) |
| C23 | 0.2524 (3) | 1.2409 (5) | 0.17989 (8) | 0.0482 (8) |
| C22 | −0.0683 (3) | 0.8504 (5) | 0.12437 (8) | 0.0547 (9) |

| | | | | |
|---|---|---|---|---|
| H22A | −0.1333 | 0.7701 | 0.1324 | 0.066* |
| C21 | 0.2249 (3) | 1.1173 (5) | 0.02085 (8) | 0.0536 (9) |
| C20 | −0.2360 (4) | 0.5594 (5) | 0.07760 (9) | 0.0566 (9) |
| C19 | 0.2586 (3) | 1.0815 (4) | 0.16097 (7) | 0.0443 (8) |
| C18 | −0.2858 (3) | 0.7483 (5) | 0.07327 (9) | 0.0569 (9) |
| H18A | −0.3620 | 0.7518 | 0.0549 | 0.068* |
| H18B | −0.3217 | 0.7874 | 0.0960 | 0.068* |
| C17 | 0.0898 (3) | 1.2038 (5) | 0.02750 (8) | 0.0539 (9) |
| H17A | 0.1075 | 1.3036 | 0.0437 | 0.065* |
| H17B | 0.0502 | 1.2501 | 0.0047 | 0.065* |
| C16 | 0.3740 (3) | 0.9695 (5) | 0.16491 (7) | 0.0488 (8) |
| C15 | 0.0413 (3) | 0.9063 (5) | 0.14764 (8) | 0.0543 (9) |
| H15A | 0.0494 | 0.8625 | 0.1712 | 0.065* |
| C14 | 0.4054 (3) | 0.8035 (5) | 0.14945 (8) | 0.0544 (9) |
| C13 | 0.1425 (3) | 1.3670 (5) | 0.18329 (9) | 0.0575 (9) |
| C12 | 0.5842 (3) | 0.8895 (6) | 0.18741 (9) | 0.0629 (10) |
| C11 | 0.5356 (4) | 0.7565 (5) | 0.16382 (10) | 0.0667 (11) |
| H11A | 0.5832 | 0.6530 | 0.1586 | 0.080* |
| C10 | 0.3210 (4) | 0.6941 (5) | 0.12208 (9) | 0.0720 (11) |
| H10A | 0.3709 | 0.5874 | 0.1170 | 0.108* |
| H10B | 0.3050 | 0.7609 | 0.1001 | 0.108* |
| H10C | 0.2331 | 0.6641 | 0.1317 | 0.108* |
| C9 | 0.4263 (4) | 1.5517 (6) | 0.24383 (9) | 0.0861 (13) |
| H9A | 0.5151 | 1.4933 | 0.2464 | 0.129* |
| H9B | 0.3863 | 1.5578 | 0.2671 | 0.129* |
| H9C | 0.4384 | 1.6695 | 0.2347 | 0.129* |
| C8 | 0.7207 (3) | 0.8948 (6) | 0.20889 (11) | 0.0952 (15) |
| H8A | 0.7273 | 1.0029 | 0.2227 | 0.143* |
| H8B | 0.7954 | 0.8897 | 0.1927 | 0.143* |
| H8C | 0.7270 | 0.7953 | 0.2251 | 0.143* |
| C7 | 0.3452 (4) | 0.8467 (6) | 0.01443 (12) | 0.0840 (13) |
| H7A | 0.4207 | 0.8733 | 0.0321 | 0.101* |
| H7B | 0.3735 | 0.8809 | −0.0094 | 0.101* |
| C6 | −0.4550 (4) | 0.1959 (6) | 0.09749 (12) | 0.0955 (14) |
| H6A | −0.4454 | 0.0703 | 0.1011 | 0.143* |
| H6B | −0.5098 | 0.2180 | 0.0754 | 0.143* |
| H6C | −0.5004 | 0.2470 | 0.1175 | 0.143* |
| C5 | 0.3318 (4) | 1.4503 (5) | 0.21813 (9) | 0.0628 (10) |
| C4 | 0.1942 (4) | 1.4927 (5) | 0.20706 (9) | 0.0663 (10) |
| H4A | 0.1455 | 1.5912 | 0.2147 | 0.080* |
| B | 0.5065 (4) | 1.1986 (6) | 0.20735 (10) | 0.0595 (11) |
| C3 | −0.0018 (4) | 1.3661 (5) | 0.16650 (10) | 0.0809 (12) |
| H3A | −0.0504 | 1.4699 | 0.1740 | 0.121* |
| H3B | −0.0498 | 1.2621 | 0.1741 | 0.121* |
| H3C | 0.0015 | 1.3657 | 0.1405 | 0.121* |
| C2 | −0.3184 (4) | 0.2748 (5) | 0.09514 (12) | 0.0856 (13) |
| H2A | −0.2714 | 0.2244 | 0.0749 | 0.103* |
| H2B | −0.2618 | 0.2531 | 0.1173 | 0.103* |
| C1 | 0.3110 (5) | 0.6528 (6) | 0.01509 (13) | 0.1050 (15) |

| | | | | |
|---|---|---|---|---|
| H1A | 0.3918 | 0.5853 | 0.0095 | 0.158* |
| H1B | 0.2364 | 0.6281 | −0.0026 | 0.158* |
| H1C | 0.2829 | 0.6207 | 0.0388 | 0.158* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O6 | 0.0483 (12) | 0.0693 (17) | 0.0549 (13) | −0.0203 (12) | −0.0077 (11) | 0.0065 (12) |
| O5 | 0.0440 (11) | 0.0644 (17) | 0.0433 (12) | −0.0055 (11) | −0.0022 (9) | 0.0107 (11) |
| O4 | 0.0510 (13) | 0.0592 (19) | 0.0629 (14) | 0.0028 (13) | 0.0033 (11) | 0.0007 (12) |
| F2 | 0.0780 (13) | 0.1115 (19) | 0.0524 (12) | −0.0054 (13) | −0.0198 (10) | −0.0045 (11) |
| C27 | 0.0427 (16) | 0.048 (2) | 0.0469 (18) | −0.0087 (16) | 0.0020 (14) | 0.0028 (16) |
| O3 | 0.0523 (14) | 0.085 (2) | 0.0860 (17) | −0.0195 (14) | 0.0077 (12) | 0.0007 (15) |
| C26 | 0.0389 (15) | 0.045 (2) | 0.0430 (18) | 0.0013 (16) | −0.0003 (14) | 0.0030 (16) |
| N2 | 0.0592 (17) | 0.055 (2) | 0.0455 (15) | −0.0085 (15) | −0.0056 (13) | −0.0067 (14) |
| C25 | 0.0404 (16) | 0.051 (2) | 0.0485 (19) | −0.0083 (16) | −0.0019 (15) | 0.0019 (16) |
| N1 | 0.0473 (15) | 0.063 (2) | 0.0497 (15) | 0.0006 (15) | −0.0027 (13) | 0.0047 (15) |
| F1 | 0.0632 (12) | 0.0949 (18) | 0.1007 (16) | −0.0268 (13) | 0.0126 (11) | −0.0043 (13) |
| O2 | 0.0625 (15) | 0.0504 (18) | 0.119 (2) | −0.0049 (13) | 0.0233 (14) | 0.0027 (15) |
| C24 | 0.0443 (17) | 0.049 (2) | 0.0424 (18) | 0.0000 (17) | 0.0010 (14) | −0.0006 (16) |
| O1 | 0.0527 (14) | 0.078 (2) | 0.117 (2) | 0.0019 (14) | 0.0122 (14) | −0.0158 (16) |
| C23 | 0.0500 (18) | 0.053 (2) | 0.0415 (18) | −0.0021 (18) | −0.0016 (15) | 0.0006 (17) |
| C22 | 0.0516 (18) | 0.063 (3) | 0.049 (2) | −0.0173 (18) | 0.0011 (16) | 0.0040 (18) |
| C21 | 0.051 (2) | 0.059 (3) | 0.051 (2) | −0.006 (2) | −0.0065 (16) | 0.0029 (19) |
| C20 | 0.0440 (19) | 0.062 (3) | 0.063 (2) | −0.004 (2) | −0.0056 (17) | −0.0109 (19) |
| C19 | 0.0437 (17) | 0.051 (2) | 0.0376 (17) | −0.0056 (16) | −0.0003 (14) | 0.0027 (16) |
| C18 | 0.0426 (17) | 0.066 (3) | 0.062 (2) | −0.0160 (19) | −0.0021 (16) | 0.0035 (19) |
| C17 | 0.0470 (18) | 0.058 (2) | 0.056 (2) | −0.0039 (18) | 0.0019 (16) | 0.0113 (18) |
| C16 | 0.0457 (17) | 0.059 (3) | 0.0411 (17) | −0.0051 (18) | −0.0008 (14) | 0.0038 (17) |
| C15 | 0.0577 (19) | 0.064 (3) | 0.0411 (18) | −0.0140 (19) | 0.0001 (16) | 0.0066 (16) |
| C14 | 0.057 (2) | 0.056 (3) | 0.051 (2) | 0.0013 (19) | 0.0070 (16) | 0.0000 (18) |
| C13 | 0.059 (2) | 0.060 (3) | 0.054 (2) | 0.000 (2) | 0.0037 (17) | 0.0042 (19) |
| C12 | 0.056 (2) | 0.076 (3) | 0.056 (2) | 0.008 (2) | 0.0037 (18) | 0.012 (2) |
| C11 | 0.066 (2) | 0.067 (3) | 0.067 (2) | 0.015 (2) | 0.0134 (19) | 0.008 (2) |
| C10 | 0.086 (3) | 0.064 (3) | 0.065 (2) | −0.002 (2) | 0.007 (2) | −0.011 (2) |
| C9 | 0.113 (3) | 0.091 (3) | 0.054 (2) | −0.027 (3) | −0.007 (2) | −0.019 (2) |
| C8 | 0.057 (2) | 0.134 (4) | 0.093 (3) | 0.017 (3) | −0.017 (2) | 0.006 (3) |
| C7 | 0.059 (2) | 0.089 (4) | 0.103 (3) | 0.014 (2) | 0.003 (2) | −0.015 (3) |
| C6 | 0.103 (3) | 0.067 (3) | 0.117 (4) | −0.027 (3) | 0.017 (3) | 0.001 (3) |
| C5 | 0.079 (3) | 0.064 (3) | 0.0453 (19) | −0.010 (2) | 0.0061 (18) | −0.0028 (19) |
| C4 | 0.087 (3) | 0.055 (3) | 0.058 (2) | 0.010 (2) | 0.010 (2) | −0.006 (2) |
| B | 0.050 (2) | 0.079 (4) | 0.050 (2) | −0.010 (2) | 0.0032 (19) | −0.003 (2) |
| C3 | 0.069 (2) | 0.082 (3) | 0.090 (3) | 0.017 (2) | −0.005 (2) | 0.002 (2) |
| C2 | 0.082 (3) | 0.048 (3) | 0.126 (4) | −0.002 (2) | −0.003 (2) | −0.002 (2) |
| C1 | 0.122 (4) | 0.069 (4) | 0.123 (4) | 0.022 (3) | 0.002 (3) | −0.018 (3) |

*Geometric parameters (Å, °)*

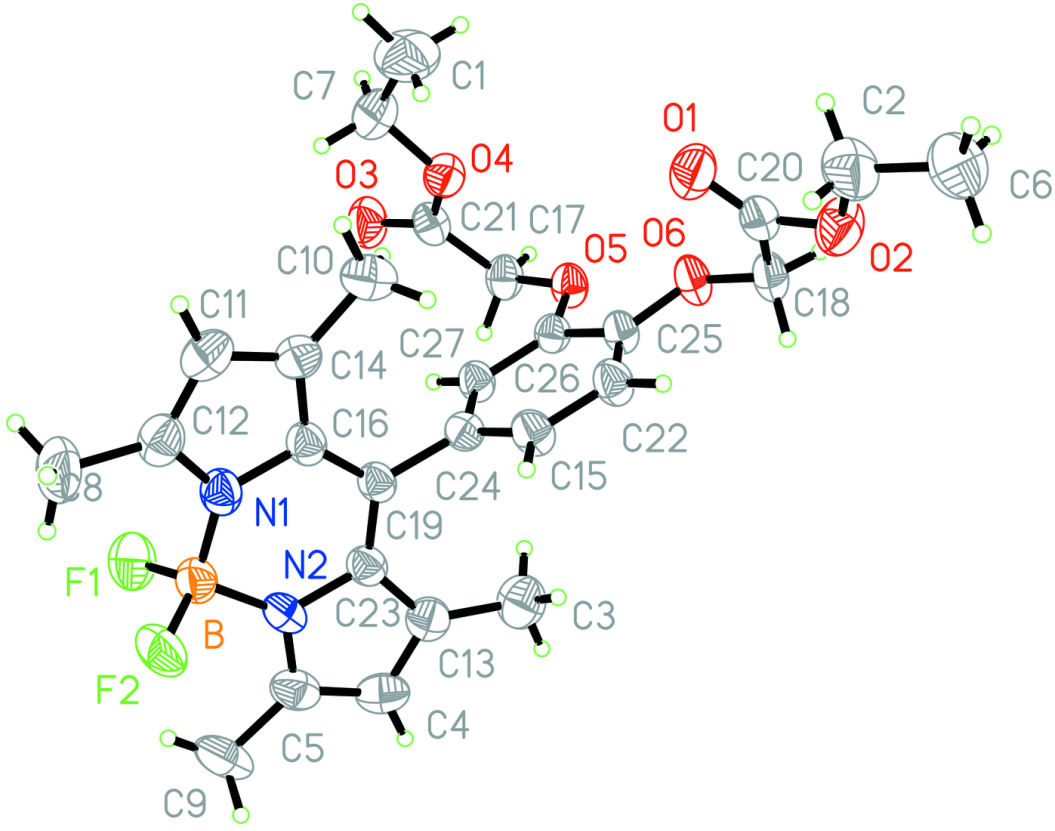
| | | | |
|---|---|---|---|
| B—N1 | 1.533 (5) | C16—C14 | 1.415 (5) |

| | | | |
|---|---|---|---|
| B—N2 | 1.537 (5) | C15—H15A | 0.9300 |
| B—F1 | 1.382 (4) | C14—C11 | 1.377 (4) |
| B—F2 | 1.384 (4) | C14—C10 | 1.506 (4) |
| O6—C25 | 1.377 (3) | C13—C4 | 1.366 (5) |
| O6—C18 | 1.399 (3) | C13—C3 | 1.486 (4) |
| O5—C26 | 1.373 (3) | C12—C11 | 1.392 (5) |
| O5—C17 | 1.413 (3) | C12—C8 | 1.494 (4) |
| O4—C21 | 1.328 (4) | C11—H11A | 0.9300 |
| O4—C7 | 1.448 (4) | C10—H10A | 0.9600 |
| C27—C26 | 1.377 (4) | C10—H10B | 0.9600 |
| C27—C24 | 1.394 (4) | C10—H10C | 0.9600 |
| C27—H27A | 0.9300 | C9—C5 | 1.487 (5) |
| O3—C21 | 1.206 (4) | C9—H9A | 0.9600 |
| C26—C25 | 1.389 (4) | C9—H9B | 0.9600 |
| N2—C5 | 1.346 (4) | C9—H9C | 0.9600 |
| N2—C23 | 1.402 (4) | C8—H8A | 0.9600 |
| C25—C22 | 1.373 (4) | C8—H8B | 0.9600 |
| N1—C12 | 1.345 (4) | C8—H8C | 0.9600 |
| N1—C16 | 1.404 (4) | C7—C1 | 1.499 (5) |
| O2—C20 | 1.323 (4) | C7—H7A | 0.9700 |
| O2—C2 | 1.461 (4) | C7—H7B | 0.9700 |
| C24—C15 | 1.365 (4) | C6—C2 | 1.448 (5) |
| C24—C19 | 1.496 (4) | C6—H6A | 0.9600 |
| O1—C20 | 1.180 (4) | C6—H6B | 0.9600 |
| C23—C19 | 1.393 (4) | C6—H6C | 0.9600 |
| C23—C13 | 1.432 (4) | C5—C4 | 1.398 (5) |
| C22—C15 | 1.386 (4) | C4—H4A | 0.9300 |
| C22—H22A | 0.9300 | C3—H3A | 0.9600 |
| C21—C17 | 1.485 (4) | C3—H3B | 0.9600 |
| C20—C18 | 1.508 (5) | C3—H3C | 0.9600 |
| C19—C16 | 1.395 (4) | C2—H2A | 0.9700 |
| C18—H18A | 0.9700 | C2—H2B | 0.9700 |
| C18—H18B | 0.9700 | C1—H1A | 0.9600 |
| C17—H17A | 0.9700 | C1—H1B | 0.9600 |
| C17—H17B | 0.9700 | C1—H1C | 0.9600 |
| C25—O6—C18 | 118.2 (2) | C14—C11—C12 | 108.7 (3) |
| C26—O5—C17 | 118.1 (2) | C14—C11—H11A | 125.7 |
| C21—O4—C7 | 116.6 (3) | C12—C11—H11A | 125.7 |
| C26—C27—C24 | 120.1 (3) | C14—C10—H10A | 109.5 |
| C26—C27—H27A | 119.9 | C14—C10—H10B | 109.5 |
| C24—C27—H27A | 119.9 | H10A—C10—H10B | 109.5 |
| O5—C26—C27 | 125.8 (3) | C14—C10—H10C | 109.5 |
| O5—C26—C25 | 114.2 (2) | H10A—C10—H10C | 109.5 |
| C27—C26—C25 | 120.0 (3) | H10B—C10—H10C | 109.5 |
| C5—N2—C23 | 108.0 (3) | C5—C9—H9A | 109.5 |
| C5—N2—B | 126.9 (3) | C5—C9—H9B | 109.5 |
| C23—N2—B | 125.1 (3) | H9A—C9—H9B | 109.5 |
| C22—C25—O6 | 125.6 (3) | C5—C9—H9C | 109.5 |
| C22—C25—C26 | 119.8 (3) | H9A—C9—H9C | 109.5 |

| | | | |
|---|---|---|---|
| O6—C25—C26 | 114.6 (3) | H9B—C9—H9C | 109.5 |
| C12—N1—C16 | 107.9 (3) | C12—C8—H8A | 109.5 |
| C12—N1—B | 126.2 (3) | C12—C8—H8B | 109.5 |
| C16—N1—B | 125.7 (3) | H8A—C8—H8B | 109.5 |
| C20—O2—C2 | 118.2 (3) | C12—C8—H8C | 109.5 |
| C15—C24—C27 | 119.2 (3) | H8A—C8—H8C | 109.5 |
| C15—C24—C19 | 121.4 (3) | H8B—C8—H8C | 109.5 |
| C27—C24—C19 | 119.3 (3) | O4—C7—C1 | 107.1 (3) |
| C19—C23—N2 | 120.1 (3) | O4—C7—H7A | 110.3 |
| C19—C23—C13 | 132.0 (3) | C1—C7—H7A | 110.3 |
| N2—C23—C13 | 107.8 (3) | O4—C7—H7B | 110.3 |
| C25—C22—C15 | 119.8 (3) | C1—C7—H7B | 110.3 |
| C25—C22—H22A | 120.1 | H7A—C7—H7B | 108.6 |
| C15—C22—H22A | 120.1 | C2—C6—H6A | 109.5 |
| O3—C21—O4 | 123.7 (3) | C2—C6—H6B | 109.5 |
| O3—C21—C17 | 123.0 (4) | H6A—C6—H6B | 109.5 |
| O4—C21—C17 | 113.3 (3) | C2—C6—H6C | 109.5 |
| O1—C20—O2 | 126.1 (4) | H6A—C6—H6C | 109.5 |
| O1—C20—C18 | 126.2 (3) | H6B—C6—H6C | 109.5 |
| O2—C20—C18 | 107.7 (3) | N2—C5—C4 | 108.9 (3) |
| C23—C19—C16 | 121.8 (3) | N2—C5—C9 | 124.1 (3) |
| C23—C19—C24 | 119.7 (3) | C4—C5—C9 | 127.0 (4) |
| C16—C19—C24 | 118.5 (3) | C13—C4—C5 | 109.4 (3) |
| O6—C18—C20 | 113.8 (3) | C13—C4—H4A | 125.3 |
| O6—C18—H18A | 108.8 | C5—C4—H4A | 125.3 |
| C20—C18—H18A | 108.8 | F1—B—F2 | 108.9 (3) |
| O6—C18—H18B | 108.8 | F1—B—N1 | 110.3 (3) |
| C20—C18—H18B | 108.8 | F2—B—N1 | 110.7 (3) |
| H18A—C18—H18B | 107.7 | F1—B—N2 | 110.2 (3) |
| O5—C17—C21 | 114.8 (3) | F2—B—N2 | 109.6 (3) |
| O5—C17—H17A | 108.6 | N1—B—N2 | 107.2 (3) |
| C21—C17—H17A | 108.6 | C13—C3—H3A | 109.5 |
| O5—C17—H17B | 108.6 | C13—C3—H3B | 109.5 |
| C21—C17—H17B | 108.6 | H3A—C3—H3B | 109.5 |
| H17A—C17—H17B | 107.5 | C13—C3—H3C | 109.5 |
| C19—C16—N1 | 119.4 (3) | H3A—C3—H3C | 109.5 |
| C19—C16—C14 | 132.9 (3) | H3B—C3—H3C | 109.5 |
| N1—C16—C14 | 107.7 (3) | C6—C2—O2 | 106.9 (3) |
| C24—C15—C22 | 121.0 (3) | C6—C2—H2A | 110.3 |
| C24—C15—H15A | 119.5 | O2—C2—H2A | 110.3 |
| C22—C15—H15A | 119.5 | C6—C2—H2B | 110.3 |
| C11—C14—C16 | 106.4 (3) | O2—C2—H2B | 110.3 |
| C11—C14—C10 | 124.1 (4) | H2A—C2—H2B | 108.6 |
| C16—C14—C10 | 129.5 (3) | C7—C1—H1A | 109.5 |
| C4—C13—C23 | 105.9 (3) | C7—C1—H1B | 109.5 |
| C4—C13—C3 | 124.6 (3) | H1A—C1—H1B | 109.5 |
| C23—C13—C3 | 129.5 (3) | C7—C1—H1C | 109.5 |
| N1—C12—C11 | 109.3 (3) | H1A—C1—H1C | 109.5 |
| N1—C12—C8 | 123.5 (4) | H1B—C1—H1C | 109.5 |

| | | | |
|---|---|---|---|
| C11—C12—C8 | 127.2 (4) | | |
| C17—O5—C26—C27 | 6.0 (4) | B—N1—C16—C14 | 173.3 (3) |
| C17—O5—C26—C25 | −174.1 (3) | C27—C24—C15—C22 | 1.1 (5) |
| C24—C27—C26—O5 | −179.7 (3) | C19—C24—C15—C22 | −178.0 (3) |
| C24—C27—C26—C25 | 0.5 (5) | C25—C22—C15—C24 | 0.3 (5) |
| C18—O6—C25—C22 | 2.3 (5) | C19—C16—C14—C11 | −179.7 (3) |
| C18—O6—C25—C26 | −177.8 (3) | N1—C16—C14—C11 | 0.9 (3) |
| O5—C26—C25—C22 | −178.9 (3) | C19—C16—C14—C10 | 1.6 (6) |
| C27—C26—C25—C22 | 0.9 (5) | N1—C16—C14—C10 | −177.7 (3) |
| O5—C26—C25—O6 | 1.1 (4) | C19—C23—C13—C4 | 176.6 (3) |
| C27—C26—C25—O6 | −179.0 (3) | N2—C23—C13—C4 | 0.0 (4) |
| C26—C27—C24—C15 | −1.5 (5) | C19—C23—C13—C3 | −1.2 (6) |
| C26—C27—C24—C19 | 177.6 (3) | N2—C23—C13—C3 | −177.7 (3) |
| C5—N2—C23—C19 | −177.7 (3) | C16—N1—C12—C11 | 1.2 (4) |
| B—N2—C23—C19 | 1.5 (5) | B—N1—C12—C11 | −173.4 (3) |
| C5—N2—C23—C13 | −0.7 (4) | C16—N1—C12—C8 | −179.6 (3) |
| B—N2—C23—C13 | 178.5 (3) | B—N1—C12—C8 | 5.7 (5) |
| O6—C25—C22—C15 | 178.6 (3) | C16—C14—C11—C12 | −0.2 (4) |
| C26—C25—C22—C15 | −1.3 (5) | C10—C14—C11—C12 | 178.5 (3) |
| C7—O4—C21—O3 | −3.2 (5) | N1—C12—C11—C14 | −0.6 (4) |
| C7—O4—C21—C17 | 175.8 (3) | C8—C12—C11—C14 | −179.7 (3) |
| C2—O2—C20—O1 | −0.6 (5) | C21—O4—C7—C1 | −174.3 (3) |
| C2—O2—C20—C18 | 178.2 (3) | C23—N2—C5—C4 | 1.1 (4) |
| N2—C23—C19—C16 | 3.5 (5) | B—N2—C5—C4 | −178.1 (3) |
| C13—C23—C19—C16 | −172.7 (3) | C23—N2—C5—C9 | 180.0 (3) |
| N2—C23—C19—C24 | −178.3 (3) | B—N2—C5—C9 | 0.8 (5) |
| C13—C23—C19—C24 | 5.5 (5) | C23—C13—C4—C5 | 0.6 (4) |
| C15—C24—C19—C23 | −98.2 (4) | C3—C13—C4—C5 | 178.5 (3) |
| C27—C24—C19—C23 | 82.7 (4) | N2—C5—C4—C13 | −1.1 (4) |
| C15—C24—C19—C16 | 80.1 (4) | C9—C5—C4—C13 | −180.0 (3) |
| C27—C24—C19—C16 | −99.0 (3) | C12—N1—B—F1 | 63.3 (4) |
| C25—O6—C18—C20 | −73.6 (4) | C16—N1—B—F1 | −110.3 (3) |
| O1—C20—C18—O6 | −6.4 (5) | C12—N1—B—F2 | −57.2 (4) |
| O2—C20—C18—O6 | 174.9 (3) | C16—N1—B—F2 | 129.1 (3) |
| C26—O5—C17—C21 | 69.4 (4) | C12—N1—B—N2 | −176.7 (3) |
| O3—C21—C17—O5 | −167.7 (3) | C16—N1—B—N2 | 9.6 (4) |
| O4—C21—C17—O5 | 13.3 (4) | C5—N2—B—F1 | −68.2 (4) |
| C23—C19—C16—N1 | −1.3 (4) | C23—N2—B—F1 | 112.8 (4) |
| C24—C19—C16—N1 | −179.5 (3) | C5—N2—B—F2 | 51.7 (5) |
| C23—C19—C16—C14 | 179.4 (3) | C23—N2—B—F2 | −127.4 (3) |
| C24—C19—C16—C14 | 1.2 (5) | C5—N2—B—N1 | 171.8 (3) |
| C12—N1—C16—C19 | 179.2 (3) | C23—N2—B—N1 | −7.2 (4) |
| B—N1—C16—C19 | −6.1 (5) | C20—O2—C2—C6 | −162.9 (3) |
| C12—N1—C16—C14 | −1.3 (3) | | |

**Fig. 1**


C7
C1
O3
O4
O1
C20
C2
C6
C21
C17
O5
O6
O2
C10
C18
C11
C27
C25
C14
C26
C12
C16
C22
C8
C24
C15
N1
C19
F1
N2
C23
C3
B
C13
F2
C4
C5
C9