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8-{4-[*N*,*N*-Bis(2-chloroethyl)amino]-phenyl}-4,4-difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

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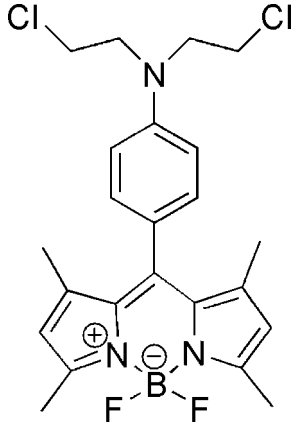
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.058; wR factor = 0.178; data-to-parameter ratio = 17.8.

In the title compound, $C_{23}H_{26}BCl_2F_2N_3$, the three boron-dipyrromethene (BODIPY) fused heterocyclic rings are almost planar [r.m.s. deviation = 0.024 (9) Å]. The dihedral angle between the planes of the benzene ring and the BODIPY fused-ring fragment is 74.9 (8)°. The sp^3-hybridized B centre appears as a slightly distorted tetrahedron, with N—B—N and F—B—F angles of 106.82 (15) and 109.83 (17)°, respectively. The two B—N distances in the central ring are almost identical, indicating delocalization of their charge. The two Cl atoms are disordered, with occupancies of approximately 0.85:0.15 and 0.60:0.40.

Related literature

For related literature, see: Stavis *et al.*, (2005); Teske *et al.*, (2006); Yee *et al.*, (2005); Peng *et al.*, (2007); Yu *et al.*, (2007).



Experimental

Crystal data

$C_{23}H_{26}BCl_2F_2N_3$
M_r = 464.18
Triclinic, $P\overline{1}$
a = 7.2673 (2) Å
b = 12.3571 (2) Å
c = 13.2963 (2) Å
α = 92.650 (1)°
β = 101.089 (1)°
γ = 90.205 (1)°
V = 1170.40 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 298 (2) K
0.55 × 0.35 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
10164 measured reflections
5337 independent reflections
3776 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.178
S = 1.03
5337 reflections
299 parameters
13 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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: PK2028).

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supplementary materials

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8-{4-[*N*,*N*-Bis(2-chloroethyl)amino]phenyl}-4,4-difluoro-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

M.-Z. Tian, S. Gao and X.-J. Peng

Comment

Boron-dipyrromethene (BODIPY) dyes are well known and have attracted much interest in the design of fluorescence labels (Stavis *et al.*, 2005; Teske *et al.*, 2006; Yee *et al.*, 2005) and molecular fluorescence sensors (Peng *et al.*, 2007). Detailed information on their molecular and crystal structures is necessary to understand their photophysical and photochemical properties (Yu *et al.*, 2007). As part of our work on BODIPY compounds, we report here the synthesis and crystal structure (Fig.1) of the title compound.

As shown in Fig.1, 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 0.024 (9) Å. The maximum deviations from the mean plane for C4, C2 and N1 are 0.043 (3) Å, 0.043 (1) Å and 0.045 (0) Å, respectively. The two B—N bond lengths are nearly the same, indicating delocalization of the charge on them. Due to steric repulsion from the C1 and C10 methyl groups, the phenyl ring is twisted out of the plane of the BODIPY fused-ring system leading to a dihedral angle of 74.9 (8)° between phenyl and BODIPY groups. The two chlorine atoms are disordered with refined major:minor occupancies of 0.851 (8):0.149 (8) and 0.605 (19):0.395 (19) for Cl1/Cl1' and Cl2/Cl2' respectively.

Experimental

2,4-Dimethylpyrrole (2.16 ml) and 4-[*N*,*N*-Bis(2-chloroethyl)amino]benzaldehyde (3.04 g) were added to CH_2Cl_2 (800 ml) in a 1 l round-bottom flask. The mixture was bubbled with N_2 and trifluoroacetic acid (0.19 ml) was added and then stirred for 1.5 h. The resulting solution was washed with 0.1 *M* NaOH (200 ml) and then water (200 ml), dried over anhydrous Na_2SO_4, and filtered, and the solvent was evaporated on a rotary evaporator. The resultant product was immediately redissolved in toluene (50 ml), and *p*-chloranil (2.73 g) was added. After the mixture stirred for 10 min, triethylamine (8 ml), and boron trifluoride etherate (7 ml) were added. The mixture was stirred for 1.5 h, poured into water, and extracted with toluene. The toluene solution was extracted three times with 100 ml portions of water, and the solvent was evaporated on a rotary evaporator. The residue was redissolved in chloroform and subjected to silica gel flash column chromatography. Elution with EtOAc/hexane(1:8, *v/v*) yielded 2.18 g of the title compound (38.03%).

Refinement

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).

Figures

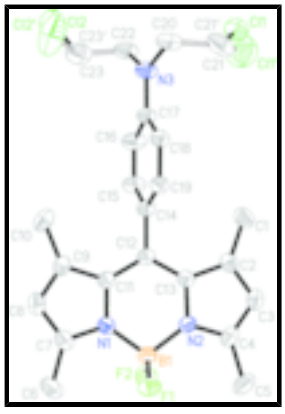


Fig. 1. The structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. Both chlorine atoms are disordered with occupancy factors of 0.851 (8):0.149 (8) and 0.605 (19):0.395 (19) for Cl1/Cl1' and Cl2/Cl2' respectively.

8-{4-[*N*,*N*-Bis(2-chloroethyl)amino]phenyl}-4,4-difluoro- 1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacene

Crystal data

$C_{23}H_{26}BCl_2F_2N_3$	$Z = 2$
$M_r = 464.18$	$F_{000} = 484$
Triclinic, $P\overline{1}$	$D_x = 1.317$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.2673$ (2) Å	Cell parameters from 3630 reflections
$b = 12.3571$ (2) Å	$\theta = 2.3$–27.5º
$c = 13.2963$ (2) Å	$\mu = 0.31$ mm^{-1}
$\alpha = 92.650$ (1)º	$T = 298$ (2) K
$\beta = 101.089$ (1)º	Block, brown red
$\gamma = 90.205$ (1)º	0.55 × 0.35 × 0.35 mm
$V = 1170.40$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3776 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.016$
Monochromator: graphite	$\theta_{max} = 27.5$º
$T = 298(2)$ K	$\theta_{min} = 2.3$º
φ and ω scans	$h = -7 \rightarrow 9$
Absorption correction: none	$k = -16 \rightarrow 15$
10164 measured reflections	$l = -17 \rightarrow 16$
5337 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.178$	$w = 1/[\sigma^2(F_o^2) + (0.0997P)^2 + 0.2019P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
5337 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
299 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
13 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.029 (4)

Special details

Experimental. ^{1}H NMR ($CDCl_3$, 400 MHz, Me_4Si): δ 1.46 (s, 6H, CH_3–H), 2.55 (s, 6H, CH_3–H), 3.66 (t, 4H, $ClCH_2$–H), 3.78 (t, 4H, NCH_2–H), 5.98 (s, 2H, pyrrole–H), 6.78 (d, 2H, Ar–H), 7.11 (d, 2H, Ar–H). ^{13}C NMR ($CDCl_3$, 400 MHz, Me_4Si): δ 14.76, 14.86, 40.42, 53.62, 112.38, 121.21, 124.01, 129.64, 132.20, 142.37, 143.24, 146.79, 155.26. HRMS (TOF MS EI$^+$): 486.1463, calculated: 486.1483.

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 $F^2 > 2\sigma(F^2)$ 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}	Occ. (<1)
N1	0.5707 (2)	0.89735 (12)	0.76379 (11)	0.0478 (4)	
N2	0.2896 (2)	0.77838 (13)	0.74161 (11)	0.0489 (4)	
N3	0.5791 (3)	0.64166 (13)	0.21042 (12)	0.0601 (4)	
B1	0.4133 (3)	0.85947 (19)	0.81732 (17)	0.0539 (5)	
F1	0.4908 (2)	0.80832 (12)	0.90633 (9)	0.0805 (4)	
F2	0.30906 (19)	0.94655 (11)	0.84098 (12)	0.0804 (4)	
C1	0.1105 (3)	0.6381 (2)	0.48592 (18)	0.0703 (6)	
H1A	0.1946	0.5808	0.4753	0.105*	
H1B	0.1209	0.6947	0.4403	0.105*	
H1C	−0.0159	0.6103	0.4725	0.105*	
C2	0.1600 (3)	0.68206 (16)	0.59418 (15)	0.0545 (4)	
C3	0.0532 (3)	0.66843 (18)	0.66860 (18)	0.0627 (5)	
H3	−0.0553	0.6261	0.6603	0.075*	
C4	0.1330 (3)	0.72743 (17)	0.75713 (17)	0.0584 (5)	
C5	0.0672 (4)	0.7367 (2)	0.8574 (2)	0.0809 (7)	

H5A	0.1515	0.6987	0.9080	0.121*	
H5B	−0.0564	0.7058	0.8489	0.121*	
H5C	0.0646	0.8117	0.8793	0.121*	
C6	0.7403 (4)	1.0122 (2)	0.91238 (18)	0.0775 (7)	
H6A	0.6195	1.0272	0.9286	0.116*	
H6B	0.8118	1.0784	0.9173	0.116*	
H6C	0.8052	0.9624	0.9598	0.116*	
C7	0.7161 (3)	0.96361 (16)	0.80636 (16)	0.0573 (5)	
C8	0.8329 (3)	0.97433 (17)	0.73636 (18)	0.0627 (5)	
H8	0.9422	1.0162	0.7474	0.075*	
C9	0.7617 (3)	0.91306 (15)	0.64747 (16)	0.0542 (5)	
C10	0.8506 (3)	0.9042 (2)	0.5557 (2)	0.0727 (6)	
H10A	0.7881	0.9512	0.5047	0.109*	
H10B	0.8406	0.8308	0.5282	0.109*	
H10C	0.9805	0.9251	0.5749	0.109*	
C11	0.5935 (2)	0.86418 (14)	0.66511 (13)	0.0456 (4)	
C12	0.4655 (2)	0.79184 (13)	0.60480 (12)	0.0432 (4)	
C13	0.3126 (2)	0.75103 (14)	0.64168 (13)	0.0465 (4)	
C14	0.4939 (2)	0.75405 (14)	0.50174 (13)	0.0457 (4)	
C15	0.4565 (3)	0.81847 (16)	0.41816 (15)	0.0613 (5)	
H15	0.4119	0.8881	0.4266	0.074*	
C16	0.4836 (4)	0.78230 (17)	0.32286 (15)	0.0668 (6)	
H16	0.4575	0.8282	0.2685	0.080*	
C17	0.5494 (3)	0.67820 (15)	0.30565 (13)	0.0496 (4)	
C18	0.5851 (3)	0.61276 (14)	0.39045 (13)	0.0470 (4)	
H18	0.6279	0.5427	0.3823	0.056*	
C19	0.5580 (2)	0.65029 (14)	0.48527 (13)	0.0453 (4)	
H19	0.5832	0.6049	0.5400	0.054*	
C20	0.6060 (4)	0.52832 (19)	0.18816 (17)	0.0719 (6)	
H20A	0.7034	0.5016	0.2413	0.086*	
H20B	0.6492	0.5202	0.1236	0.086*	
C21	0.4299 (5)	0.4592 (2)	0.1811 (2)	0.0951 (9)	0.851 (8)
H21A	0.3871	0.4655	0.2459	0.114*	0.851 (8)
H21B	0.4590	0.3838	0.1686	0.114*	0.851 (8)
Cl1	0.24906 (19)	0.49980 (10)	0.08129 (12)	0.0987 (6)	0.851 (8)
C21'	0.4299 (5)	0.4592 (2)	0.1811 (2)	0.0951 (9)	0.149 (8)
H21C	0.4203	0.4416	0.2503	0.114*	0.149 (8)
H21D	0.4479	0.3916	0.1444	0.114*	0.149 (8)
Cl1'	0.2157 (19)	0.5112 (12)	0.123 (2)	0.177 (5)*	0.149 (8)
C22	0.5763 (4)	0.7157 (2)	0.12880 (15)	0.0736 (6)	
H22A	0.4616	0.7571	0.1212	0.088*	
H22B	0.5743	0.6740	0.0650	0.088*	
C23	0.7391 (5)	0.7925 (2)	0.1464 (2)	0.1021 (10)	0.605 (19)
H23A	0.7473	0.8303	0.2127	0.122*	0.605 (19)
H23B	0.7181	0.8460	0.0944	0.122*	0.605 (19)
Cl2	0.9506 (7)	0.7285 (3)	0.1428 (6)	0.1265 (15)	0.605 (19)
C23'	0.7391 (5)	0.7925 (2)	0.1464 (2)	0.1021 (10)	0.395 (19)
H23C	0.7273	0.8447	0.2014	0.122*	0.395 (19)
H23D	0.7384	0.8317	0.0848	0.122*	0.395 (19)

Cl2'	0.9593 (12)	0.7225 (7)	0.1794 (12)	0.170 (3)	0.395 (19)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0511 (8)	0.0480 (8)	0.0430 (8)	−0.0007 (6)	0.0059 (6)	0.0008 (6)
N2	0.0529 (8)	0.0517 (8)	0.0445 (8)	−0.0005 (6)	0.0150 (6)	0.0039 (6)
N3	0.0892 (12)	0.0533 (9)	0.0410 (8)	0.0024 (8)	0.0203 (8)	0.0029 (7)
B1	0.0622 (12)	0.0569 (12)	0.0440 (11)	−0.0007 (10)	0.0148 (9)	−0.0005 (9)
F1	0.1003 (10)	0.0957 (10)	0.0423 (6)	−0.0168 (8)	0.0036 (6)	0.0149 (6)
F2	0.0800 (9)	0.0718 (8)	0.0938 (10)	0.0025 (7)	0.0346 (7)	−0.0246 (7)
C1	0.0573 (12)	0.0789 (15)	0.0679 (14)	−0.0061 (10)	−0.0006 (10)	−0.0127 (11)
C2	0.0487 (10)	0.0528 (10)	0.0593 (11)	0.0008 (8)	0.0043 (8)	−0.0003 (8)
C3	0.0516 (10)	0.0631 (12)	0.0741 (14)	−0.0093 (9)	0.0142 (9)	0.0040 (10)
C4	0.0556 (10)	0.0591 (11)	0.0659 (12)	−0.0007 (9)	0.0232 (9)	0.0099 (9)
C5	0.0847 (16)	0.0926 (17)	0.0770 (16)	−0.0041 (13)	0.0432 (13)	0.0111 (13)
C6	0.0873 (16)	0.0758 (15)	0.0597 (13)	−0.0107 (12)	−0.0066 (11)	−0.0098 (11)
C7	0.0585 (11)	0.0505 (10)	0.0580 (11)	−0.0018 (8)	−0.0007 (9)	0.0029 (8)
C8	0.0554 (11)	0.0561 (11)	0.0744 (14)	−0.0096 (9)	0.0063 (10)	0.0081 (10)
C9	0.0535 (10)	0.0471 (9)	0.0644 (12)	0.0005 (8)	0.0146 (9)	0.0136 (8)
C10	0.0698 (13)	0.0726 (14)	0.0854 (16)	0.0000 (11)	0.0348 (12)	0.0221 (12)
C11	0.0486 (9)	0.0443 (9)	0.0446 (9)	0.0035 (7)	0.0097 (7)	0.0075 (7)
C12	0.0484 (9)	0.0419 (8)	0.0401 (8)	0.0069 (7)	0.0095 (7)	0.0068 (7)
C13	0.0488 (9)	0.0461 (9)	0.0441 (9)	0.0017 (7)	0.0081 (7)	0.0015 (7)
C14	0.0514 (9)	0.0458 (9)	0.0402 (9)	0.0040 (7)	0.0096 (7)	0.0037 (7)
C15	0.0917 (15)	0.0483 (10)	0.0467 (10)	0.0224 (10)	0.0180 (10)	0.0097 (8)
C16	0.1033 (17)	0.0577 (11)	0.0420 (10)	0.0247 (11)	0.0165 (10)	0.0169 (8)
C17	0.0630 (11)	0.0492 (9)	0.0375 (9)	0.0041 (8)	0.0115 (8)	0.0038 (7)
C18	0.0576 (10)	0.0406 (8)	0.0430 (9)	0.0049 (7)	0.0097 (7)	0.0030 (7)
C19	0.0518 (9)	0.0435 (9)	0.0403 (9)	0.0020 (7)	0.0067 (7)	0.0088 (7)
C20	0.1028 (17)	0.0660 (13)	0.0484 (11)	0.0183 (12)	0.0199 (11)	−0.0043 (9)
C21	0.149 (3)	0.0555 (13)	0.0733 (16)	−0.0035 (15)	0.0061 (17)	−0.0082 (12)
Cl1	0.1037 (8)	0.1132 (8)	0.0763 (8)	−0.0152 (5)	0.0128 (5)	−0.0067 (5)
C21'	0.149 (3)	0.0555 (13)	0.0733 (16)	−0.0035 (15)	0.0061 (17)	−0.0082 (12)
C22	0.1150 (19)	0.0703 (14)	0.0382 (10)	0.0052 (13)	0.0203 (11)	0.0079 (9)
C23	0.165 (3)	0.0740 (16)	0.0763 (17)	−0.0158 (18)	0.0457 (19)	0.0090 (14)
Cl2	0.127 (2)	0.1102 (19)	0.155 (3)	−0.0298 (15)	0.067 (2)	−0.018 (2)
C23'	0.165 (3)	0.0740 (16)	0.0763 (17)	−0.0158 (18)	0.0457 (19)	0.0090 (14)
Cl2'	0.108 (3)	0.194 (5)	0.215 (7)	−0.011 (3)	0.031 (4)	0.092 (5)

Geometric parameters (Å, °)

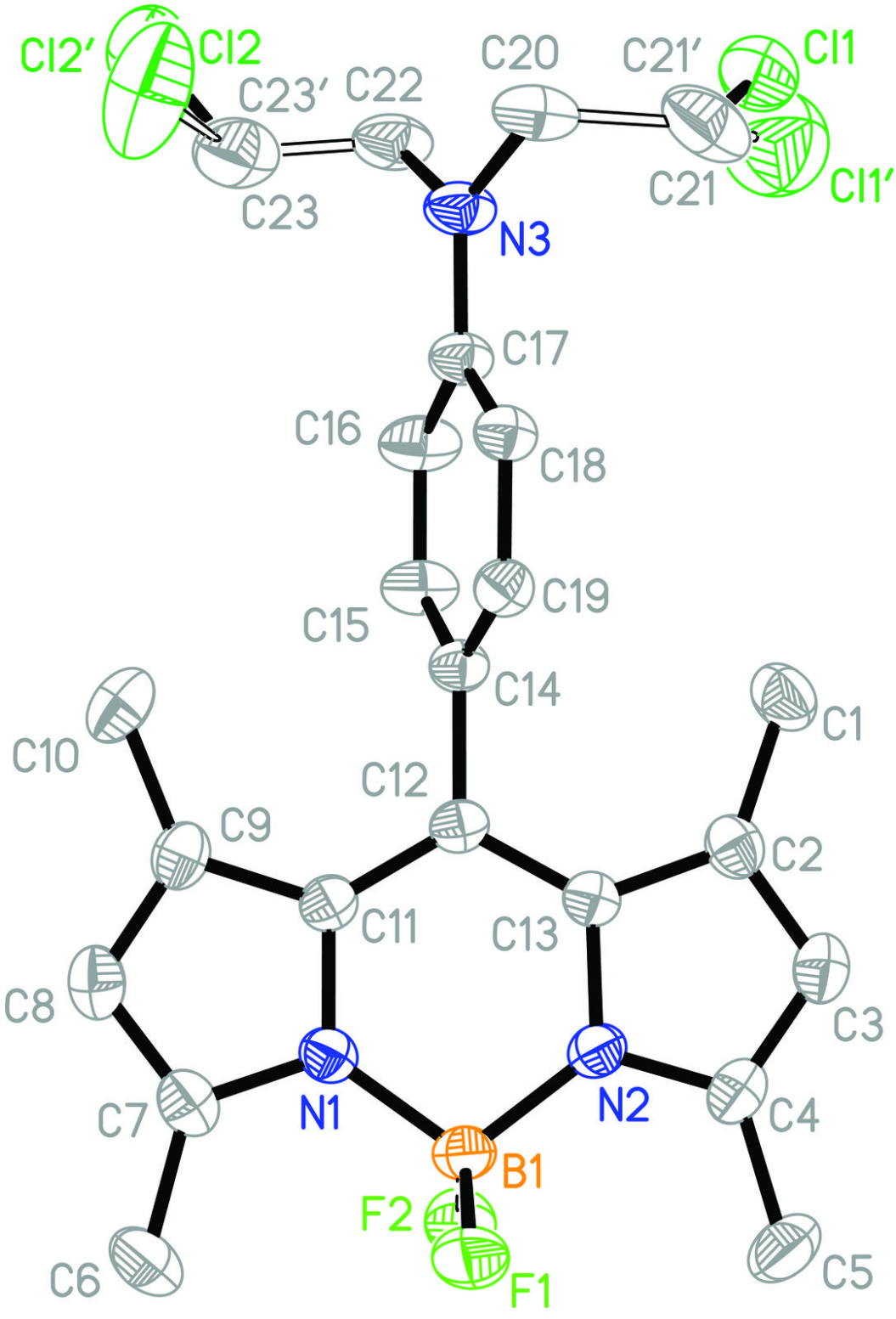
N1—C7	1.352 (2)	C9—C11	1.426 (3)
N1—C11	1.397 (2)	C9—C10	1.486 (3)
N1—B1	1.541 (3)	C10—H10A	0.9600
N2—C4	1.354 (2)	C10—H10B	0.9600
N2—C13	1.397 (2)	C10—H10C	0.9600
N2—B1	1.542 (3)	C11—C12	1.396 (2)
N3—C17	1.381 (2)	C12—C13	1.399 (2)

N3—C20	1.440 (3)	C12—C14	1.479 (2)
N3—C22	1.449 (3)	C14—C15	1.382 (2)
B1—F2	1.377 (3)	C14—C19	1.388 (2)
B1—F1	1.390 (3)	C15—C16	1.374 (3)
C1—C2	1.491 (3)	C15—H15	0.9300
C1—H1A	0.9600	C16—C17	1.398 (3)
C1—H1B	0.9600	C16—H16	0.9300
C1—H1C	0.9600	C17—C18	1.402 (2)
C2—C3	1.385 (3)	C18—C19	1.373 (2)
C2—C13	1.425 (3)	C18—H18	0.9300
C3—C4	1.381 (3)	C19—H19	0.9300
C3—H3	0.9300	C20—C21	1.521 (4)
C4—C5	1.500 (3)	C20—H20A	0.9700
C5—H5A	0.9600	C20—H20B	0.9700
C5—H5B	0.9600	C21—Cl1	1.771 (3)
C5—H5C	0.9600	C21—H21A	0.9700
C6—C7	1.485 (3)	C21—H21B	0.9700
C6—H6A	0.9600	C22—C23	1.490 (4)
C6—H6B	0.9600	C22—H22A	0.9700
C6—H6C	0.9600	C22—H22B	0.9700
C7—C8	1.385 (3)	C23—Cl2	1.738 (6)
C8—C9	1.385 (3)	C23—H23A	0.9700
C8—H8	0.9300	C23—H23B	0.9700
C7—N1—C11	108.32 (16)	C9—C10—H10C	109.5
C7—N1—B1	125.80 (16)	H10A—C10—H10C	109.5
C11—N1—B1	125.78 (15)	H10B—C10—H10C	109.5
C4—N2—C13	108.03 (16)	C12—C11—N1	120.37 (15)
C4—N2—B1	126.30 (16)	C12—C11—C9	131.70 (17)
C13—N2—B1	125.52 (15)	N1—C11—C9	107.89 (16)
C17—N3—C20	120.94 (16)	C11—C12—C13	120.92 (16)
C17—N3—C22	120.87 (17)	C11—C12—C14	120.12 (15)
C20—N3—C22	118.14 (17)	C13—C12—C14	118.92 (15)
F2—B1—F1	109.83 (17)	N2—C13—C12	120.45 (16)
F2—B1—N1	110.24 (17)	N2—C13—C2	108.03 (16)
F1—B1—N1	109.69 (17)	C12—C13—C2	131.52 (17)
F2—B1—N2	110.44 (17)	C15—C14—C19	117.18 (16)
F1—B1—N2	109.78 (17)	C15—C14—C12	122.32 (15)
N1—B1—N2	106.82 (15)	C19—C14—C12	120.50 (15)
C2—C1—H1A	109.5	C16—C15—C14	121.69 (17)
C2—C1—H1B	109.5	C16—C15—H15	119.2
H1A—C1—H1B	109.5	C14—C15—H15	119.2
C2—C1—H1C	109.5	C15—C16—C17	121.56 (17)
H1A—C1—H1C	109.5	C15—C16—H16	119.2
H1B—C1—H1C	109.5	C17—C16—H16	119.2
C3—C2—C13	105.50 (17)	N3—C17—C16	121.94 (16)
C3—C2—C1	125.10 (19)	N3—C17—C18	121.54 (16)
C13—C2—C1	129.27 (19)	C16—C17—C18	116.52 (16)
C4—C3—C2	109.38 (18)	C19—C18—C17	121.19 (16)
C4—C3—H3	125.3	C19—C18—H18	119.4

C2—C3—H3	125.3	C17—C18—H18	119.4
N2—C4—C3	109.04 (18)	C18—C19—C14	121.86 (15)
N2—C4—C5	122.6 (2)	C18—C19—H19	119.1
C3—C4—C5	128.32 (19)	C14—C19—H19	119.1
C4—C5—H5A	109.5	N3—C20—C21	113.8 (2)
C4—C5—H5B	109.5	N3—C20—H20A	108.8
H5A—C5—H5B	109.5	C21—C20—H20A	108.8
C4—C5—H5C	109.5	N3—C20—H20B	108.8
H5A—C5—H5C	109.5	C21—C20—H20B	108.8
H5B—C5—H5C	109.5	H20A—C20—H20B	107.7
C7—C6—H6A	109.5	C20—C21—Cl1	111.20 (19)
C7—C6—H6B	109.5	C20—C21—H21A	109.4
H6A—C6—H6B	109.5	Cl1—C21—H21A	109.4
C7—C6—H6C	109.5	C20—C21—H21B	109.4
H6A—C6—H6C	109.5	Cl1—C21—H21B	109.4
H6B—C6—H6C	109.5	H21A—C21—H21B	108.0
N1—C7—C8	108.81 (18)	N3—C22—C23	113.8 (2)
N1—C7—C6	123.2 (2)	N3—C22—H22A	108.8
C8—C7—C6	128.0 (2)	C23—C22—H22A	108.8
C9—C8—C7	109.43 (18)	N3—C22—H22B	108.8
C9—C8—H8	125.3	C23—C22—H22B	108.8
C7—C8—H8	125.3	H22A—C22—H22B	107.7
C8—C9—C11	105.54 (17)	C22—C23—Cl2	112.9 (2)
C8—C9—C10	124.60 (19)	C22—C23—H23A	109.0
C11—C9—C10	129.86 (19)	Cl2—C23—H23A	109.0
C9—C10—H10A	109.5	C22—C23—H23B	109.0
C9—C10—H10B	109.5	Cl2—C23—H23B	109.0
H10A—C10—H10B	109.5	H23A—C23—H23B	107.8
C7—N1—B1—F2	64.4 (2)	N1—C11—C12—C14	−177.88 (14)
C11—N1—B1—F2	−119.52 (19)	C9—C11—C12—C14	−0.7 (3)
C7—N1—B1—F1	−56.7 (2)	C4—N2—C13—C12	−179.44 (16)
C11—N1—B1—F1	119.42 (18)	B1—N2—C13—C12	4.9 (3)
C7—N1—B1—N2	−175.60 (16)	C4—N2—C13—C2	1.3 (2)
C11—N1—B1—N2	0.5 (2)	B1—N2—C13—C2	−174.41 (17)
C4—N2—B1—F2	−58.5 (3)	C11—C12—C13—N2	−2.8 (2)
C13—N2—B1—F2	116.38 (19)	C14—C12—C13—N2	175.01 (15)
C4—N2—B1—F1	62.7 (3)	C11—C12—C13—C2	176.29 (18)
C13—N2—B1—F1	−122.37 (19)	C14—C12—C13—C2	−5.9 (3)
C4—N2—B1—N1	−178.40 (17)	C3—C2—C13—N2	−1.4 (2)
C13—N2—B1—N1	−3.5 (2)	C1—C2—C13—N2	174.43 (19)
C13—C2—C3—C4	1.1 (2)	C3—C2—C13—C12	179.38 (18)
C1—C2—C3—C4	−175.0 (2)	C1—C2—C13—C12	−4.8 (3)
C13—N2—C4—C3	−0.6 (2)	C11—C12—C14—C15	−76.0 (2)
B1—N2—C4—C3	175.05 (18)	C13—C12—C14—C15	106.2 (2)
C13—N2—C4—C5	178.77 (19)	C11—C12—C14—C19	104.8 (2)
B1—N2—C4—C5	−5.6 (3)	C13—C12—C14—C19	−73.0 (2)
C2—C3—C4—N2	−0.3 (2)	C19—C14—C15—C16	−0.8 (3)
C2—C3—C4—C5	−179.7 (2)	C12—C14—C15—C16	180.0 (2)
C11—N1—C7—C8	0.2 (2)	C14—C15—C16—C17	0.4 (4)

B1—N1—C7—C8	176.89 (18)	C20—N3—C17—C16	−166.0 (2)
C11—N1—C7—C6	−178.47 (18)	C22—N3—C17—C16	11.4 (3)
B1—N1—C7—C6	−1.8 (3)	C20—N3—C17—C18	14.5 (3)
N1—C7—C8—C9	−0.5 (2)	C22—N3—C17—C18	−168.2 (2)
C6—C7—C8—C9	178.2 (2)	C15—C16—C17—N3	−179.2 (2)
C7—C8—C9—C11	0.5 (2)	C15—C16—C17—C18	0.3 (3)
C7—C8—C9—C10	−179.54 (19)	N3—C17—C18—C19	178.95 (18)
C7—N1—C11—C12	177.82 (16)	C16—C17—C18—C19	−0.6 (3)
B1—N1—C11—C12	1.2 (3)	C17—C18—C19—C14	0.1 (3)
C7—N1—C11—C9	0.07 (19)	C15—C14—C19—C18	0.5 (3)
B1—N1—C11—C9	−176.60 (17)	C12—C14—C19—C18	179.77 (16)
C8—C9—C11—C12	−177.74 (18)	C17—N3—C20—C21	69.8 (3)
C10—C9—C11—C12	2.3 (3)	C22—N3—C20—C21	−107.6 (2)
C8—C9—C11—N1	−0.3 (2)	N3—C20—C21—Cl1	61.2 (2)
C10—C9—C11—N1	179.69 (19)	C17—N3—C22—C23	69.9 (3)
N1—C11—C12—C13	−0.1 (2)	C20—N3—C22—C23	−112.6 (3)
C9—C11—C12—C13	177.05 (18)	N3—C22—C23—Cl2	67.2 (4)

Fig. 1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