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Redetermined structure of methyl 3-{4,4-difluoro-2-[2-(methoxycarbonyl)ethyl]-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacen-6-yl}propionate

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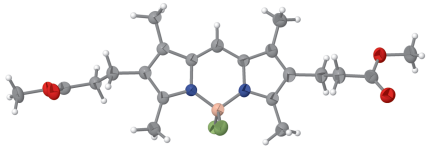
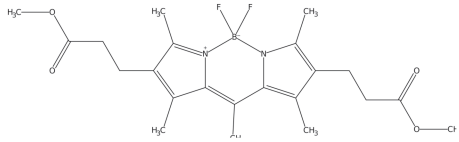
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{21}H_{27}BF_2N_2O_4$, a highly fluorescent boron–dipyrromethene dye, the methylpropionate moieties have different conformations. In the crystal, weak C—H···F and C—H···O interactions link the molecules. Some optical properties are presented.

3D view



Chemical scheme



Structure description

Fluorescent dyes are of great interest for labeling for analytics in biology and medicine (*e.g.*, Carpenter & Verkman, 2010; He *et al.*, 2003; Marfin *et al.*, 2017; Namkung *et al.*, 2009). Boron–dipyrromethene dyes (bodipy) show high quantum yields and excellent photostability and 9-aryl-substituted compounds have been the most investigated. The syntheses of these dyes usually consist of the condensation of pyrroles with aldehydes. To synthesize 9*H*-bodipy dyes, orthoformates have been used but here, dimethylformamide is the source of the central carbon atom in the title compound, $C_{21}H_{27}BF_2N_2O_4$ (**I**) (Fig. 1).

Despite the high formal symmetry of **I**, the molecule shows no inherent symmetry in its crystalline form. This is due to the methylpropionate moieties: the C17 branch adopts an all-*anti* conformation lying to one side of the π-system, while the C11 branch has an *s*–*cis* conformation on the other side of the π-system. The dihedral angles of these units with respect to the central fused-ring system are 84.3 (2) (C17 branch) and 74.6 (2)° (C11 branch). The 2,3,4-trisubstitution on the pyrrole rings enlarges the bond angles involving the methyl groups [C6—C5—C25 = 127.7 (4); C6—C7—C26 = 128.3 (4); C2—C1—C23 = 127.9 (4); C2—C3—C24 = 127.9 (4)°]. The near identical B4—N3*A* [1.537 (6) Å] and B4—N4*A* [1.535 (6) Å] bond lengths indicate the expected delocalization of charge (compare the chemical scheme).

In the extended structure of **I**, four molecules fill the unit cell, which are arranged in layers lying parallel to the *ac*-plane and weak C—H··· and C—H···O interactions link

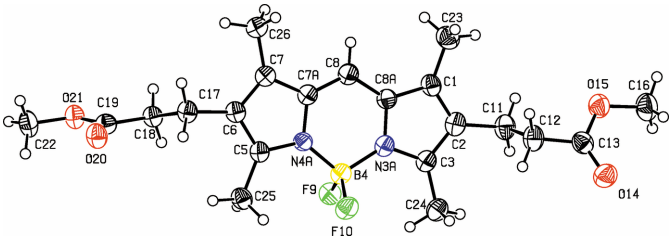


Figure 1
View of compound **I**. Displacement ellipsoids are drawn at the 50% probability level.

the molecules ((Fig. 2), Table 1). Within the plane a herring-bone pattern is formed and a twofold screw axis relates the molecular entities. These crystallographic results confirm recently reported deposited data (Uppal *et al.*, 2020).

Synthesis and crystallization

The compound was obtained as a side-product in the condensation of the pyrrole with a formylated cryptand (Jochem *et al.*, 2022). To the cryptand (97 mg), containing 5% dimethylformamide (4.9 mg, 0.066 mmol) in dry chloroform (10 ml) was added 3,5-dimethyl-4-(methoxycarbonyl)eth-2-ylpyrrole (65.0 mg, 0.359 mmol). Then, 10 µl of trifluoroacetic acid was added and stirred for 26 h. Diiso-propylethylamine (1 ml) was added followed by dichlorodicyanoquinone (54 mg) and stirred for 2 h. Afterwards, BF_3 diethyl ether solution (40%, 1 ml) was added dropwise and stirred. After complete addition, the mixture slowly turned red–violet and started fluorescing after about one h. After 20 h and addition of water (MilliQ, 20 ml), the organic phase was separated, washed with water and dried over Na_2CO_3. Purification *via* column chromatography (SiO_2/CH_2Cl_2) led to the title compound being eluated first: it crystallized from chloroform/2-propanol as a red solid (12.5 mg) and was recrystallized readily from the mixed solvents of acetonitrile and methanol. HR–ESI–MS: found: 421.2106 $[M + H]^+$, calculated 421.2105 for $C_{21}H_{27}BF_2N_2O_4^+$; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.98 (*s*, 1H), 3.67 (*s*, 6H), 2.71 (*dd*, *J* = 8.6, 6.9 Hz, 4H), 2.50 (*s*, 6H), 2.44 (*dd*, *J* = 8.6, 7.0 Hz, 4H), 2.19 (*s*, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 173.25, 155.23, 137.92, 132.64, 128.15, 119.38, 51.86, 34.27, 19.65, 12.80, 9.71. ^{19}F NMR (282 MHz, $CDCl_3$) δ = −146.29 (*dd*, *J* = 66.4, 33.2 Hz). Optical properties: the title compound has a high of solubility in a broad range of polar solvents but very limited solubility in toluene and alkanes. Bright sunlight led to photochemical decomposition only in very polar media whereas 10^{-5} *M* solutions in less polar solvents remained stable. The absorption spectra in CH_2Cl_2 shows a peak at 527 nm with emission at 536 nm: increasing solvent polarity provokes bathochromic shifts of max. 3 nm.

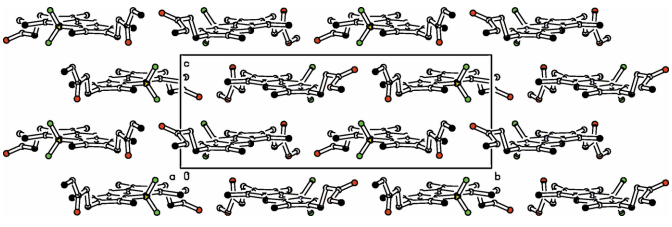


Figure 2
Partial packing diagram. View along the *a* axis.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*A*···F9^{i}	0.98	2.37	3.324 (7)	163
C18—H18*A*···O14^{ii}	0.99	2.57	3.268 (6)	128
C23—H23*B*···O20^{iii}	0.98	2.54	3.470 (7)	158

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+1, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{27}BF_2N_2O_4$
M_r	420.25
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, *b*, *c* (Å)	11.9299 (8), 21.6278 (17), 8.2665 (6)
β (°)	108.251 (6)
V (Å^3)	2025.6 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.20 × 0.10 × 0.04
Data collection	
Diffractometer	Stoe *IPDS* 2T
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2020)
T_{min}, T_{max}	0.985, 0.995
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9943, 4863, 2663
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.664
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.090, 0.238, 1.09
No. of reflections	4863
No. of parameters	277
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.48

Computer programs: *X-AREA WinXpose*, *Recipe* and *Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Shelxdrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2024). **9**, x240884 [https://doi.org/10.1107/S2414314624008848]

Redetermined structure of methyl 3-{4,4-difluoro-2-[2-(methoxycarbonyl)-ethyl]-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacen-6-yl}propionate

Dieter Schollmeyer, Matthias Jochen and Heiner Detert

Methyl 3-{4,4-difluoro-2-[2-(methoxycarbonyl)ethyl]-1,3,5,7-tetramethyl-4-bora-3a,4a-diaza-*s*-indacen-6-yl}propionate

Crystal data

$C_{21}H_{27}BF_2N_2O_4$
M_r = 420.25
Monoclinic, $P2_1/c$
a = 11.9299 (8) Å
b = 21.6278 (17) Å
c = 8.2665 (6) Å
β = 108.251 (6)°
V = 2025.6 (3) Å^3
Z = 4
$F(000)$ = 888
D_x = 1.378 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6884 reflections
θ = 2.6–28.3°
μ = 0.11 mm^{-1}
T = 120 K
Plate, red
0.20 × 0.10 × 0.04 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
Absorption correction: integration (XRED32; Stoe & Cie, 2020)
T_{min} = 0.985, T_{max} = 0.995
9943 measured reflections
4863 independent reflections
2663 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 28.2°, θ_{min} = 2.6°
h = −15→15
k = −26→28
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.090
$wR(F^2)$ = 0.238
S = 1.09
4863 reflections
277 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 5.4328P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

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

Refinement. Hydrogen atoms attached to carbon atoms were placed at calculated positions and were refined in the riding-model approximation with C—H = 0.95–0.99 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(methyl C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6168 (4)	0.1527 (2)	0.1730 (5)	0.0375 (10)
C2	0.6242 (4)	0.0886 (2)	0.1758 (5)	0.0401 (10)
N3A	0.4545 (3)	0.11329 (17)	0.2243 (4)	0.0357 (8)
C3	0.5240 (4)	0.0654 (2)	0.2098 (5)	0.0365 (10)
N4A	0.2907 (3)	0.17653 (16)	0.2632 (4)	0.0320 (8)
B4	0.3319 (5)	0.1102 (2)	0.2480 (6)	0.0345 (10)
C5	0.1906 (4)	0.1955 (2)	0.2927 (5)	0.0321 (9)
C6	0.1870 (4)	0.2612 (2)	0.2985 (5)	0.0339 (9)
C7	0.2882 (4)	0.2830 (2)	0.2691 (5)	0.0334 (9)
C7A	0.3531 (4)	0.2300 (2)	0.2478 (5)	0.0332 (9)
C8	0.4593 (4)	0.2254 (2)	0.2161 (5)	0.0378 (10)
H8	0.499101	0.262106	0.202465	0.045*
C8A	0.5107 (4)	0.1680 (2)	0.2034 (5)	0.0344 (9)
F9	0.2522 (2)	0.08108 (12)	0.1056 (3)	0.0419 (6)
F10	0.3365 (2)	0.07568 (12)	0.3926 (3)	0.0463 (7)
C11	0.7237 (4)	0.0497 (2)	0.1543 (6)	0.0447 (11)
H11A	0.765621	0.073310	0.088177	0.054*
H11B	0.690710	0.011961	0.089141	0.054*
C12	0.8112 (4)	0.0313 (2)	0.3255 (6)	0.0462 (12)
H12A	0.845614	0.069219	0.388989	0.055*
H12B	0.768455	0.009078	0.392899	0.055*
C13	0.9092 (4)	−0.0090 (2)	0.3081 (6)	0.0421 (11)
O14	0.9331 (3)	−0.05959 (17)	0.3704 (5)	0.0525 (9)
O15	0.9673 (3)	0.01777 (16)	0.2139 (4)	0.0482 (8)
C16	1.0658 (5)	−0.0171 (3)	0.1959 (7)	0.0571 (14)
H16A	1.112435	0.009131	0.144368	0.086*
H16B	1.036517	−0.053078	0.122765	0.086*
H16C	1.115511	−0.031056	0.308257	0.086*
C17	0.0914 (4)	0.2973 (2)	0.3389 (5)	0.0365 (10)
H17A	0.120908	0.339517	0.374704	0.044*
H17B	0.074036	0.277407	0.436257	0.044*
C18	−0.0232 (4)	0.3022 (2)	0.1904 (5)	0.0343 (9)
H18A	−0.009617	0.328626	0.100521	0.041*
H18B	−0.046000	0.260563	0.141415	0.041*
C19	−0.1220 (4)	0.3287 (2)	0.2435 (5)	0.0372 (10)
O20	−0.1238 (3)	0.33229 (16)	0.3882 (4)	0.0449 (8)
O21	−0.2120 (3)	0.34756 (15)	0.1088 (4)	0.0412 (7)

C22	−0.3121 (4)	0.3733 (3)	0.1478 (7)	0.0512 (13)
H22A	−0.376353	0.380654	0.041848	0.077*
H22B	−0.289255	0.412509	0.208816	0.077*
H22C	−0.338522	0.344308	0.219404	0.077*
C23	0.7046 (5)	0.1981 (2)	0.1491 (7)	0.0506 (12)
H23A	0.663003	0.233367	0.082459	0.076*
H23B	0.753553	0.178161	0.088545	0.076*
H23C	0.754893	0.212549	0.260557	0.076*
C24	0.4960 (4)	0.0000 (2)	0.2359 (6)	0.0420 (11)
H24A	0.523288	−0.026676	0.160100	0.063*
H24B	0.410510	−0.004618	0.210398	0.063*
H24C	0.535739	−0.011763	0.354578	0.063*
C25	0.0994 (4)	0.1511 (2)	0.3080 (6)	0.0399 (10)
H25A	0.045183	0.172126	0.357789	0.060*
H25B	0.137629	0.116608	0.381501	0.060*
H25C	0.055169	0.135340	0.194887	0.060*
C26	0.3262 (4)	0.3483 (2)	0.2625 (5)	0.0377 (10)
H26A	0.339927	0.355885	0.153316	0.057*
H26B	0.399334	0.355734	0.355804	0.057*
H26C	0.264443	0.376188	0.273969	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.039 (2)	0.046 (3)	0.031 (2)	0.002 (2)	0.0166 (19)	−0.0004 (19)
C2	0.043 (2)	0.045 (3)	0.032 (2)	0.006 (2)	0.0103 (19)	−0.0036 (19)
N3A	0.040 (2)	0.037 (2)	0.0308 (18)	0.0052 (16)	0.0126 (16)	0.0006 (15)
C3	0.042 (2)	0.038 (2)	0.031 (2)	0.004 (2)	0.0129 (18)	−0.0005 (18)
N4A	0.0363 (19)	0.0325 (19)	0.0314 (18)	0.0011 (15)	0.0164 (15)	0.0021 (14)
B4	0.042 (3)	0.038 (3)	0.025 (2)	0.003 (2)	0.013 (2)	0.0021 (19)
C5	0.035 (2)	0.039 (2)	0.0240 (19)	0.0021 (18)	0.0110 (17)	−0.0012 (16)
C6	0.038 (2)	0.041 (2)	0.0244 (19)	0.0009 (19)	0.0122 (17)	−0.0015 (17)
C7	0.038 (2)	0.037 (2)	0.025 (2)	0.0002 (18)	0.0088 (17)	−0.0012 (16)
C7A	0.039 (2)	0.036 (2)	0.025 (2)	0.0024 (19)	0.0123 (17)	−0.0001 (17)
C8	0.042 (2)	0.041 (3)	0.033 (2)	0.004 (2)	0.0153 (19)	0.0032 (19)
C8A	0.039 (2)	0.034 (2)	0.034 (2)	0.0003 (19)	0.0155 (18)	−0.0020 (17)
F9	0.0437 (14)	0.0432 (15)	0.0382 (14)	−0.0020 (12)	0.0123 (11)	−0.0047 (11)
F10	0.0596 (17)	0.0445 (16)	0.0405 (14)	0.0089 (13)	0.0238 (13)	0.0098 (12)
C11	0.044 (3)	0.050 (3)	0.041 (3)	0.005 (2)	0.014 (2)	−0.009 (2)
C12	0.045 (3)	0.056 (3)	0.038 (2)	0.009 (2)	0.014 (2)	−0.003 (2)
C13	0.036 (2)	0.050 (3)	0.038 (2)	−0.002 (2)	0.0085 (19)	0.002 (2)
O14	0.053 (2)	0.049 (2)	0.053 (2)	0.0036 (17)	0.0130 (17)	0.0062 (17)
O15	0.0476 (19)	0.051 (2)	0.052 (2)	0.0070 (16)	0.0231 (16)	0.0032 (16)
C16	0.053 (3)	0.067 (4)	0.057 (3)	0.012 (3)	0.025 (3)	0.002 (3)
C17	0.041 (2)	0.044 (3)	0.028 (2)	0.005 (2)	0.0146 (18)	−0.0009 (18)
C18	0.037 (2)	0.040 (2)	0.027 (2)	0.0010 (19)	0.0126 (17)	−0.0018 (17)
C19	0.043 (2)	0.039 (2)	0.033 (2)	−0.001 (2)	0.0158 (19)	−0.0009 (18)
O20	0.0460 (18)	0.058 (2)	0.0358 (17)	0.0070 (16)	0.0203 (14)	0.0003 (15)

O21	0.0360 (16)	0.051 (2)	0.0364 (16)	0.0061 (15)	0.0112 (13)	0.0013 (14)
C22	0.037 (2)	0.066 (3)	0.054 (3)	0.011 (2)	0.019 (2)	0.004 (3)
C23	0.050 (3)	0.053 (3)	0.055 (3)	−0.003 (2)	0.025 (2)	−0.003 (2)
C24	0.047 (3)	0.040 (3)	0.039 (2)	0.004 (2)	0.012 (2)	−0.001 (2)
C25	0.042 (2)	0.048 (3)	0.036 (2)	0.000 (2)	0.019 (2)	0.0010 (19)
C26	0.044 (2)	0.037 (2)	0.033 (2)	0.000 (2)	0.0123 (19)	−0.0003 (18)

Geometric parameters (Å, º)

C1—C2	1.389 (6)	C13—O15	1.326 (5)
C1—C8A	1.405 (6)	O15—C16	1.443 (6)
C1—C23	1.494 (6)	C16—H16A	0.9800
C2—C3	1.404 (6)	C16—H16B	0.9800
C2—C11	1.510 (6)	C16—H16C	0.9800
N3A—C3	1.356 (5)	C17—C18	1.528 (6)
N3A—C8A	1.396 (5)	C17—H17A	0.9900
N3A—B4	1.537 (6)	C17—H17B	0.9900
C3—C24	1.483 (6)	C18—C19	1.496 (6)
N4A—C5	1.355 (5)	C18—H18A	0.9900
N4A—C7A	1.403 (5)	C18—H18B	0.9900
N4A—B4	1.535 (6)	C19—O20	1.205 (5)
B4—F10	1.395 (5)	C19—O21	1.345 (5)
B4—F9	1.409 (5)	O21—C22	1.442 (5)
C5—C6	1.424 (6)	C22—H22A	0.9800
C5—C25	1.486 (6)	C22—H22B	0.9800
C6—C7	1.386 (6)	C22—H22C	0.9800
C6—C17	1.504 (6)	C23—H23A	0.9800
C7—C7A	1.424 (6)	C23—H23B	0.9800
C7—C26	1.490 (6)	C23—H23C	0.9800
C7A—C8	1.375 (6)	C24—H24A	0.9800
C8—C8A	1.403 (6)	C24—H24B	0.9800
C8—H8	0.9500	C24—H24C	0.9800
C11—C12	1.525 (7)	C25—H25A	0.9800
C11—H11A	0.9900	C25—H25B	0.9800
C11—H11B	0.9900	C25—H25C	0.9800
C12—C13	1.501 (6)	C26—H26A	0.9800
C12—H12A	0.9900	C26—H26B	0.9800
C12—H12B	0.9900	C26—H26C	0.9800
C13—O14	1.205 (6)		

C2—C1—C8A	106.8 (4)	C13—O15—C16	115.1 (4)
C2—C1—C23	127.9 (4)	O15—C16—H16A	109.5
C8A—C1—C23	125.3 (4)	O15—C16—H16B	109.5
C1—C2—C3	107.8 (4)	H16A—C16—H16B	109.5
C1—C2—C11	127.0 (4)	O15—C16—H16C	109.5
C3—C2—C11	125.1 (4)	H16A—C16—H16C	109.5
C3—N3A—C8A	107.8 (3)	H16B—C16—H16C	109.5
C3—N3A—B4	127.6 (4)	C6—C17—C18	114.1 (3)

C8A—N3A—B4	124.4 (4)	C6—C17—H17A	108.7
N3A—C3—C2	109.1 (4)	C18—C17—H17A	108.7
N3A—C3—C24	122.9 (4)	C6—C17—H17B	108.7
C2—C3—C24	127.9 (4)	C18—C17—H17B	108.7
C5—N4A—C7A	106.8 (3)	H17A—C17—H17B	107.6
C5—N4A—B4	128.4 (4)	C19—C18—C17	112.3 (3)
C7A—N4A—B4	124.8 (3)	C19—C18—H18A	109.1
F10—B4—F9	108.2 (4)	C17—C18—H18A	109.1
F10—B4—N4A	111.0 (3)	C19—C18—H18B	109.1
F9—B4—N4A	109.7 (4)	C17—C18—H18B	109.1
F10—B4—N3A	110.4 (4)	H18A—C18—H18B	107.9
F9—B4—N3A	109.4 (3)	O20—C19—O21	123.0 (4)
N4A—B4—N3A	108.2 (4)	O20—C19—C18	125.2 (4)
N4A—C5—C6	110.2 (4)	O21—C19—C18	111.8 (3)
N4A—C5—C25	122.0 (4)	C19—O21—C22	115.7 (3)
C6—C5—C25	127.7 (4)	O21—C22—H22A	109.5
C7—C6—C5	107.2 (4)	O21—C22—H22B	109.5
C7—C6—C17	128.7 (4)	H22A—C22—H22B	109.5
C5—C6—C17	123.9 (4)	O21—C22—H22C	109.5
C6—C7—C7A	106.6 (4)	H22A—C22—H22C	109.5
C6—C7—C26	128.3 (4)	H22B—C22—H22C	109.5
C7A—C7—C26	125.0 (4)	C1—C23—H23A	109.5
C8—C7A—N4A	120.2 (4)	C1—C23—H23B	109.5
C8—C7A—C7	130.7 (4)	H23A—C23—H23B	109.5
N4A—C7A—C7	109.1 (3)	C1—C23—H23C	109.5
C7A—C8—C8A	121.9 (4)	H23A—C23—H23C	109.5
C7A—C8—H8	119.0	H23B—C23—H23C	109.5
C8A—C8—H8	119.0	C3—C24—H24A	109.5
N3A—C8A—C8	120.2 (4)	C3—C24—H24B	109.5
N3A—C8A—C1	108.5 (4)	H24A—C24—H24B	109.5
C8—C8A—C1	131.3 (4)	C3—C24—H24C	109.5
C2—C11—C12	111.8 (4)	H24A—C24—H24C	109.5
C2—C11—H11A	109.3	H24B—C24—H24C	109.5
C12—C11—H11A	109.3	C5—C25—H25A	109.5
C2—C11—H11B	109.3	C5—C25—H25B	109.5
C12—C11—H11B	109.3	H25A—C25—H25B	109.5
H11A—C11—H11B	107.9	C5—C25—H25C	109.5
C13—C12—C11	112.9 (4)	H25A—C25—H25C	109.5
C13—C12—H12A	109.0	H25B—C25—H25C	109.5
C11—C12—H12A	109.0	C7—C26—H26A	109.5
C13—C12—H12B	109.0	C7—C26—H26B	109.5
C11—C12—H12B	109.0	H26A—C26—H26B	109.5
H12A—C12—H12B	107.8	C7—C26—H26C	109.5
O14—C13—O15	123.4 (4)	H26A—C26—H26C	109.5
O14—C13—C12	125.0 (4)	H26B—C26—H26C	109.5
O15—C13—C12	111.6 (4)		

C8A—C1—C2—C3	−0.8 (5)	C17—C6—C7—C26	−2.9 (7)

C23—C1—C2—C3	177.2 (4)	C5—N4A—C7A—C8	−179.6 (4)
C8A—C1—C2—C11	−178.2 (4)	B4—N4A—C7A—C8	0.2 (6)
C23—C1—C2—C11	−0.2 (8)	C5—N4A—C7A—C7	0.1 (4)
C8A—N3A—C3—C2	−1.3 (5)	B4—N4A—C7A—C7	180.0 (4)
B4—N3A—C3—C2	175.1 (4)	C6—C7—C7A—C8	−179.9 (4)
C8A—N3A—C3—C24	176.0 (4)	C26—C7—C7A—C8	−0.7 (7)
B4—N3A—C3—C24	−7.6 (7)	C6—C7—C7A—N4A	0.4 (4)
C1—C2—C3—N3A	1.3 (5)	C26—C7—C7A—N4A	179.6 (4)
C11—C2—C3—N3A	178.8 (4)	N4A—C7A—C8—C8A	−2.0 (6)
C1—C2—C3—C24	−175.8 (4)	C7—C7A—C8—C8A	178.3 (4)
C11—C2—C3—C24	1.7 (7)	C3—N3A—C8A—C8	−178.6 (4)
C5—N4A—B4—F10	−55.6 (6)	B4—N3A—C8A—C8	4.9 (6)
C7A—N4A—B4—F10	124.6 (4)	C3—N3A—C8A—C1	0.8 (5)
C5—N4A—B4—F9	63.9 (5)	B4—N3A—C8A—C1	−175.8 (4)
C7A—N4A—B4—F9	−116.0 (4)	C7A—C8—C8A—N3A	−0.5 (6)
C5—N4A—B4—N3A	−176.9 (4)	C7A—C8—C8A—C1	−179.7 (4)
C7A—N4A—B4—N3A	3.3 (5)	C2—C1—C8A—N3A	0.0 (5)
C3—N3A—B4—F10	56.7 (6)	C23—C1—C8A—N3A	−178.1 (4)
C8A—N3A—B4—F10	−127.4 (4)	C2—C1—C8A—C8	179.3 (5)
C3—N3A—B4—F9	−62.3 (5)	C23—C1—C8A—C8	1.2 (8)
C8A—N3A—B4—F9	113.6 (4)	C1—C2—C11—C12	95.7 (6)
C3—N3A—B4—N4A	178.3 (4)	C3—C2—C11—C12	−81.3 (6)
C8A—N3A—B4—N4A	−5.8 (5)	C2—C11—C12—C13	178.2 (4)
C7A—N4A—C5—C6	−0.6 (5)	C11—C12—C13—O14	−123.1 (5)
B4—N4A—C5—C6	179.6 (4)	C11—C12—C13—O15	56.6 (6)
C7A—N4A—C5—C25	177.1 (4)	O14—C13—O15—C16	−2.4 (7)
B4—N4A—C5—C25	−2.8 (6)	C12—C13—O15—C16	177.9 (4)
N4A—C5—C6—C7	0.8 (5)	C7—C6—C17—C18	105.4 (5)
C25—C5—C6—C7	−176.6 (4)	C5—C6—C17—C18	−78.1 (5)
N4A—C5—C6—C17	−176.4 (3)	C6—C17—C18—C19	170.2 (4)
C25—C5—C6—C17	6.2 (7)	C17—C18—C19—O20	−17.2 (6)
C5—C6—C7—C7A	−0.7 (5)	C17—C18—C19—O21	164.1 (4)
C17—C6—C7—C7A	176.3 (4)	O20—C19—O21—C22	0.7 (7)
C5—C6—C7—C26	−179.8 (4)	C18—C19—O21—C22	179.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*A*···F9^{i}	0.98	2.37	3.324 (7)	163
C18—H18*A*···O14^{ii}	0.99	2.57	3.268 (6)	128
C23—H23*B*···O20^{iii}	0.98	2.54	3.470 (7)	158

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) *x*+1, −*y*+1/2, *z*−1/2.