Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethyl 2-*tert*-butyl-6,9-dibromo-4,11-dioxo-5,10-dihydro-*cis*-1*H*,3*H*,4*H*,11*H*-2-azo-3a,4a,10a,11a-tetraazabenz[*f*]-indeno[2,1,7-*ija*]azulene-11b,11c-dicarboxylate

Jing Qin,* Hui-Zhen Guo and Li-Ping Cao

Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: qj0312@mails.ccnu.edu.cn

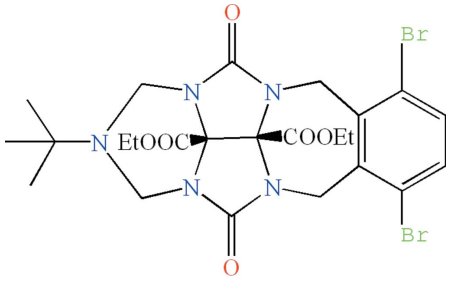
Received 20 October 2007; accepted 23 November 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; disorder in main residue; R factor = 0.080; wR factor = 0.232; data-to-parameter ratio = 11.6.

In the title compound, $C_{24}H_{29}Br_2N_5O_6$, a glycoluril derivative, the 1,4-dibromobenzene ring is fused to the seven-membered ring of the glycoluril unit containing two N atoms. The two five-membered rings in the glycoluril unit are approximately planar and the dihedral angle between them is 69.8 (2)°. The six-membered ring containing three N atoms adopts a chair conformation. The crystal packing is stabilized by an intermolecular non-classical C—H···O hydrogen bond and a weak C—H···π interaction. Both of the ester groups are found to be disordered over two positions. The occupancies of the disordered positions were refined to 0.73(1):0.27(1) and 0.56(1):0.44(1).

Related literature

For the preparation of the title compound, see: Lai & Yap (1993); Li *et al.* (2006). For potential applications of the title compound, see: Wu *et al.* (2002); Rowan *et al.* (1999); Chakraborty *et al.* (2002); Lehn (1995); Diederich (1991); Purse & Rebek (2005); Yin *et al.* (2006). For the crystal structures of similar compounds, see: Chen *et al.* (2007); Wang *et al.* (2006).



Experimental

Crystal data

$C_{24}H_{29}Br_2N_5O_6$
M_r = 643.34
Monoclinic, $P2_1/n$
a = 10.4151 (8) Å
b = 11.3801 (8) Å
c = 23.1649 (17) Å
β = 92.692 (1)°
V = 2742.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.00 mm^{-1}
T = 293 (2) K
0.10 × 0.06 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
18064 measured reflections
4828 independent reflections
2185 reflections with $I > 2\sigma(I)$
R_{int} = 0.120

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.232
S = 0.88
4828 reflections
415 parameters
22 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···Br2	0.97	2.59	3.190 (8)	120
C7—H7B···Br1	0.97	2.61	3.206 (7)	120
C7—H7A···O3^{i}	0.97	2.55	3.125 (17)	118
C14—H14C···$Cg1^{ii}$	0.97	2.97	3.89 (4)	160

Symmetry codes: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$. $Cg1$ is the centroid of the benzene C1–C6 ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

The authors thank Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2227).

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supporting information

Acta Cryst. (2008). E**64**, o37–o38 [https://doi.org/10.1107/S1600536807062472]

Diethyl 2-*tert*-butyl-6,9-dibromo-4,11-dioxo-5,10-dihydro-*cis*-1*H*,3*H*,4*H*,11*H*-2-azo-3a,4a,10a,11a-tetraazabenz[*f*]indeno[2,1,7-*ija*]azulene-11b,11c-dicarboxylate

Jing Qin, Hui-Zhen Guo and Li-Ping Cao

S1. Comment

Glycoluril derivatives have shown applications in many fields such as polymer cross-linking, explosives, slow-release fertilizers, iodogens stabilisers of organic compounds against photodegradation and reagent in combinatorial chemistry (Wu *et al.*, 2002; Rowan *et al.*, 1999; Chakraborty *et al.*, 2002). Synthetic receptors with molecular cavities have been used as models and play an important role in studying the complex biological systems, such as protein folding, molecular recognition of substrates by enzymes, formation of membranes (Lehn, 1995; Diederich, 1991) and molecular catalysis (Purse & Rebek, 2005; Yin *et al.,* 2006; Li *et al.*, 2006). In addition, the crystal structures of glycoluril derivatives have been studied by our group (Chen *et al.*, 2007; Wang *et al.*, 2006). Herein we report the crystal structures of title compound (Fig. 1).

The dihedral angel between two five-membered rings in glycoluril unit is 69.8 (2)°. The six-membered ring of N2/C15/N3/C20/N5/C19 displays a chair conformation. By a combination of a non-classical H-bonding, *e.g.* C7—H7A···O3^{i} [symmetry code: (i) 5/2 - *x*, 1/2 + *y*, 1/2 - *z*] and a weak C—H···π interaction (Table 1; *Cg*1 is the centroid of the benzene C1–C6 ring), molecules are joined together forming a one-dimensional chain running parallel to the *b* axis (Fig. 2).

S2. Experimental

The title compound was synthesized according to the procedure of Lai & Yap (1993) and Li *et al.* (2006). Single crystals suitable for X-ray data collection were obtained by slow evaporation of a chloroform and methaol solution in a ratio of 20:1 at 293 K.

S3. Refinement

Each diethoxycarbonyl group was treated as disordered over two positions. For the ester group containing O3/O4, the occupation factors of the two sites were refined by using restraint commands of *DFIX*, *SADI* and *EDAP*, giving the final occupancy of 0.73 (1)/0.27 (1) for the major and minor components, respectively. Similarly, the ester group containing O5/O6 was also treated as disorder and the final occupancy were 0.56 (1)/0.44 (1) for the corresponding components, respectively. The final largest residual density peak is located about 1.51 (1) Å from atom H5. Attempted to model the probable disorder or any other solvent molecules failed, which may be caused by the existence of the two heavy atoms, Br1 and Br2, leading to the contamination some positions. The large R_{int} value is probably due to the poor crystal quality; we tried to selected better crystals for diffraction experiment, but failed. All H atoms bonded to C atoms were located at the geometrical positions with C—H = 0.96 Å (methyl C), 0.97 Å (methylene C) and 0.93Å (aromatic C), and the U_{iso}(H) values were set k times of U_{eq}(C), k = 1.5 for methyl H atom and 1.2 for the other H atoms.

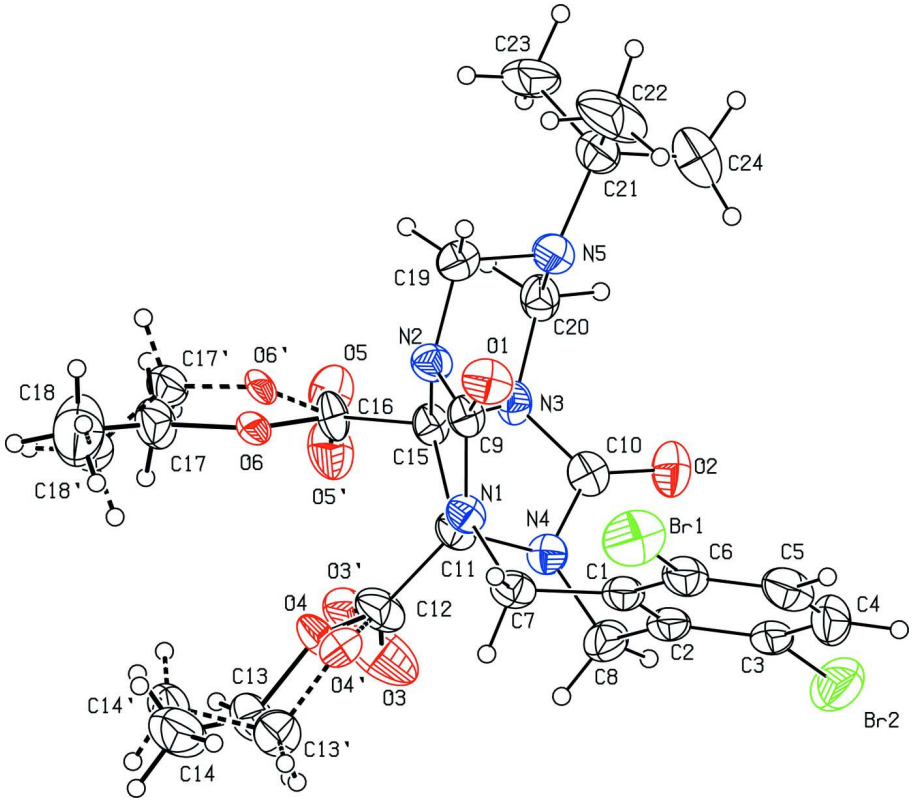

Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 10% probability level. H atoms are represented by spheres of arbitrary radius. Bonds to atoms of the minor disorder components are drawn as dashed lines.

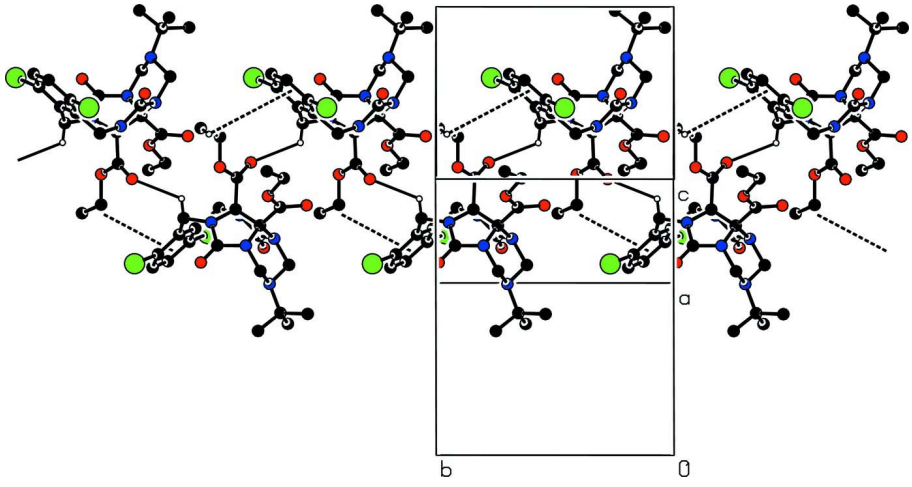

Figure 2

Part of the crystal packing showing the formation of one-dimensional chain running parallel to the *b* axis by the C—H···O and C—H···π interactions. H atoms not involved in the motif have been omitted for clarity.

Diethyl 2-*tert*-butyl-6,9-dibromo-4,11-dioxo-5,10-dihydro-*cis*-1H,3*H*,4H,11*H*- 2-azo-3a,4a,10*a*,11*a*-tetraazabenz[*f*]indeno[2,1,7-ija]azulene-11*b*,11*c*- dicarboxylate

Crystal data

$C_{24}H_{29}Br_2N_5O_6$
$M_r = 643.34$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.4151$ (8) Å
$b = 11.3801$ (8) Å
$c = 23.1649$ (17) Å
$\beta = 92.692$ (1)°
$V = 2742.6$ (3) Å^3
$Z = 4$
$F(000) = 1304$
$D_x = 1.558$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2626 reflections
$\theta = 2.2$–18.8°
$\mu = 3.00$ mm^{-1}
$T = 293$ K
Block, blue
0.10 × 0.06 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
18064 measured reflections
4828 independent reflections
2185 reflections with $I > 2\sigma(I)$
$R_{int} = 0.120$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 11$
$l = -27 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.232$
$S = 0.88$
4828 reflections
415 parameters
22 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1377P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.91$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Special details

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 $F^2 > \sigma(F^2)$ 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)
Br1	0.87255 (10)	1.26953 (9)	0.14865 (5)	0.0940 (5)	
Br2	1.27715 (11)	0.96144 (11)	0.00489 (5)	0.1022 (5)	
C1	1.0559 (7)	1.0823 (7)	0.1398 (3)	0.0510 (19)	
C2	1.1426 (7)	1.0152 (7)	0.1078 (4)	0.056 (2)	

C3	1.1591 (8)	1.0401 (8)	0.0494 (4)	0.070 (3)	
C4	1.0897 (10)	1.1305 (9)	0.0219 (4)	0.077 (3)	
H4	1.1011	1.1472	−0.0168	0.093*	
C5	1.0050 (9)	1.1941 (8)	0.0523 (5)	0.078 (3)	
H5	0.9577	1.2537	0.0338	0.093*	
C6	0.9880 (7)	1.1713 (7)	0.1106 (4)	0.062 (2)	
C7	1.0491 (7)	1.0630 (6)	0.2031 (3)	0.0517 (19)	
H7A	1.1346	1.0725	0.2209	0.062*	
H7B	0.9948	1.1234	0.2187	0.062*	
C8	1.2156 (7)	0.9152 (8)	0.1365 (4)	0.063 (2)	
H8A	1.2672	0.8770	0.1082	0.075*	
H8B	1.2738	0.9466	0.1666	0.075*	
C9	0.8717 (7)	0.9214 (6)	0.2150 (3)	0.0457 (18)	
C10	1.0663 (7)	0.7450 (6)	0.1288 (4)	0.051 (2)	
C11	1.0791 (7)	0.8450 (6)	0.2169 (3)	0.0475 (18)	
C12	1.1894 (8)	0.8394 (8)	0.2628 (4)	0.068 (2)	
C13	1.2920 (14)	0.9101 (19)	0.3468 (8)	0.074 (5)	0.73 (3)
H13A	1.3671	0.9391	0.3280	0.089*	0.73 (3)
H13B	1.3097	0.8309	0.3605	0.089*	0.73 (3)
C14	1.261 (2)	0.991 (2)	0.3979 (8)	0.107 (8)	0.73 (3)
H14A	1.2380	1.0674	0.3835	0.160*	0.73 (3)
H14B	1.3347	0.9965	0.4241	0.160*	0.73 (3)
H14C	1.1901	0.9582	0.4177	0.160*	0.73 (3)
O3	1.2862 (18)	0.7808 (19)	0.2575 (12)	0.124 (8)	0.73 (3)
O4	1.1805 (17)	0.9107 (18)	0.3066 (7)	0.066 (6)	0.73 (3)
C13′	1.281 (4)	0.976 (5)	0.3379 (12)	0.065 (13)	0.27 (3)
H13C	1.2783	1.0614	0.3412	0.077*	0.27 (3)
H13D	1.3659	0.9549	0.3254	0.077*	0.27 (3)
C14′	1.260 (4)	0.921 (4)	0.3968 (16)	0.061 (13)	0.27 (3)
H14D	1.2240	0.8438	0.3914	0.092*	0.27 (3)
H14E	1.2020	0.9689	0.4176	0.092*	0.27 (3)
H14F	1.3407	0.9152	0.4185	0.092*	0.27 (3)
O3′	1.240 (4)	0.745 (2)	0.274 (2)	0.070 (11)	0.27 (3)
O4′	1.185 (6)	0.938 (3)	0.2953 (17)	0.067 (16)	0.27 (3)
C15	0.9798 (7)	0.7420 (6)	0.2205 (3)	0.0448 (18)	
C16	1.0060 (8)	0.6547 (6)	0.2707 (3)	0.058 (2)	
C19	0.7462 (7)	0.7404 (7)	0.2009 (3)	0.054 (2)	
H19A	0.6720	0.7920	0.2010	0.065*	
H19B	0.7292	0.6727	0.2248	0.065*	
C20	0.8754 (7)	0.6202 (7)	0.1429 (3)	0.058 (2)	
H20A	0.8892	0.5907	0.1044	0.070*	
H20B	0.8573	0.5539	0.1676	0.070*	
C21	0.6505 (8)	0.6557 (8)	0.1086 (4)	0.070 (2)	
C22	0.5477 (10)	0.7499 (11)	0.1080 (6)	0.124 (5)	
H22A	0.5338	0.7744	0.1468	0.186*	
H22B	0.4691	0.7190	0.0907	0.186*	
H22C	0.5749	0.8160	0.0859	0.186*	
C23	0.6001 (10)	0.5414 (9)	0.1369 (5)	0.106 (4)	

H23A	0.6650	0.4816	0.1361	0.159*	
H23B	0.5241	0.5149	0.1157	0.159*	
H23C	0.5802	0.5572	0.1762	0.159*	
C24	0.6860 (12)	0.6247 (13)	0.0476 (4)	0.127 (5)	
H24A	0.7421	0.6840	0.0334	0.191*	
H24B	0.6094	0.6206	0.0229	0.191*	
H24C	0.7288	0.5500	0.0478	0.191*	
N1	0.9991 (5)	0.9471 (5)	0.2194 (2)	0.0448 (14)	
N2	0.8576 (5)	0.8014 (5)	0.2239 (3)	0.0495 (15)	
N3	0.9906 (6)	0.6825 (5)	0.1656 (2)	0.0486 (15)	
N4	1.1327 (5)	0.8276 (5)	0.1619 (3)	0.0484 (15)	
N5	0.7677 (6)	0.7018 (5)	0.1411 (3)	0.0511 (16)	
O1	0.7850 (5)	0.9907 (4)	0.2079 (2)	0.0591 (14)	
O2	1.0786 (6)	0.7265 (5)	0.0781 (3)	0.0711 (16)	
O5	1.030 (3)	0.5476 (9)	0.2619 (7)	0.064 (5)	0.57 (4)
O6	0.986 (2)	0.7074 (12)	0.3223 (4)	0.042 (4)	0.57 (4)
C17	1.006 (4)	0.641 (2)	0.3743 (5)	0.070 (8)	0.57 (4)
H17A	1.0955	0.6186	0.3793	0.084*	0.57 (4)
H17B	0.9547	0.5695	0.3717	0.084*	0.57 (4)
C18	0.967 (5)	0.714 (4)	0.4262 (7)	0.134 (19)	0.57 (4)
H18A	1.0018	0.7915	0.4235	0.201*	0.57 (4)
H18B	0.9995	0.6772	0.4612	0.201*	0.57 (4)
H18C	0.8748	0.7181	0.4264	0.201*	0.57 (4)
O6′	0.937 (2)	0.689 (2)	0.3156 (8)	0.044 (6)	0.43 (4)
O5′	1.095 (3)	0.581 (3)	0.2663 (10)	0.086 (9)	0.43 (4)
C17′	0.963 (4)	0.622 (3)	0.3649 (11)	0.059 (11)	0.43 (4)
H17C	1.0296	0.5654	0.3572	0.071*	0.43 (4)
H17D	0.8861	0.5783	0.3739	0.071*	0.43 (4)
C18′	1.006 (4)	0.697 (3)	0.4175 (12)	0.058 (9)	0.43 (4)
H18D	1.0815	0.7407	0.4088	0.086*	0.43 (4)
H18E	1.0249	0.6468	0.4501	0.086*	0.43 (4)
H18F	0.9384	0.7505	0.4264	0.086*	0.43 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0846 (8)	0.0613 (6)	0.1359 (11)	0.0207 (5)	0.0027 (6)	0.0263 (6)
Br2	0.1050 (9)	0.1171 (10)	0.0874 (8)	−0.0272 (7)	0.0366 (6)	−0.0131 (6)
C1	0.044 (4)	0.050 (5)	0.058 (5)	−0.010 (4)	−0.004 (4)	0.004 (4)
C2	0.048 (4)	0.055 (5)	0.063 (6)	−0.018 (4)	−0.005 (4)	0.007 (4)
C3	0.068 (6)	0.078 (6)	0.063 (6)	−0.042 (5)	−0.012 (5)	0.015 (5)
C4	0.098 (8)	0.079 (7)	0.054 (6)	−0.016 (6)	−0.008 (5)	0.017 (5)
C5	0.080 (7)	0.055 (6)	0.095 (8)	−0.016 (5)	−0.037 (6)	0.029 (6)
C6	0.057 (5)	0.056 (5)	0.073 (6)	−0.007 (4)	−0.014 (4)	0.016 (5)
C7	0.052 (5)	0.038 (4)	0.065 (5)	−0.004 (3)	−0.006 (4)	−0.001 (4)
C8	0.046 (5)	0.077 (6)	0.066 (5)	0.001 (4)	0.010 (4)	0.009 (5)
C9	0.052 (5)	0.040 (5)	0.044 (5)	0.008 (4)	−0.003 (4)	0.003 (3)
C10	0.055 (5)	0.041 (5)	0.055 (6)	0.014 (4)	−0.002 (4)	0.000 (4)

C11	0.051 (4)	0.038 (4)	0.053 (5)	0.001 (3)	−0.012 (4)	0.004 (4)
C12	0.058 (6)	0.067 (7)	0.078 (8)	0.006 (5)	−0.026 (5)	0.007 (6)
C13	0.066 (9)	0.087 (13)	0.066 (12)	0.006 (9)	−0.021 (8)	0.019 (11)
C14	0.112 (14)	0.102 (18)	0.104 (17)	−0.035 (15)	−0.031 (11)	−0.003 (13)
O3	0.094 (12)	0.121 (13)	0.15 (2)	0.043 (11)	−0.054 (11)	−0.037 (11)
O4	0.056 (9)	0.090 (9)	0.050 (7)	−0.002 (8)	−0.023 (7)	0.001 (9)
C13′	0.07 (2)	0.06 (3)	0.07 (3)	0.00 (2)	0.00 (2)	0.00 (2)
C14′	0.06 (2)	0.07 (3)	0.05 (3)	0.00 (2)	−0.001 (18)	−0.01 (2)
O3′	0.06 (2)	0.07 (2)	0.08 (2)	0.010 (16)	−0.020 (16)	0.016 (15)
O4′	0.06 (2)	0.08 (3)	0.06 (3)	−0.022 (17)	0.009 (16)	0.01 (2)
C15	0.057 (5)	0.033 (4)	0.044 (4)	0.005 (3)	−0.005 (4)	0.006 (3)
C16	0.071 (6)	0.064 (6)	0.037 (5)	0.012 (5)	−0.014 (4)	0.015 (4)
C19	0.050 (5)	0.053 (5)	0.061 (5)	0.000 (4)	0.006 (4)	0.004 (4)
C20	0.078 (6)	0.051 (5)	0.046 (5)	−0.005 (4)	0.001 (4)	−0.003 (4)
C21	0.076 (6)	0.071 (6)	0.062 (6)	−0.019 (5)	−0.013 (5)	0.008 (5)
C22	0.089 (8)	0.112 (9)	0.163 (12)	0.003 (7)	−0.075 (8)	0.024 (8)
C23	0.097 (8)	0.103 (8)	0.115 (8)	−0.063 (6)	−0.026 (6)	0.012 (7)
C24	0.130 (10)	0.180 (13)	0.069 (8)	−0.028 (9)	−0.025 (7)	−0.005 (8)
N1	0.043 (4)	0.041 (4)	0.049 (4)	0.007 (3)	−0.005 (3)	0.002 (3)
N2	0.041 (3)	0.053 (4)	0.054 (4)	0.003 (3)	−0.003 (3)	0.002 (3)
N3	0.065 (4)	0.043 (3)	0.039 (3)	0.000 (3)	0.001 (3)	0.004 (3)
N4	0.048 (4)	0.048 (4)	0.050 (4)	0.004 (3)	0.006 (3)	−0.004 (3)
N5	0.056 (4)	0.038 (4)	0.058 (4)	−0.002 (3)	−0.009 (3)	0.007 (3)
O1	0.054 (3)	0.041 (3)	0.082 (4)	0.018 (3)	−0.006 (3)	0.004 (3)
O2	0.086 (4)	0.081 (4)	0.047 (4)	0.016 (3)	0.011 (3)	0.002 (3)
O5	0.082 (13)	0.040 (7)	0.070 (8)	0.018 (6)	0.008 (8)	0.017 (6)
O6	0.035 (9)	0.050 (7)	0.040 (7)	−0.003 (6)	−0.011 (5)	0.009 (5)
C17	0.08 (2)	0.071 (16)	0.058 (16)	0.005 (14)	−0.015 (12)	0.016 (14)
C18	0.14 (4)	0.17 (4)	0.09 (2)	0.02 (2)	0.013 (19)	0.03 (2)
O6′	0.042 (11)	0.054 (10)	0.034 (9)	−0.006 (9)	−0.020 (8)	0.013 (7)
O5′	0.096 (17)	0.089 (16)	0.073 (12)	0.027 (16)	−0.017 (13)	0.027 (11)
C17′	0.07 (2)	0.058 (16)	0.054 (17)	−0.017 (14)	−0.011 (13)	0.004 (13)
C18′	0.058 (17)	0.058 (17)	0.06 (2)	0.000 (13)	0.003 (13)	0.000 (15)

Geometric parameters (Å, º)

Br1—C6	1.890 (9)	C14′—H14E	0.9600
Br2—C3	1.869 (10)	C14′—H14F	0.9600
C1—C6	1.392 (11)	C15—N2	1.446 (9)
C1—C2	1.418 (11)	C15—N3	1.451 (9)
C1—C7	1.488 (10)	C15—C16	1.544 (9)
C2—C3	1.402 (11)	C16—O5′	1.260 (8)
C2—C8	1.505 (11)	C16—O5	1.263 (8)
C3—C4	1.394 (13)	C16—O6′	1.350 (9)
C4—C5	1.362 (13)	C16—O6	1.361 (8)
C4—H4	0.9300	C19—N2	1.434 (9)
C5—C6	1.395 (13)	C19—N5	1.480 (9)
C5—H5	0.9300	C19—H19A	0.9700

C7—N1	1.475 (9)	C19—H19B	0.9700
C7—H7A	0.9700	C20—N5	1.455 (9)
C7—H7B	0.9700	C20—N3	1.470 (9)
C8—N4	1.460 (9)	C20—H20A	0.9700
C8—H8A	0.9700	C20—H20B	0.9700
C8—H8B	0.9700	C21—N5	1.498 (10)
C9—O1	1.204 (8)	C21—C22	1.515 (14)
C9—N1	1.358 (9)	C21—C24	1.520 (13)
C9—N2	1.391 (9)	C21—C23	1.559 (12)
C10—O2	1.205 (9)	C22—H22A	0.9600
C10—N4	1.378 (9)	C22—H22B	0.9600
C10—N3	1.385 (10)	C22—H22C	0.9600
C11—N4	1.429 (9)	C23—H23A	0.9600
C11—N1	1.432 (8)	C23—H23B	0.9600
C11—C12	1.530 (11)	C23—H23C	0.9600
C11—C15	1.569 (10)	C24—H24A	0.9600
C12—O3′	1.219 (14)	C24—H24B	0.9600
C12—O3	1.220 (12)	C24—H24C	0.9600
C12—O4	1.305 (9)	O6—C17	1.429 (9)
C12—O4′	1.352 (11)	C17—C18	1.531 (10)
C13—O4	1.455 (10)	C17—H17A	0.9700
C13—C14	1.542 (10)	C17—H17B	0.9700
C13—H13A	0.9700	C18—H18A	0.9600
C13—H13B	0.9700	C18—H18B	0.9600
C14—H14A	0.9600	C18—H18C	0.9600
C14—H14B	0.9600	O6′—C17′	1.389 (10)
C14—H14C	0.9600	C17′—C18′	1.540 (10)
C13′—O4′	1.44 (5)	C17′—H17C	0.9700
C13′—C14′	1.531 (11)	C17′—H17D	0.9700
C13′—H13C	0.9700	C18′—H18D	0.9600
C13′—H13D	0.9700	C18′—H18E	0.9600
C14′—H14D	0.9600	C18′—H18F	0.9600

C6—C1—C2	117.4 (7)	O5′—C16—C15	118.2 (13)
C6—C1—C7	122.6 (7)	O5—C16—C15	121.9 (9)
C2—C1—C7	119.7 (7)	O6′—C16—C15	108.2 (10)
C3—C2—C1	120.3 (8)	O6—C16—C15	110.4 (8)
C3—C2—C8	119.8 (8)	N2—C19—N5	109.6 (6)
C1—C2—C8	119.9 (7)	N2—C19—H19A	109.7
C4—C3—C2	120.4 (9)	N5—C19—H19A	109.7
C4—C3—Br2	116.2 (7)	N2—C19—H19B	109.7
C2—C3—Br2	123.3 (7)	N5—C19—H19B	109.7
C5—C4—C3	119.4 (9)	H19A—C19—H19B	108.2
C5—C4—H4	120.3	N5—C20—N3	108.5 (6)
C3—C4—H4	120.3	N5—C20—H20A	110.0
C4—C5—C6	121.0 (8)	N3—C20—H20A	110.0
C4—C5—H5	119.5	N5—C20—H20B	110.0
C6—C5—H5	119.5	N3—C20—H20B	110.0

C1—C6—C5	121.4 (8)	H20A—C20—H20B	108.4
C1—C6—Br1	121.6 (7)	N5—C21—C22	108.5 (8)
C5—C6—Br1	117.0 (7)	N5—C21—C24	108.7 (7)
N1—C7—C1	114.8 (6)	C22—C21—C24	110.9 (9)
N1—C7—H7A	108.6	N5—C21—C23	111.4 (7)
C1—C7—H7A	108.6	C22—C21—C23	110.1 (9)
N1—C7—H7B	108.6	C24—C21—C23	107.3 (9)
C1—C7—H7B	108.6	C21—C22—H22A	109.5
H7A—C7—H7B	107.6	C21—C22—H22B	109.5
N4—C8—C2	113.4 (6)	H22A—C22—H22B	109.5
N4—C8—H8A	108.9	C21—C22—H22C	109.5
C2—C8—H8A	108.9	H22A—C22—H22C	109.5
N4—C8—H8B	108.9	H22B—C22—H22C	109.5
C2—C8—H8B	108.9	C21—C23—H23A	109.5
H8A—C8—H8B	107.7	C21—C23—H23B	109.5
O1—C9—N1	126.4 (7)	H23A—C23—H23B	109.5
O1—C9—N2	125.5 (7)	C21—C23—H23C	109.5
N1—C9—N2	108.0 (6)	H23A—C23—H23C	109.5
O2—C10—N4	126.0 (8)	H23B—C23—H23C	109.5
O2—C10—N3	126.8 (7)	C21—C24—H24A	109.5
N4—C10—N3	107.1 (7)	C21—C24—H24B	109.5
N4—C11—N1	113.7 (6)	H24A—C24—H24B	109.5
N4—C11—C12	107.6 (6)	C21—C24—H24C	109.5
N1—C11—C12	115.1 (6)	H24A—C24—H24C	109.5
N4—C11—C15	103.3 (6)	H24B—C24—H24C	109.5
N1—C11—C15	102.6 (5)	C9—N1—C11	113.0 (6)
C12—C11—C15	114.0 (6)	C9—N1—C7	121.9 (6)
O3—C12—O4	120.5 (14)	C11—N1—C7	120.1 (6)
O3′—C12—O4′	130 (3)	C9—N2—C19	120.6 (6)
O3′—C12—C11	119 (3)	C9—N2—C15	110.6 (6)
O3—C12—C11	123.4 (13)	C19—N2—C15	116.9 (6)
O4—C12—C11	115.8 (9)	C10—N3—C15	111.7 (6)
O4′—C12—C11	107.9 (19)	C10—N3—C20	120.4 (6)
O4—C13—C14	107.3 (9)	C15—N3—C20	116.3 (6)
O4—C13—H13A	110.3	C10—N4—C11	112.6 (6)
C14—C13—H13A	110.3	C10—N4—C8	122.2 (7)
O4—C13—H13B	110.3	C11—N4—C8	121.8 (6)
C14—C13—H13B	110.3	C20—N5—C19	108.3 (6)
H13A—C13—H13B	108.5	C20—N5—C21	113.5 (6)
C12—O4—C13	114.3 (12)	C19—N5—C21	114.7 (6)
O4′—C13′—C14′	111 (3)	C16—O6—C17	119.0 (10)
O4′—C13′—H13C	109.5	O6—C17—C18	110.0 (10)
C14′—C13′—H13C	109.5	O6—C17—H17A	109.7
O4′—C13′—H13D	109.5	C18—C17—H17A	109.7
C14′—C13′—H13D	109.5	O6—C17—H17B	109.7
H13C—C13′—H13D	108.0	C18—C17—H17B	109.7
C13′—C14′—H14D	109.5	H17A—C17—H17B	108.2
C13′—C14′—H14E	109.5	C16—O6′—C17′	112.5 (19)

H14D—C14′—H14E	109.5	O6′—C17′—C18′	112.5 (11)
C13′—C14′—H14F	109.5	O6′—C17′—H17C	109.1
H14D—C14′—H14F	109.5	C18′—C17′—H17C	109.1
H14E—C14′—H14F	109.5	O6′—C17′—H17D	109.1
C12—O4′—C13′	126 (4)	C18′—C17′—H17D	109.1
N2—C15—N3	111.7 (6)	H17C—C17′—H17D	107.8
N2—C15—C16	112.6 (6)	C17′—C18′—H18D	109.5
N3—C15—C16	110.0 (5)	C17′—C18′—H18E	109.5
N2—C15—C11	103.7 (5)	H18D—C18′—H18E	109.5
N3—C15—C11	102.9 (5)	C17′—C18′—H18F	109.5
C16—C15—C11	115.4 (6)	H18D—C18′—H18F	109.5
O5′—C16—O6′	132.6 (12)	H18E—C18′—H18F	109.5
O5—C16—O6	127.4 (9)		

C6—C1—C2—C3	0.8 (10)	O1—C9—N1—C7	−14.0 (11)
C7—C1—C2—C3	−173.8 (6)	N2—C9—N1—C7	169.9 (6)
C6—C1—C2—C8	−178.1 (7)	N4—C11—N1—C9	101.4 (7)
C7—C1—C2—C8	7.4 (10)	C12—C11—N1—C9	−133.8 (7)
C1—C2—C3—C4	−0.5 (11)	C15—C11—N1—C9	−9.5 (7)
C8—C2—C3—C4	178.4 (7)	N4—C11—N1—C7	−54.1 (8)
C1—C2—C3—Br2	176.8 (5)	C12—C11—N1—C7	70.7 (9)
C8—C2—C3—Br2	−4.3 (10)	C15—C11—N1—C7	−165.0 (6)
C2—C3—C4—C5	−0.4 (13)	C1—C7—N1—C9	−77.0 (8)
Br2—C3—C4—C5	−177.9 (7)	C1—C7—N1—C11	76.3 (8)
C3—C4—C5—C6	0.9 (13)	O1—C9—N2—C19	28.2 (11)
C2—C1—C6—C5	−0.3 (11)	N1—C9—N2—C19	−155.8 (6)
C7—C1—C6—C5	174.1 (7)	O1—C9—N2—C15	169.9 (7)
C2—C1—C6—Br1	−178.4 (5)	N1—C9—N2—C15	−14.1 (8)
C7—C1—C6—Br1	−4.0 (10)	N5—C19—N2—C9	87.2 (8)
C4—C5—C6—C1	−0.6 (13)	N5—C19—N2—C15	−52.3 (8)
C4—C5—C6—Br1	177.7 (7)	N3—C15—N2—C9	−102.2 (7)
C6—C1—C7—N1	119.2 (7)	C16—C15—N2—C9	133.3 (6)
C2—C1—C7—N1	−66.5 (9)	C11—C15—N2—C9	7.9 (7)
C3—C2—C8—N4	−122.8 (7)	N3—C15—N2—C19	41.0 (8)
C1—C2—C8—N4	56.1 (10)	C16—C15—N2—C19	−83.5 (8)
N4—C11—C12—O3′	−71 (2)	C11—C15—N2—C19	151.1 (6)
N1—C11—C12—O3′	161 (2)	O2—C10—N3—C15	−169.2 (7)
C15—C11—C12—O3′	43 (3)	N4—C10—N3—C15	15.2 (8)
N4—C11—C12—O3	−30 (2)	O2—C10—N3—C20	−27.3 (11)
N1—C11—C12—O3	−157.6 (19)	N4—C10—N3—C20	157.1 (6)
C15—C11—C12—O3	84 (2)	N2—C15—N3—C10	102.2 (7)
N4—C11—C12—O4	143.8 (16)	C16—C15—N3—C10	−131.8 (6)
N1—C11—C12—O4	15.8 (18)	C11—C15—N3—C10	−8.4 (7)
C15—C11—C12—O4	−102.3 (16)	N2—C15—N3—C20	−41.4 (8)
N4—C11—C12—O4′	127 (3)	C16—C15—N3—C20	84.5 (8)
N1—C11—C12—O4′	−1 (3)	C11—C15—N3—C20	−152.0 (6)
C15—C11—C12—O4′	−119 (3)	N5—C20—N3—C10	−86.7 (8)
O3′—C12—O4—C13	39 (3)	N5—C20—N3—C15	53.6 (8)

O3—C12—O4—C13	−1 (3)	O2—C10—N4—C11	168.1 (7)
O4′—C12—O4—C13	−109 (11)	N3—C10—N4—C11	−16.2 (8)
C11—C12—O4—C13	−175.0 (15)	O2—C10—N4—C8	8.7 (11)
C14—C13—O4—C12	−175 (3)	N3—C10—N4—C8	−175.7 (6)
O3′—C12—O4′—C13′	30 (8)	N1—C11—N4—C10	−99.8 (7)
O3—C12—O4′—C13′	−14 (7)	C12—C11—N4—C10	131.5 (6)
O4—C12—O4′—C13′	70 (7)	C15—C11—N4—C10	10.6 (7)
C11—C12—O4′—C13′	−170 (4)	N1—C11—N4—C8	59.7 (8)
C14′—C13′—O4′—C12	−87 (8)	C12—C11—N4—C8	−69.0 (8)
N4—C11—C15—N2	−117.7 (6)	C15—C11—N4—C8	170.1 (6)
N1—C11—C15—N2	0.7 (7)	C2—C8—N4—C10	77.2 (9)
C12—C11—C15—N2	125.8 (7)	C2—C8—N4—C11	−80.3 (9)
N4—C11—C15—N3	−1.2 (6)	N3—C20—N5—C19	−62.2 (7)
N1—C11—C15—N3	117.2 (6)	N3—C20—N5—C21	169.2 (6)
C12—C11—C15—N3	−117.6 (7)	N2—C19—N5—C20	62.2 (7)
N4—C11—C15—C16	118.6 (6)	N2—C19—N5—C21	−169.8 (6)
N1—C11—C15—C16	−122.9 (6)	C22—C21—N5—C20	−177.9 (8)
C12—C11—C15—C16	2.2 (9)	C24—C21—N5—C20	−57.2 (10)
N2—C15—C16—O5′	166 (3)	C23—C21—N5—C20	60.8 (10)
N3—C15—C16—O5′	40 (3)	C22—C21—N5—C19	56.9 (9)
C11—C15—C16—O5′	−76 (3)	C24—C21—N5—C19	177.6 (8)
N2—C15—C16—O5	123.9 (17)	C23—C21—N5—C19	−64.4 (10)
N3—C15—C16—O5	−1.5 (19)	O5′—C16—O6—C17	−36 (3)
C11—C15—C16—O5	−117.3 (18)	O5—C16—O6—C17	7 (2)
N2—C15—C16—O6′	−24.2 (17)	O6′—C16—O6—C17	91 (4)
N3—C15—C16—O6′	−149.6 (16)	C15—C16—O6—C17	−180.0 (17)
C11—C15—C16—O6′	94.6 (16)	C16—O6—C17—C18	−175 (4)
N2—C15—C16—O6	−49.9 (14)	O5′—C16—O6′—C17′	−6 (3)
N3—C15—C16—O6	−175.3 (13)	O5—C16—O6′—C17′	37 (2)
C11—C15—C16—O6	68.9 (14)	O6—C16—O6′—C17′	−75 (3)
O1—C9—N1—C11	−169.0 (7)	C15—C16—O6′—C17′	−174.6 (14)
N2—C9—N1—C11	14.9 (8)	C16—O6′—C17′—C18′	124 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···Br2	0.97	2.59	3.190 (8)	120
C7—H7*B*···Br1	0.97	2.61	3.206 (7)	120
C7—H7*A*···O3^{i}	0.97	2.55	3.125 (17)	118
C14—H14*C*···*Cg*1^{ii}	0.97	2.97	3.89 (4)	160

Symmetry codes: (i) −*x*+5/2, *y*+1/2, −*z*+1/2; (ii) −*x*+5/2, *y*−1/2, −*z*+1/2.