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

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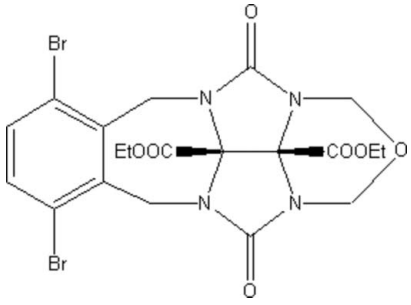
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.042; wR factor = 0.106; data-to-parameter ratio = 15.2.

In the molecule of the title compound, $C_{20}H_{20}Br_2N_4O_7$, one ethyl group is disordered over two positions; site-occupation factors were fixed at 0.63 and 0.37. The non-planar seven- and six-membered rings adopt chair conformations, while the two five-membered rings have envelope conformations. In the crystal structure, C—H···O and C—H···Br hydrogen bonds result in the formation of a tape-like structure.

Related literature

For general background, see: Burnett *et al.* (2003); Wu *et al.* (2002). For ring conformation puckering parameters, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{20}Br_2N_4O_7$
M_r = 588.22
Orthorhombic, $P2_12_12_1$
a = 10.6430 (12) Å
b = 11.2669 (13) Å
c = 18.673 (2) Å
V = 2239.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.67 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
T_{min} = 0.406, T_{max} = 0.527
(expected range = 0.369–0.480)
13113 measured reflections
4879 independent reflections
3828 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
S = 1.00
4879 reflections
320 parameters
40 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Absolute structure: Flack (1983), 2108 Friedel pairs
Flack parameter: 0.019 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O5	0.97	2.41	2.981 (5)	117
C8—H8A···Br1	0.97	2.59	3.198 (4)	121
C7—H7B···Br2	0.97	2.58	3.189 (3)	121
C19—H19A···O2^{i}	0.97	2.46	3.274 (6)	141
C11—H11A···O4^{i}	0.97	2.55	3.343 (5)	139
C8—H8B···O1^{ii}	0.97	2.44	3.178 (5)	133
C7—H7B···O6^{iii}	0.97	2.52	3.302 (5)	137
C6—H6···O2^{iv}	0.93	2.46	3.182 (5)	134

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 2001); 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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2354).

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supplementary materials

Acta Cryst. (2007). E**63**, o4541 [**doi:10.1107/S1600536807054438**]

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

K. Wang, J. Li and N. She

Comment

Diethoxycarbonyl glycoluril bearing a range of electron withdrawing functional groups on its convex face is an important building block for both molecular and supramolecular chemistry (Burnett *et al.*, 2003). The title compound, (I), derived from diethoxycarbonyl glycoluril is an important intermediate for methylene-bridged glycoluril dimers. We report herein its crystal structure.

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987).

When the crystal structure was solved, the atoms C15, H15A, H15B, C16, H16A, H16B and H16C were found to be disordered.

Rings B (N1/N2/C1/C2/C7/C8/C13) and E (N3/N4/O3/C11/C12/C17) are not planar, having total puckering amplitudes, Q_T, of 2.312 (3) and 1.267 (2) Å, respectively. They adopt chair conformations [φ = 108.31 (2)° and θ = 4.40 (3)° (for ring E)] (Cremer & Pople, 1975). Rings C and D have envelope conformations with atoms C9 and C10 displaced by −0.207 (3) Å and −0.246 (2) Å from the planes of the other ring atoms, respectively. Ring A (C1—C6) is, of course, planar.

In the crystal structure, C—H···O and C—H···Br hydrogen bonds (Table 2, Fig. 2) result in the formation of a tape-like structure, in which they may be effective in the stabilization of the structure.

Experimental

The title compound was synthesized according to the procedure reported (Wu *et al.*, 2002). Crystals appropriate for X-ray analysis were obtained by slow evaporation of the dichloromethane solution at 283 K.

Refinement

When the crystal structure was solved, the atoms C15, H15A, H15B, C16, H16A, H16B and H16C were found to be disordered. During refinement, the occupancies of disordered C and H atoms were kept fixed as C15 = 0.63, C15' = 0.37, C16 = 0.63, C16' = 0.37, H15A = 0.63, H15C = 0.37, H15B = 0.63, H15D = 0.37, H16A = 0.63, H16D = 0.37, H16B = 0.63, H16E = 0.37, H16C = 0.63 and H16F = 0.37. H atoms were positioned geometrically with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

Figures

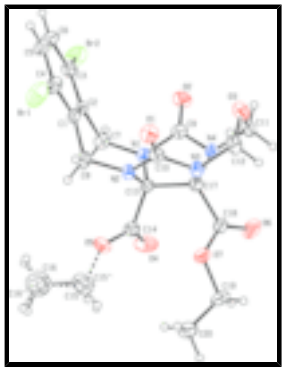


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

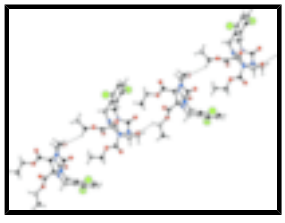


Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

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

Crystal data

$C_{20}H_{20}Br_2N_4O_7$	F_{000} = 1176
M_r = 588.22	D_x = 1.745 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4974 reflections
a = 10.6430 (12) Å	θ = 2.6–24.1°
b = 11.2669 (13) Å	μ = 3.67 mm^{-1}
c = 18.673 (2) Å	T = 298 (2) K
V = 2239.1 (4) Å^3	Block, colorless
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4879 independent reflections
Radiation source: fine-focus sealed tube	3828 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 298(2) K	θ_{max} = 27.0°
φ andω scans	θ_{min} = 2.1°
Absorption correction: multi-scan (SADABS; Sheldrick,1996)	h = −13→7
T_{min} = 0.406, T_{max} = 0.527	k = −14→14
13113 measured reflections	l = −23→23

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.106$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.87$ e Å^{-3}
4879 reflections	$\Delta\rho_{min} = -0.56$ e Å^{-3}
320 parameters	Extinction correction: none
40 restraints	Absolute structure: Flack (1983), with 2108 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.019 (10)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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	1.24828 (5)	0.42440 (5)	0.82344 (3)	0.0778 (2)	
Br2	0.83248 (6)	0.04207 (5)	0.93545 (3)	0.07348 (19)	
C1	1.0072 (3)	0.3091 (3)	0.81524 (19)	0.0326 (8)	
C2	0.9179 (4)	0.2254 (3)	0.83950 (17)	0.0327 (8)	
C3	0.9429 (4)	0.1627 (4)	0.9009 (2)	0.0430 (9)	
C4	1.1153 (4)	0.3249 (3)	0.8550 (2)	0.0457 (9)	
C5	1.1361 (5)	0.2618 (4)	0.9183 (3)	0.0585 (13)	
H5	1.2084	0.2754	0.9450	0.070*	
C6	1.0509 (5)	0.1809 (4)	0.9407 (2)	0.0580 (12)	
H6	1.0648	0.1378	0.9824	0.070*	
C7	0.7998 (3)	0.2026 (3)	0.7960 (2)	0.0365 (8)	
H7A	0.8238	0.1706	0.7498	0.044*	
H7B	0.7500	0.1428	0.8203	0.044*	
C8	0.9871 (3)	0.3731 (3)	0.7445 (2)	0.0355 (8)	
H8A	1.0591	0.4240	0.7360	0.043*	

H8B	0.9855	0.3143	0.7066	0.043*	
C9	0.6464 (3)	0.3534 (3)	0.83720 (18)	0.0348 (8)	
C10	0.8689 (3)	0.5529 (3)	0.77131 (17)	0.0325 (8)	
C11	0.5539 (4)	0.5491 (4)	0.8602 (2)	0.0487 (10)	
H11A	0.4868	0.5948	0.8384	0.058*	
H11B	0.5211	0.5126	0.9034	0.058*	
C12	0.6992 (5)	0.6817 (3)	0.8162 (2)	0.0543 (11)	
H12A	0.7661	0.7361	0.8290	0.065*	
H12B	0.6320	0.7273	0.7945	0.065*	
C13	0.7531 (3)	0.3920 (3)	0.72946 (16)	0.0291 (7)	
C14	0.7293 (4)	0.3329 (3)	0.65579 (19)	0.0396 (9)	
C15	0.8078 (10)	0.2619 (9)	0.5512 (4)	0.069 (3)	0.63
H15A	0.7429	0.2037	0.5611	0.083*	0.63
H15B	0.7800	0.3106	0.5115	0.083*	0.63
C16	0.9245 (11)	0.2013 (11)	0.5321 (7)	0.106 (4)	0.63
H16A	0.9931	0.2565	0.5342	0.160*	0.63
H16B	0.9176	0.1700	0.4844	0.160*	0.63
H16C	0.9394	0.1375	0.5651	0.160*	0.63
C15'	0.8403 (13)	0.3069 (15)	0.5401 (5)	0.059 (4)	0.37
H15C	0.8332	0.3781	0.5112	0.070*	0.37
H15D	0.7735	0.2531	0.5263	0.070*	0.37
C16'	0.9655 (12)	0.2493 (13)	0.5272 (8)	0.055 (3)	0.37
H16D	1.0286	0.3094	0.5217	0.083*	0.37
H16E	0.9614	0.2020	0.4845	0.083*	0.37
H16F	0.9864	0.1996	0.5672	0.083*	0.37
C17	0.6607 (3)	0.4976 (3)	0.74721 (17)	0.0328 (7)	
C18	0.5684 (3)	0.5340 (4)	0.6883 (2)	0.0411 (8)	
C19	0.5544 (5)	0.5840 (6)	0.5650 (2)	0.0721 (15)	
H19A	0.5350	0.6682	0.5649	0.086*	
H19B	0.4759	0.5404	0.5663	0.086*	
C20	0.6242 (5)	0.5533 (5)	0.4999 (2)	0.0605 (12)	
H20A	0.6445	0.4703	0.5005	0.091*	
H20B	0.7003	0.5989	0.4978	0.091*	
H20C	0.5735	0.5705	0.4586	0.091*	
N1	0.7219 (3)	0.3072 (2)	0.78463 (15)	0.0307 (7)	
N2	0.8741 (3)	0.4448 (2)	0.73897 (14)	0.0312 (6)	
N3	0.7466 (3)	0.5948 (2)	0.76439 (15)	0.0361 (7)	
N4	0.5948 (3)	0.4576 (3)	0.81106 (15)	0.0368 (7)	
O1	0.9548 (3)	0.6058 (2)	0.79841 (15)	0.0480 (7)	
O2	0.6257 (3)	0.3098 (3)	0.89449 (14)	0.0496 (7)	
O3	0.6542 (3)	0.6257 (3)	0.87831 (15)	0.0538 (7)	
O4	0.6307 (3)	0.2891 (3)	0.64205 (16)	0.0628 (9)	
O5	0.8276 (3)	0.3372 (3)	0.61512 (14)	0.0626 (9)	
O6	0.4591 (3)	0.5415 (4)	0.69709 (17)	0.0794 (11)	
O7	0.6289 (3)	0.5551 (3)	0.62817 (13)	0.0467 (7)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0410 (2)	0.0734 (3)	0.1189 (5)	−0.0152 (3)	−0.0261 (3)	0.0093 (3)
Br2	0.0752 (3)	0.0742 (3)	0.0710 (3)	0.0035 (3)	0.0081 (3)	0.0415 (3)
C1	0.0276 (17)	0.0334 (18)	0.0368 (19)	0.0073 (14)	−0.0038 (16)	−0.0003 (15)
C2	0.0355 (19)	0.0350 (18)	0.0278 (18)	0.0133 (16)	0.0007 (15)	−0.0003 (14)
C3	0.054 (2)	0.041 (2)	0.034 (2)	0.0088 (19)	0.0053 (18)	0.0040 (16)
C4	0.039 (2)	0.042 (2)	0.057 (2)	0.0005 (18)	−0.0129 (19)	−0.0028 (18)
C5	0.049 (3)	0.064 (3)	0.062 (3)	0.012 (2)	−0.027 (2)	−0.010 (2)
C6	0.067 (3)	0.066 (3)	0.041 (2)	0.020 (3)	−0.015 (2)	0.007 (2)
C7	0.0379 (19)	0.0313 (17)	0.040 (2)	−0.0056 (16)	−0.0014 (17)	0.0063 (15)
C8	0.0272 (17)	0.0390 (19)	0.0402 (19)	0.0005 (16)	0.0049 (16)	0.0027 (16)
C9	0.0302 (19)	0.0404 (19)	0.0339 (18)	−0.0081 (16)	−0.0006 (15)	0.0048 (15)
C10	0.0343 (18)	0.0302 (17)	0.0329 (17)	−0.0049 (15)	0.0014 (15)	0.0063 (14)
C11	0.046 (2)	0.059 (3)	0.041 (2)	0.012 (2)	0.0123 (18)	−0.001 (2)
C12	0.062 (3)	0.037 (2)	0.064 (3)	0.005 (2)	0.010 (2)	−0.008 (2)
C13	0.0270 (16)	0.0334 (16)	0.0270 (17)	0.0007 (16)	−0.0017 (14)	0.0036 (13)
C14	0.049 (3)	0.0387 (19)	0.0315 (19)	0.0049 (19)	−0.0007 (18)	0.0036 (15)
C15	0.081 (6)	0.080 (5)	0.045 (4)	−0.003 (4)	0.004 (4)	−0.012 (4)
C16	0.108 (4)	0.106 (4)	0.105 (4)	0.002 (2)	0.001 (2)	−0.002 (2)
C15'	0.057 (7)	0.078 (8)	0.040 (6)	0.007 (7)	0.009 (6)	−0.004 (6)
C16'	0.056 (4)	0.055 (4)	0.055 (4)	0.001 (2)	0.001 (2)	−0.002 (2)
C17	0.0313 (18)	0.0362 (17)	0.0310 (16)	−0.0005 (16)	0.0039 (16)	0.0040 (13)
C18	0.034 (2)	0.050 (2)	0.039 (2)	0.0094 (18)	0.0005 (17)	0.0057 (17)
C19	0.068 (3)	0.106 (4)	0.042 (2)	0.034 (3)	−0.013 (2)	0.017 (3)
C20	0.071 (3)	0.064 (3)	0.046 (2)	0.008 (3)	−0.011 (2)	0.005 (2)
N1	0.0276 (16)	0.0320 (14)	0.0326 (15)	0.0005 (12)	0.0053 (12)	0.0059 (12)
N2	0.0278 (14)	0.0289 (15)	0.0369 (15)	0.0004 (12)	0.0041 (12)	0.0036 (12)
N3	0.0369 (16)	0.0283 (13)	0.0432 (17)	0.0045 (15)	0.0015 (14)	0.0002 (12)
N4	0.0337 (15)	0.0392 (15)	0.0376 (16)	0.0040 (14)	0.0086 (14)	0.0014 (13)
O1	0.0427 (16)	0.0432 (15)	0.0579 (17)	−0.0130 (13)	−0.0049 (14)	−0.0044 (13)
O2	0.0588 (19)	0.0536 (16)	0.0366 (14)	−0.0038 (15)	0.0131 (13)	0.0123 (12)
O3	0.0584 (19)	0.0532 (16)	0.0499 (17)	0.0027 (17)	0.0067 (15)	−0.0128 (13)
O4	0.069 (2)	0.070 (2)	0.0498 (17)	−0.0255 (19)	−0.0159 (16)	−0.0074 (15)
O5	0.0555 (19)	0.101 (3)	0.0312 (14)	0.020 (2)	0.0018 (15)	−0.0157 (15)
O6	0.0393 (17)	0.135 (3)	0.063 (2)	0.027 (2)	−0.0009 (16)	0.022 (2)
O7	0.0426 (15)	0.0613 (17)	0.0363 (13)	0.0075 (14)	−0.0027 (12)	0.0137 (12)

Geometric parameters (Å, °)

Br1—C4	1.899 (4)	C13—N2	1.430 (5)
Br2—C3	1.909 (4)	C13—N1	1.443 (4)
C1—C4	1.381 (5)	C13—C14	1.549 (5)
C1—C2	1.413 (5)	C13—C17	1.579 (5)
C1—C8	1.519 (5)	C14—O4	1.187 (5)
C2—C3	1.373 (5)	C14—O5	1.294 (5)
C2—C7	1.519 (5)	C15—C16	1.462 (9)

C3—C6	1.384 (7)	C15—O5	1.479 (7)
C4—C5	1.397 (6)	C15—H15A	0.9700
C5—C6	1.352 (7)	C15—H15B	0.9700
C5—H5	0.9300	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—N1	1.457 (4)	C16—H16C	0.9600
C7—H7A	0.9700	C15'—O5	1.448 (9)
C7—H7B	0.9700	C15'—C16'	1.502 (9)
C8—N2	1.453 (5)	C15'—H15C	0.9700
C8—H8A	0.9700	C15'—H15D	0.9700
C8—H8B	0.9700	C16'—H16D	0.9600
C9—O2	1.197 (4)	C16'—H16E	0.9600
C9—N1	1.371 (5)	C16'—H16F	0.9600
C9—N4	1.386 (5)	C17—N4	1.455 (4)
C10—O1	1.203 (4)	C17—N3	1.463 (5)
C10—N2	1.360 (4)	C17—C18	1.530 (5)
C10—N3	1.391 (5)	C18—O6	1.178 (4)
C11—O3	1.414 (5)	C18—O7	1.316 (4)
C11—N4	1.447 (5)	C19—O7	1.458 (5)
C11—H11A	0.9700	C19—C20	1.467 (6)
C11—H11B	0.9700	C19—H19A	0.9700
C12—O3	1.404 (5)	C19—H19B	0.9700
C12—N3	1.466 (5)	C20—H20A	0.9600
C12—H12A	0.9700	C20—H20B	0.9600
C12—H12B	0.9700	C20—H20C	0.9600
C4—C1—C2	118.3 (3)	O5—C14—C13	111.9 (4)
C4—C1—C8	121.5 (3)	C16—C15—O5	110.1 (9)
C2—C1—C8	120.1 (3)	C16—C15—H15A	109.6
C3—C2—C1	118.8 (4)	O5—C15—H15A	109.6
C3—C2—C7	121.3 (4)	C16—C15—H15B	109.6
C1—C2—C7	119.9 (3)	O5—C15—H15B	109.6
C2—C3—C6	122.2 (4)	H15A—C15—H15B	108.1
C2—C3—Br2	122.0 (3)	O5—C15'—C16'	109.8 (10)
C6—C3—Br2	115.8 (3)	O5—C15'—H15C	109.7
C1—C4—C5	121.4 (4)	C16'—C15'—H15C	109.7
C1—C4—Br1	122.0 (3)	O5—C15'—H15D	109.7
C5—C4—Br1	116.4 (3)	C16'—C15'—H15D	109.7
C6—C5—C4	119.9 (4)	H15C—C15'—H15D	108.2
C6—C5—H5	120.1	C15'—C16'—H16D	109.5
C4—C5—H5	120.1	C15'—C16'—H16E	109.5
C5—C6—C3	119.4 (4)	H16D—C16'—H16E	109.5
C5—C6—H6	120.3	C15'—C16'—H16F	109.5
C3—C6—H6	120.3	H16D—C16'—H16F	109.5
N1—C7—C2	114.3 (3)	H16E—C16'—H16F	109.5
N1—C7—H7A	108.7	N4—C17—N3	110.7 (3)
C2—C7—H7A	108.7	N4—C17—C18	111.3 (3)
N1—C7—H7B	108.7	N3—C17—C18	111.0 (3)
C2—C7—H7B	108.7	N4—C17—C13	103.9 (3)
H7A—C7—H7B	107.6	N3—C17—C13	102.8 (3)

N2—C8—C1	116.2 (3)	C18—C17—C13	116.8 (3)
N2—C8—H8A	108.2	O6—C18—O7	126.1 (4)
C1—C8—H8A	108.2	O6—C18—C17	123.5 (4)
N2—C8—H8B	108.2	O7—C18—C17	110.4 (3)
C1—C8—H8B	108.2	O7—C19—C20	110.0 (4)
H8A—C8—H8B	107.4	O7—C19—H19A	109.7
O2—C9—N1	126.3 (3)	C20—C19—H19A	109.7
O2—C9—N4	126.1 (4)	O7—C19—H19B	109.7
N1—C9—N4	107.6 (3)	C20—C19—H19B	109.7
O1—C10—N2	126.8 (3)	H19A—C19—H19B	108.2
O1—C10—N3	125.6 (3)	C19—C20—H20A	109.5
N2—C10—N3	107.5 (3)	C19—C20—H20B	109.5
O3—C11—N4	111.1 (3)	H20A—C20—H20B	109.5
O3—C11—H11A	109.4	C19—C20—H20C	109.5
N4—C11—H11A	109.4	H20A—C20—H20C	109.5
O3—C11—H11B	109.4	H20B—C20—H20C	109.5
N4—C11—H11B	109.4	C9—N1—C13	113.3 (3)
H11A—C11—H11B	108.0	C9—N1—C7	122.4 (3)
O3—C12—N3	111.3 (3)	C13—N1—C7	120.6 (3)
O3—C12—H12A	109.4	C10—N2—C13	113.0 (3)
N3—C12—H12A	109.4	C10—N2—C8	120.0 (3)
O3—C12—H12B	109.4	C13—N2—C8	121.5 (3)
N3—C12—H12B	109.4	C10—N3—C17	110.5 (3)
H12A—C12—H12B	108.0	C10—N3—C12	119.2 (3)
N2—C13—N1	113.2 (3)	C17—N3—C12	115.4 (3)
N2—C13—C14	115.9 (3)	C9—N4—C11	120.0 (3)
N1—C13—C14	108.2 (3)	C9—N4—C17	111.1 (3)
N2—C13—C17	102.8 (3)	C11—N4—C17	116.4 (3)
N1—C13—C17	101.9 (3)	C12—O3—C11	109.5 (3)
C14—C13—C17	114.1 (3)	C14—O5—C15'	129.4 (7)
O4—C14—O5	127.1 (4)	C14—O5—C15	109.7 (5)
O4—C14—C13	121.0 (4)	C18—O7—C19	117.7 (3)
C4—C1—C2—C3	−0.6 (5)	C2—C7—N1—C9	−76.3 (4)
C8—C1—C2—C3	175.3 (3)	C2—C7—N1—C13	80.4 (4)
C4—C1—C2—C7	−178.0 (3)	O1—C10—N2—C13	164.1 (3)
C8—C1—C2—C7	−2.1 (5)	N3—C10—N2—C13	−18.8 (4)
C1—C2—C3—C6	1.7 (6)	O1—C10—N2—C8	9.8 (5)
C7—C2—C3—C6	179.0 (4)	N3—C10—N2—C8	−173.2 (3)
C1—C2—C3—Br2	−176.8 (3)	N1—C13—N2—C10	−97.6 (3)
C7—C2—C3—Br2	0.5 (5)	C14—C13—N2—C10	136.6 (3)
C2—C1—C4—C5	−1.1 (6)	C17—C13—N2—C10	11.5 (3)
C8—C1—C4—C5	−177.0 (4)	N1—C13—N2—C8	56.3 (4)
C2—C1—C4—Br1	174.4 (3)	C14—C13—N2—C8	−69.5 (4)
C8—C1—C4—Br1	−1.4 (5)	C17—C13—N2—C8	165.4 (3)
C1—C4—C5—C6	1.9 (7)	C1—C8—N2—C10	76.9 (4)
Br1—C4—C5—C6	−173.9 (4)	C1—C8—N2—C13	−75.3 (4)
C4—C5—C6—C3	−0.9 (7)	O1—C10—N3—C17	−164.6 (3)
C2—C3—C6—C5	−0.9 (7)	N2—C10—N3—C17	18.3 (4)
Br2—C3—C6—C5	177.6 (3)	O1—C10—N3—C12	−27.3 (5)

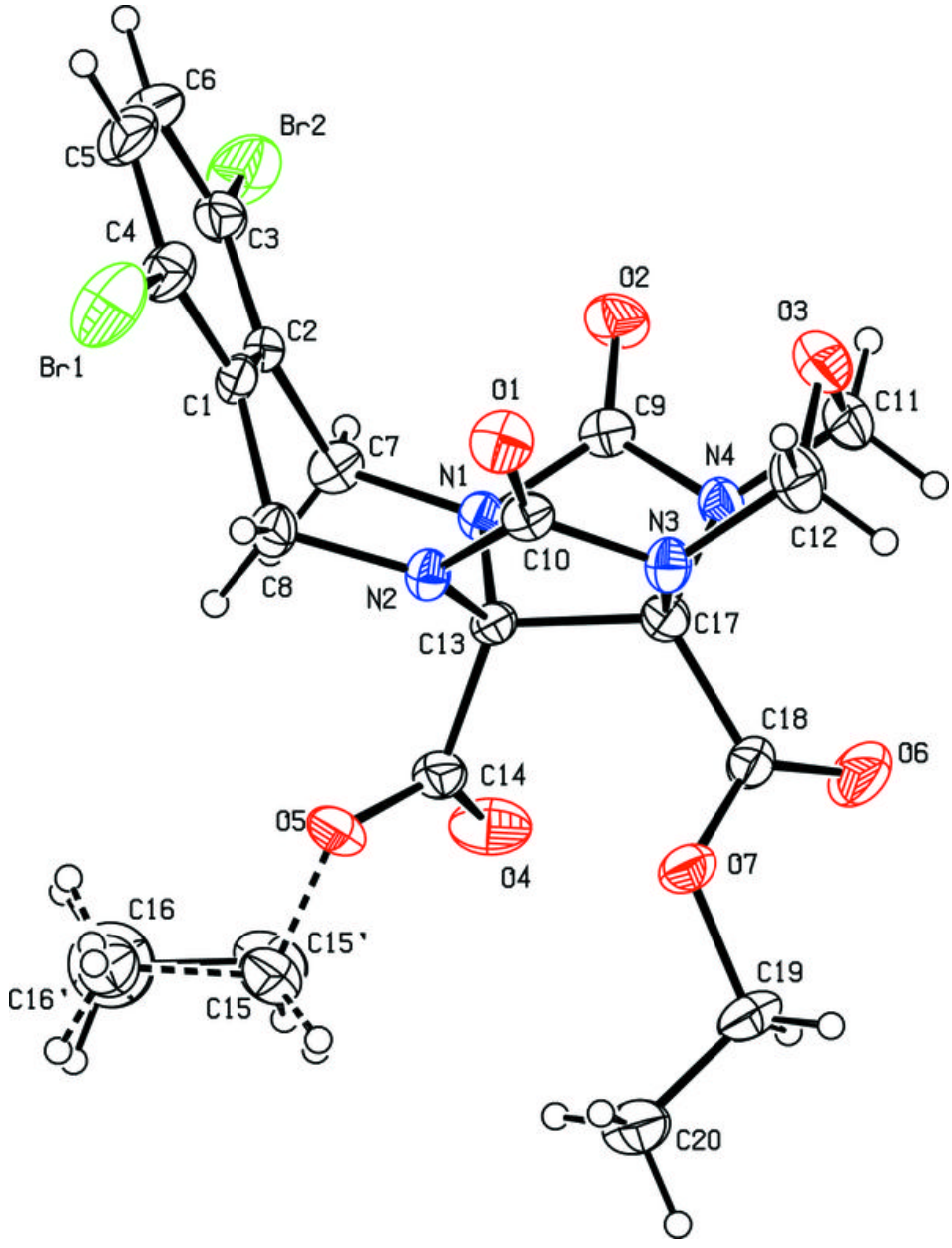
C3—C2—C7—N1	124.0 (4)	N2—C10—N3—C12	155.5 (3)
C1—C2—C7—N1	−58.7 (4)	N4—C17—N3—C10	99.6 (3)
C4—C1—C8—N2	−123.9 (4)	C18—C17—N3—C10	−136.3 (3)
C2—C1—C8—N2	60.4 (4)	C13—C17—N3—C10	−10.8 (3)
N2—C13—C14—O4	−178.2 (3)	N4—C17—N3—C12	−39.4 (4)
N1—C13—C14—O4	53.5 (5)	C18—C17—N3—C12	84.6 (4)
C17—C13—C14—O4	−59.1 (5)	C13—C17—N3—C12	−149.8 (3)
N2—C13—C14—O5	2.8 (4)	O3—C12—N3—C10	−83.8 (5)
N1—C13—C14—O5	−125.6 (3)	O3—C12—N3—C17	51.5 (5)
C17—C13—C14—O5	121.9 (4)	O2—C9—N4—C11	26.9 (6)
N2—C13—C17—N4	−115.6 (3)	N1—C9—N4—C11	−155.2 (3)
N1—C13—C17—N4	1.8 (3)	O2—C9—N4—C17	167.4 (4)
C14—C13—C17—N4	118.2 (3)	N1—C9—N4—C17	−14.7 (4)
N2—C13—C17—N3	−0.1 (3)	O3—C11—N4—C9	86.7 (4)
N1—C13—C17—N3	117.3 (3)	O3—C11—N4—C17	−51.8 (4)
C14—C13—C17—N3	−126.4 (3)	N3—C17—N4—C9	−102.1 (3)
N2—C13—C17—C18	121.5 (3)	C18—C17—N4—C9	134.0 (3)
N1—C13—C17—C18	−121.1 (3)	C13—C17—N4—C9	7.6 (4)
C14—C13—C17—C18	−4.7 (4)	N3—C17—N4—C11	40.0 (4)
N4—C17—C18—O6	8.2 (6)	C18—C17—N4—C11	−83.9 (4)
N3—C17—C18—O6	−115.5 (5)	C13—C17—N4—C11	149.7 (3)
C13—C17—C18—O6	127.2 (5)	N3—C12—O3—C11	−61.0 (5)
N4—C17—C18—O7	−171.9 (3)	N4—C11—O3—C12	61.0 (4)
N3—C17—C18—O7	64.4 (4)	O4—C14—O5—C15'	10.0 (11)
C13—C17—C18—O7	−52.9 (4)	C13—C14—O5—C15'	−171.0 (9)
O2—C9—N1—C13	−165.7 (4)	O4—C14—O5—C15	−8.9 (7)
N4—C9—N1—C13	16.4 (4)	C13—C14—O5—C15	170.0 (5)
O2—C9—N1—C7	−7.4 (6)	C16'—C15'—O5—C14	−143.0 (10)
N4—C9—N1—C7	174.7 (3)	C16'—C15'—O5—C15	−98 (2)
N2—C13—N1—C9	98.5 (3)	C16—C15—O5—C14	−142.1 (9)
C14—C13—N1—C9	−131.6 (3)	C16—C15—O5—C15'	73.5 (18)
C17—C13—N1—C9	−11.1 (4)	O6—C18—O7—C19	−3.7 (7)
N2—C13—N1—C7	−60.1 (4)	C17—C18—O7—C19	176.4 (4)
C14—C13—N1—C7	69.7 (4)	C20—C19—O7—C18	−154.9 (4)
C17—C13—N1—C7	−169.8 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8B···O5	0.97	2.41	2.981 (5)	117
C8—H8A···Br1	0.97	2.59	3.198 (4)	121
C7—H7B···Br2	0.97	2.58	3.189 (3)	121
C19—H19A···O2^{i}	0.97	2.46	3.274 (6)	141
C11—H11A···O4^{i}	0.97	2.55	3.343 (5)	139
C8—H8B···O1^{ii}	0.97	2.44	3.178 (5)	133
C7—H7B···O6^{iii}	0.97	2.52	3.302 (5)	137
C6—H6···O2^{iv}	0.93	2.46	3.182 (5)	134

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) −*x*+2, *y*−1/2, −*z*+3/2; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) *x*+1/2, −*y*+1/2, −*z*+2.

Fig. 1


C6
Br2
C5
C3
C4
C2
Br1
C1
C7
O2
O3
O1
C9
C11
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N4
N3
C12
C10
C8
N2
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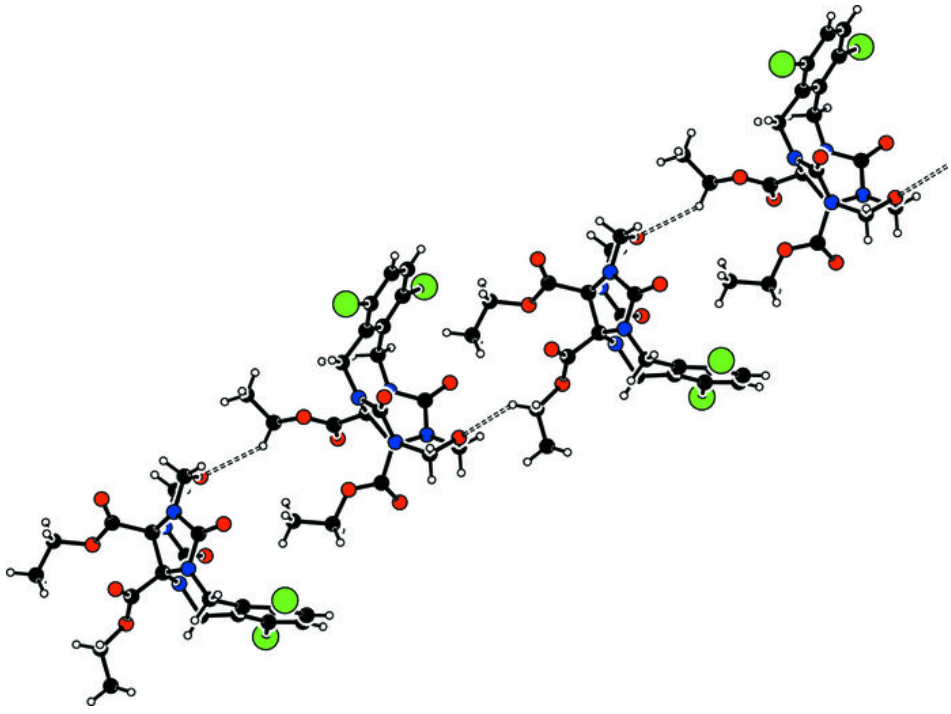
Fig. 2