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Diethyl 6-(4-bromophenyl)-1,4-dioxo-perhydro-2,3,4a,6,7a-pentaazacyclo-penta[*cd*]indene-2a,7b-dicarboxylate methanol hemisolvate

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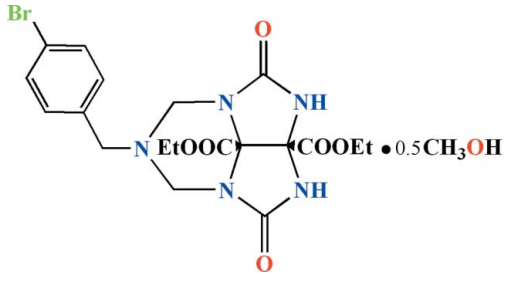
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.045; wR factor = 0.136; data-to-parameter ratio = 13.5.

The title compound, $C_{19}H_{22}BrN_5O_6\cdot0.5CH_3OH$, is a derivative of glycoluril, with two ethyl acetate substituents on the 'convex' face of the glycoluril system. The N atoms from separate rings of the glycouril unit are incorporated into a triazacyclohexane ring, in which the third N atom bears a 4-bromobenzyl substituent. The title compound, containing two free *syn*-urea NH groups and two ureidyl C═O, assembles into one-dimensional helical hydrogen-bonded chains in the solid state, running parallel to the [101] direction. One ethyl group is disordered over two positions; the site-occupancy factors are *ca* 0.52 and 0.48.

Related literature

For the preparation of the title compound, see: Li *et al.* (2006). For general background literature regarding glycoluril and its derivatives, see: Freeman *et al.* (1981); Rebek (2005); Rowan *et al.* (1999); Wu *et al.* (2002). For patterns in hydrogen bonding, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{19}H_{22}BrN_5O_6\cdot0.5CH_4O$
M_r = 510.33
Monoclinic, $C2/c$
a = 24.06370 (14) Å
b = 14.6142 (7) Å
c = 15.1014 (7) Å
β = 123.918 (1)°
V = 4407.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.92 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
22693 measured reflections
4339 independent reflections
2828 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.136
S = 1.02
4339 reflections
322 parameters
31 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O5^{i}	0.93	2.54	3.222 (4)	130
C9—H9A···O2^{ii}	0.97	2.56	3.318 (5)	136
N2—H2···O2^{iii}	0.85 (4)	2.08 (4)	2.914 (3)	168 (3)
N1—H1···O1^{iv}	0.852 (18)	1.98 (1)	2.828 (3)	171 (3)

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

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: *PLATON*.

We 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: FJ2067).

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supplementary materials

Acta Cryst. (2007). E**63**, o4740 [**doi:10.1107/S1600536807057790**]

Diethyl 6-(4-bromophenyl)-1,4-dioxoperhydro-2,3,4a,6,7a-pentaazacyclopenta[*cd*]indene-2a,7b-dicarboxylate methanol hemisolvate

L.-P. Cao, Z.-G. Wang and Y. Hu

Comment

Glycoluril, a biurea compound known for about 130 years, has established an impressive career as building block for molecular and supramolecular chemistry during the recent two decades (Freeman *et al.*, 1981; Rebek, 2005; Rowan *et al.*, 1999; Wu *et al.*, 2002). As a part of our ongoing investigation into glycoluril derivatives (Li *et al.*, 2006), we report here the structure of the title compound (I) (Fig. 1).

The molecular structure of (I) is shown in Fig. 1. It has four fused ring two imidazole and one triazahexane rings. The crystal packing is stabilized by intermolecular non-classical N—H···O hydrogen bonds (Table 1). Interestingly, the two hydrogen-bond donating NH groups and hydrogen-bond accepting ureidyl C═O groups become fully hydrogen-bonded through the formation of $R_2^2(8)$ (Bernstein *et al.* 1995) motifs comprisring N—H···O hydrogen-bonds (Fig 2).

Experimental

The title compound was synthesized according to the procedure of Li *et al.* (2006) in 56% isolated yield. Crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a 1,2-dichloroethane and methanol solution in ratio of 4:1 at 293 K.

Refinement

All H atoms bonded to C atoms were initially located in difference Fourier maps. They were then constrained to their ideal geometry with C–H=0.96Å (methyl), 0.97Å (methylene), with their U_{iso} values being set to 1.5 times of U_{eq} (methyl C) and 1.2 times of U_{eq}(methylene). The H atoms of the amine N atoms were found in difference maps and refined with U_{iso}(H) set to 1.2U_{eq}(N) and the N—H distances refined freely. The H atoms on the disordered methanol solvate were not included in the model. The occupancies of the disordered atoms C5/C5' and C6/C6' was refined to 0.518 (12)/0.482 (12).

Figures

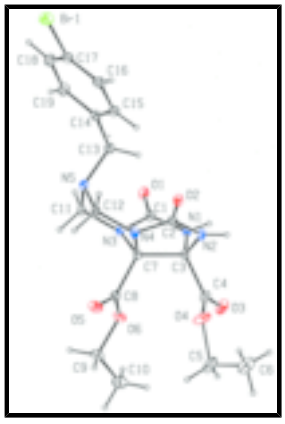


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 10% probability level. H atoms are represented by spheres of arbitrary radius. Atoms of the minor disorder components and the solvent molecule have been omitted for clarity.

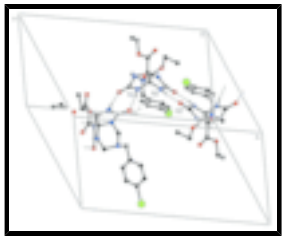


Fig. 2. Packing of (I) with hydrogen bonds drawn as dashed lines showing the formation of a right-handed helical hydrogen-bonded $R_2^2(8)$ ribbon involving *syn*-N atoms and C=O. H atoms not involved in hydrogen bonds have been omitted for clarity. Symmetry codes: i. $-x + 1/2, -y + 3/2, -z$; ii. $-x, y, -z - 1/2$.

Diethyl 6-(4-bromophenyl)-1,4-dioxoperhydro-2,3,4a,6,7a- pentaazacyclopenta[cd]indene-2a,7 b-dicarboxylate methanol hemisolvate

Crystal data

$C_{19}H_{22}BrN_5O_6 \cdot 0.5CH_4O$	$F_{000} = 2088$
$M_r = 510.33$	$D_x = 1.538$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 6618 reflections
a = 24.06370 (14) Å	θ = 2.7–23.9º
b = 14.6142 (7) Å	μ = 1.92 mm^{-1}
c = 15.1014 (7) Å	*T* = 294 (2) K
β = 123.918 (1)º	Block, colorless
V = 4407.0 (3) Å^3	0.30 × 0.20 × 0.20 mm
Z = 8	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2828 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.081$
Monochromator: graphite	$\theta_{max} = 26.0$º
T = 294(2) K	$\theta_{min} = 1.7$º
φ and ω scans	*h* = −26→29
Absorption correction: none	*k* = −18→18

22693 measured reflections
4339 independent reflections

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.136$

$S = 1.02$

4339 reflections

322 parameters

31 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.50$ e Å^{-3}

$\Delta\rho_{min} = -0.38$ e Å^{-3}

Extinction correction: none

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	0.09382 (2)	1.15884 (3)	0.26032 (4)	0.0817 (2)	
C1	0.03759 (14)	0.59152 (19)	−0.0950 (2)	0.0388 (6)	
C2	0.20106 (13)	0.70999 (18)	0.0667 (2)	0.0377 (6)	
C3	0.14998 (13)	0.57862 (18)	−0.0369 (2)	0.0363 (6)	
C4	0.18373 (16)	0.4953 (2)	−0.0486 (3)	0.0496 (8)	
C5	0.2896 (5)	0.4035 (6)	0.0604 (12)	0.103 (4)	0.518 (12)
H5A	0.3077	0.3821	0.1324	0.123*	0.518 (12)
H5B	0.2652	0.3543	0.0102	0.123*	0.518 (12)
C6	0.3427 (5)	0.4415 (7)	0.0492 (10)	0.106 (4)	0.518 (12)
H6A	0.3236	0.4576	−0.0240	0.159*	0.518 (12)
H6B	0.3771	0.3964	0.0713	0.159*	0.518 (12)
H6C	0.3617	0.4950	0.0931	0.159*	0.518 (12)
C5'	0.2856 (6)	0.4261 (9)	0.0065 (8)	0.097 (4)	0.482 (12)
H5'1	0.2560	0.3833	−0.0495	0.116*	0.482 (12)

H5'2	0.3091	0.4618	−0.0171	0.116*	0.482 (12)
C6'	0.3339 (7)	0.3768 (10)	0.1071 (9)	0.139 (6)	0.482 (12)
H6'1	0.3760	0.4085	0.1445	0.208*	0.482 (12)
H6'2	0.3403	0.3158	0.0907	0.208*	0.482 (12)
H6'3	0.3167	0.3741	0.1511	0.208*	0.482 (12)
C7	0.14412 (13)	0.57875 (17)	0.0619 (2)	0.0331 (6)	
C8	0.17281 (14)	0.49725 (19)	0.1388 (2)	0.0418 (7)	
C9	0.1625 (2)	0.3379 (2)	0.1509 (3)	0.0704 (11)	
H9A	0.2080	0.3427	0.2134	0.084*	
H9B	0.1328	0.3296	0.1745	0.084*	
C10	0.1570 (3)	0.2609 (3)	0.0866 (4)	0.1003 (15)	
H10A	0.1120	0.2568	0.0247	0.150*	
H10B	0.1681	0.2057	0.1277	0.150*	
H10C	0.1872	0.2689	0.0647	0.150*	
C11	0.05125 (14)	0.6256 (2)	0.0780 (2)	0.0398 (6)	
H11A	0.0029	0.6327	0.0346	0.048*	
H11B	0.0628	0.5857	0.1372	0.048*	
C12	0.15451 (14)	0.7051 (2)	0.1800 (2)	0.0396 (6)	
H12A	0.1694	0.6682	0.2429	0.047*	
H12B	0.1749	0.7651	0.2038	0.047*	
C13	0.05662 (15)	0.7874 (2)	0.0380 (2)	0.0472 (7)	
H13A	0.0095	0.7770	−0.0159	0.057*	
H13B	0.0805	0.7856	0.0034	0.057*	
C14	0.06560 (14)	0.8802 (2)	0.0884 (2)	0.0448 (7)	
C15	0.12066 (16)	0.9345 (2)	0.1205 (3)	0.0539 (8)	
H15	0.1524	0.9155	0.1076	0.065*	
C16	0.12924 (17)	1.0173 (2)	0.1718 (3)	0.0578 (8)	
H16	0.1673	1.0524	0.1955	0.069*	
C17	0.08157 (17)	1.0465 (2)	0.1871 (3)	0.0540 (8)	
C18	0.02511 (18)	0.9961 (2)	0.1529 (3)	0.0589 (9)	
H18	−0.0078	1.0178	0.1618	0.071*	
C19	0.01780 (17)	0.9126 (2)	0.1050 (3)	0.0556 (8)	
H19	−0.0199	0.8773	0.0833	0.067*	
C20	0.0414 (9)	0.7513 (8)	0.2701 (12)	0.145 (6)	0.50
N1	0.08160 (12)	0.58339 (18)	−0.12421 (18)	0.0457 (6)	
H1	0.0683 (15)	0.591 (2)	−0.1891 (11)	0.055*	
N2	0.18829 (12)	0.66033 (15)	−0.0188 (2)	0.0416 (6)	
H2	0.2078 (14)	0.673 (2)	−0.050 (2)	0.050*	
N3	0.07245 (10)	0.58278 (14)	0.01361 (17)	0.0340 (5)	
N4	0.17724 (11)	0.66263 (14)	0.11747 (18)	0.0369 (5)	
N5	0.08203 (11)	0.71459 (15)	0.11935 (17)	0.0389 (5)	
O1	−0.02299 (10)	0.60162 (15)	−0.15411 (15)	0.0513 (5)	
O2	0.22887 (10)	0.78421 (13)	0.09463 (17)	0.0487 (5)	
O3	0.15792 (14)	0.44551 (19)	−0.1224 (2)	0.0829 (8)	
O4	0.24576 (13)	0.4891 (2)	0.0336 (3)	0.0911 (9)	
O5	0.21328 (12)	0.50418 (16)	0.23280 (18)	0.0673 (7)	
O6	0.14464 (12)	0.42050 (14)	0.08704 (17)	0.0559 (6)	
O7	0.0000	0.6840 (8)	0.2500	0.259 (6)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1095 (4)	0.0501 (2)	0.0870 (4)	−0.00416 (19)	0.0558 (3)	−0.00919 (18)
C1	0.0356 (17)	0.0446 (15)	0.0341 (16)	−0.0026 (12)	0.0182 (14)	−0.0042 (12)
C2	0.0352 (15)	0.0418 (16)	0.0393 (17)	−0.0030 (12)	0.0227 (14)	−0.0032 (12)
C3	0.0356 (15)	0.0450 (15)	0.0316 (15)	−0.0021 (12)	0.0208 (13)	−0.0032 (11)
C4	0.052 (2)	0.0520 (18)	0.057 (2)	−0.0044 (15)	0.0381 (18)	−0.0108 (16)
C5	0.081 (6)	0.132 (8)	0.126 (8)	0.021 (5)	0.077 (6)	−0.005 (6)
C6	0.086 (6)	0.105 (7)	0.137 (8)	−0.007 (5)	0.069 (6)	−0.037 (6)
C5'	0.107 (7)	0.102 (7)	0.104 (7)	0.075 (5)	0.072 (6)	0.024 (5)
C6'	0.136 (9)	0.162 (9)	0.117 (8)	0.057 (7)	0.070 (7)	−0.021 (7)
C7	0.0320 (14)	0.0369 (14)	0.0297 (14)	−0.0039 (11)	0.0168 (12)	−0.0024 (11)
C8	0.0374 (16)	0.0472 (17)	0.0387 (18)	0.0026 (13)	0.0201 (15)	−0.0001 (13)
C9	0.090 (3)	0.050 (2)	0.073 (3)	0.0143 (18)	0.047 (2)	0.0213 (17)
C10	0.151 (4)	0.046 (2)	0.126 (4)	0.011 (2)	0.091 (4)	0.007 (2)
C11	0.0394 (16)	0.0507 (16)	0.0359 (16)	−0.0030 (13)	0.0251 (14)	−0.0018 (13)
C12	0.0473 (17)	0.0447 (16)	0.0292 (15)	−0.0055 (13)	0.0228 (14)	−0.0068 (12)
C13	0.0502 (18)	0.0537 (18)	0.0413 (17)	0.0043 (14)	0.0278 (15)	0.0044 (14)
C14	0.0458 (17)	0.0475 (16)	0.0413 (17)	0.0045 (14)	0.0244 (15)	0.0063 (14)
C15	0.0523 (19)	0.0483 (18)	0.072 (2)	0.0076 (14)	0.0412 (19)	0.0126 (16)
C16	0.0521 (19)	0.0439 (18)	0.074 (2)	−0.0026 (14)	0.0331 (18)	0.0085 (16)
C17	0.068 (2)	0.0418 (17)	0.0489 (19)	0.0032 (16)	0.0308 (18)	0.0037 (14)
C18	0.062 (2)	0.060 (2)	0.065 (2)	0.0054 (17)	0.0413 (19)	−0.0040 (17)
C19	0.053 (2)	0.061 (2)	0.059 (2)	−0.0059 (15)	0.0346 (18)	−0.0066 (16)
C20	0.264 (17)	0.088 (7)	0.205 (14)	−0.024 (9)	0.206 (15)	−0.019 (8)
N1	0.0354 (14)	0.0769 (18)	0.0236 (13)	−0.0032 (12)	0.0157 (12)	−0.0052 (12)
N2	0.0499 (15)	0.0457 (13)	0.0437 (15)	−0.0092 (11)	0.0350 (13)	−0.0068 (11)
N3	0.0311 (12)	0.0439 (13)	0.0284 (12)	−0.0023 (9)	0.0174 (10)	−0.0004 (9)
N4	0.0376 (13)	0.0408 (12)	0.0349 (13)	−0.0068 (9)	0.0217 (11)	−0.0069 (9)
N5	0.0408 (14)	0.0434 (13)	0.0363 (13)	−0.0005 (10)	0.0240 (12)	−0.0021 (10)
O1	0.0347 (12)	0.0783 (16)	0.0351 (11)	0.0017 (10)	0.0159 (10)	−0.0034 (10)
O2	0.0554 (13)	0.0481 (12)	0.0547 (13)	−0.0153 (10)	0.0381 (11)	−0.0126 (10)
O3	0.105 (2)	0.0745 (17)	0.089 (2)	−0.0117 (15)	0.0669 (18)	−0.0389 (15)
O4	0.0538 (16)	0.0819 (19)	0.118 (2)	0.0206 (13)	0.0355 (17)	−0.0182 (17)
O5	0.0646 (15)	0.0651 (15)	0.0386 (14)	0.0072 (12)	0.0082 (13)	0.0049 (11)
O6	0.0678 (14)	0.0384 (11)	0.0448 (12)	−0.0010 (10)	0.0211 (12)	0.0051 (9)
O7	0.349 (14)	0.218 (10)	0.433 (17)	0.000	0.356 (15)	0.000

Geometric parameters (Å, °)

Br1—C17	1.908 (3)	C9—O6	1.452 (4)
C1—O1	1.221 (3)	C9—H9A	0.9700
C1—N1	1.361 (4)	C9—H9B	0.9700
C1—N3	1.369 (4)	C10—H10A	0.9600
C2—O2	1.220 (3)	C10—H10B	0.9600
C2—N2	1.359 (3)	C10—H10C	0.9600
C2—N4	1.373 (3)	C11—N5	1.453 (4)

C3—N1	1.425 (4)	C11—N3	1.466 (3)
C3—N2	1.439 (3)	C11—H11A	0.9700
C3—C4	1.528 (4)	C11—H11B	0.9700
C3—C7	1.576 (4)	C12—N5	1.455 (4)
C4—O3	1.177 (4)	C12—N4	1.467 (3)
C4—O4	1.307 (4)	C12—H12A	0.9700
C5—C6	1.487 (9)	C12—H12B	0.9700
C5—O4	1.537 (8)	C13—N5	1.475 (4)
C5—H5A	0.9700	C13—C14	1.512 (4)
C5—H5B	0.9700	C13—H13A	0.9700
C6—H6A	0.9600	C13—H13B	0.9700
C6—H6B	0.9600	C14—C15	1.379 (4)
C6—H6C	0.9600	C14—C19	1.390 (4)
C5'—C6'	1.487 (9)	C15—C16	1.388 (5)
C5'—O4	1.541 (7)	C15—H15	0.9300
C5'—H5'1	0.9700	C16—C17	1.359 (5)
C5'—H5'2	0.9700	C16—H16	0.9300
C6'—H6'1	0.9600	C17—C18	1.368 (5)
C6'—H6'2	0.9600	C18—C19	1.378 (4)
C6'—H6'3	0.9600	C18—H18	0.9300
C7—N4	1.448 (3)	C19—H19	0.9300
C7—N3	1.451 (3)	C20—O7	1.310 (13)
C7—C8	1.532 (4)	C20—C20^{i}	1.73 (3)
C8—O5	1.197 (3)	N1—H1	0.851 (10)
C8—O6	1.318 (3)	N2—H2	0.85 (4)
C9—C10	1.443 (6)	O7—C20^{i}	1.310 (13)
O1—C1—N1	126.9 (3)	N5—C11—H11A	109.1
O1—C1—N3	124.8 (2)	N3—C11—H11A	109.1
N1—C1—N3	108.2 (2)	N5—C11—H11B	109.1
O2—C2—N2	126.4 (2)	N3—C11—H11B	109.1
O2—C2—N4	125.1 (2)	H11A—C11—H11B	107.8
N2—C2—N4	108.5 (2)	N5—C12—N4	112.4 (2)
N1—C3—N2	115.0 (2)	N5—C12—H12A	109.1
N1—C3—C4	110.2 (2)	N4—C12—H12A	109.1
N2—C3—C4	111.0 (2)	N5—C12—H12B	109.1
N1—C3—C7	102.07 (19)	N4—C12—H12B	109.1
N2—C3—C7	101.9 (2)	H12A—C12—H12B	107.9
C4—C3—C7	116.4 (2)	N5—C13—C14	110.6 (2)
O3—C4—O4	125.2 (3)	N5—C13—H13A	109.5
O3—C4—C3	124.6 (3)	C14—C13—H13A	109.5
O4—C4—C3	110.1 (3)	N5—C13—H13B	109.5
C6—C5—O4	100.0 (7)	C14—C13—H13B	109.5
C6—C5—H5A	111.8	H13A—C13—H13B	108.1
O4—C5—H5A	111.8	C15—C14—C19	118.0 (3)
C6—C5—H5B	111.8	C15—C14—C13	122.2 (3)
O4—C5—H5B	111.8	C19—C14—C13	119.8 (3)
H5A—C5—H5B	109.5	C14—C15—C16	120.8 (3)
C6'—C5'—O4	105.4 (7)	C14—C15—H15	119.6

C6'—C5'—H5'1	110.7	C16—C15—H15	119.6
O4—C5'—H5'1	110.7	C17—C16—C15	119.4 (3)
C6'—C5'—H5'2	110.7	C17—C16—H16	120.3
O4—C5'—H5'2	110.7	C15—C16—H16	120.3
H5'1—C5'—H5'2	108.8	C16—C17—C18	121.4 (3)
C5'—C6'—H6'1	109.5	C16—C17—Br1	119.8 (3)
C5'—C6'—H6'2	109.5	C18—C17—Br1	118.8 (2)
H6'1—C6'—H6'2	109.5	C17—C18—C19	118.9 (3)
C5'—C6'—H6'3	109.5	C17—C18—H18	120.5
H6'1—C6'—H6'3	109.5	C19—C18—H18	120.5
H6'2—C6'—H6'3	109.5	C18—C19—C14	121.3 (3)
N4—C7—N3	111.9 (2)	C18—C19—H19	119.3
N4—C7—C8	110.5 (2)	C14—C19—H19	119.3
N3—C7—C8	108.6 (2)	O7—C20—C20^{i}	48.6 (8)
N4—C7—C3	103.8 (2)	C1—N1—C3	114.2 (2)
N3—C7—C3	103.5 (2)	C1—N1—H1	120 (2)
C8—C7—C3	118.4 (2)	C3—N1—H1	125 (2)
O5—C8—O6	125.7 (3)	C2—N2—C3	113.9 (2)
O5—C8—C7	123.9 (3)	C2—N2—H2	121 (2)
O6—C8—C7	110.3 (2)	C3—N2—H2	124 (2)
C10—C9—O6	108.7 (3)	C1—N3—C7	111.6 (2)
C10—C9—H9A	110.0	C1—N3—C11	123.1 (2)
O6—C9—H9A	110.0	C7—N3—C11	116.1 (2)
C10—C9—H9B	110.0	C2—N4—C7	111.7 (2)
O6—C9—H9B	110.0	C2—N4—C12	124.6 (2)
H9A—C9—H9B	108.3	C7—N4—C12	116.7 (2)
C9—C10—H10A	109.5	C11—N5—C12	109.3 (2)
C9—C10—H10B	109.5	C11—N5—C13	114.3 (2)
H10A—C10—H10B	109.5	C12—N5—C13	112.8 (2)
C9—C10—H10C	109.5	C4—O4—C5	123.9 (6)
H10A—C10—H10C	109.5	C4—O4—C5'	109.8 (5)
H10B—C10—H10C	109.5	C8—O6—C9	116.8 (3)
N5—C11—N3	112.6 (2)	C20^{i}—O7—C20	82.7 (16)
N1—C3—C4—O3	4.3 (4)	C7—C3—N2—C2	−4.5 (3)
N2—C3—C4—O3	−124.3 (3)	O1—C1—N3—C7	−175.6 (3)
C7—C3—C4—O3	119.8 (3)	N1—C1—N3—C7	6.7 (3)
N1—C3—C4—O4	−177.6 (3)	O1—C1—N3—C11	−30.3 (4)
N2—C3—C4—O4	53.8 (3)	N1—C1—N3—C11	152.1 (2)
C7—C3—C4—O4	−62.1 (3)	N4—C7—N3—C1	106.8 (2)
N1—C3—C7—N4	−116.5 (2)	C8—C7—N3—C1	−131.0 (2)
N2—C3—C7—N4	2.6 (2)	C3—C7—N3—C1	−4.4 (3)
C4—C3—C7—N4	123.5 (2)	N4—C7—N3—C11	−41.3 (3)
N1—C3—C7—N3	0.5 (3)	C8—C7—N3—C11	81.0 (3)
N2—C3—C7—N3	119.6 (2)	C3—C7—N3—C11	−152.4 (2)
C4—C3—C7—N3	−119.6 (2)	N5—C11—N3—C1	−93.4 (3)
N1—C3—C7—C8	120.7 (2)	N5—C11—N3—C7	50.6 (3)
N2—C3—C7—C8	−120.2 (2)	O2—C2—N4—C7	177.3 (3)
C4—C3—C7—C8	0.6 (3)	N2—C2—N4—C7	−2.6 (3)

N4—C7—C8—O5	5.2 (4)	O2—C2—N4—C12	27.9 (4)
N3—C7—C8—O5	−117.8 (3)	N2—C2—N4—C12	−152.0 (2)
C3—C7—C8—O5	124.7 (3)	N3—C7—N4—C2	−111.1 (2)
N4—C7—C8—O6	−177.2 (2)	C8—C7—N4—C2	127.8 (2)
N3—C7—C8—O6	59.7 (3)	C3—C7—N4—C2	−0.2 (3)
C3—C7—C8—O6	−57.8 (3)	N3—C7—N4—C12	41.0 (3)
N5—C13—C14—C15	94.6 (3)	C8—C7—N4—C12	−80.2 (3)
N5—C13—C14—C19	−84.5 (3)	C3—C7—N4—C12	151.9 (2)
C19—C14—C15—C16	2.6 (5)	N5—C12—N4—C2	98.4 (3)
C13—C14—C15—C16	−176.5 (3)	N5—C12—N4—C7	−49.7 (3)
C14—C15—C16—C17	−2.3 (5)	N3—C11—N5—C12	−55.9 (3)
C15—C16—C17—C18	−0.1 (5)	N3—C11—N5—C13	71.5 (3)
C15—C16—C17—Br1	178.6 (2)	N4—C12—N5—C11	55.3 (3)
C16—C17—C18—C19	2.1 (5)	N4—C12—N5—C13	−73.1 (3)
Br1—C17—C18—C19	−176.7 (3)	C14—C13—N5—C11	154.6 (2)
C17—C18—C19—C14	−1.7 (5)	C14—C13—N5—C12	−79.8 (3)
C15—C14—C19—C18	−0.6 (5)	O3—C4—O4—C5	−15.6 (7)
C13—C14—C19—C18	178.5 (3)	C3—C4—O4—C5	166.3 (5)
O1—C1—N1—C3	175.9 (3)	O3—C4—O4—C5'	16.1 (7)
N3—C1—N1—C3	−6.5 (3)	C3—C4—O4—C5'	−162.0 (6)
N2—C3—N1—C1	−105.8 (3)	C6—C5—O4—C4	116.7 (8)
C4—C3—N1—C1	127.9 (3)	C6—C5—O4—C5'	45.3 (12)
C7—C3—N1—C1	3.6 (3)	C6'—C5'—O4—C4	−144.8 (11)
O2—C2—N2—C3	−175.3 (3)	C6'—C5'—O4—C5	−21.6 (12)
N4—C2—N2—C3	4.6 (3)	O5—C8—O6—C9	2.6 (5)
N1—C3—N2—C2	105.0 (3)	C7—C8—O6—C9	−174.8 (3)
C4—C3—N2—C2	−129.0 (3)	C10—C9—O6—C8	−151.3 (3)

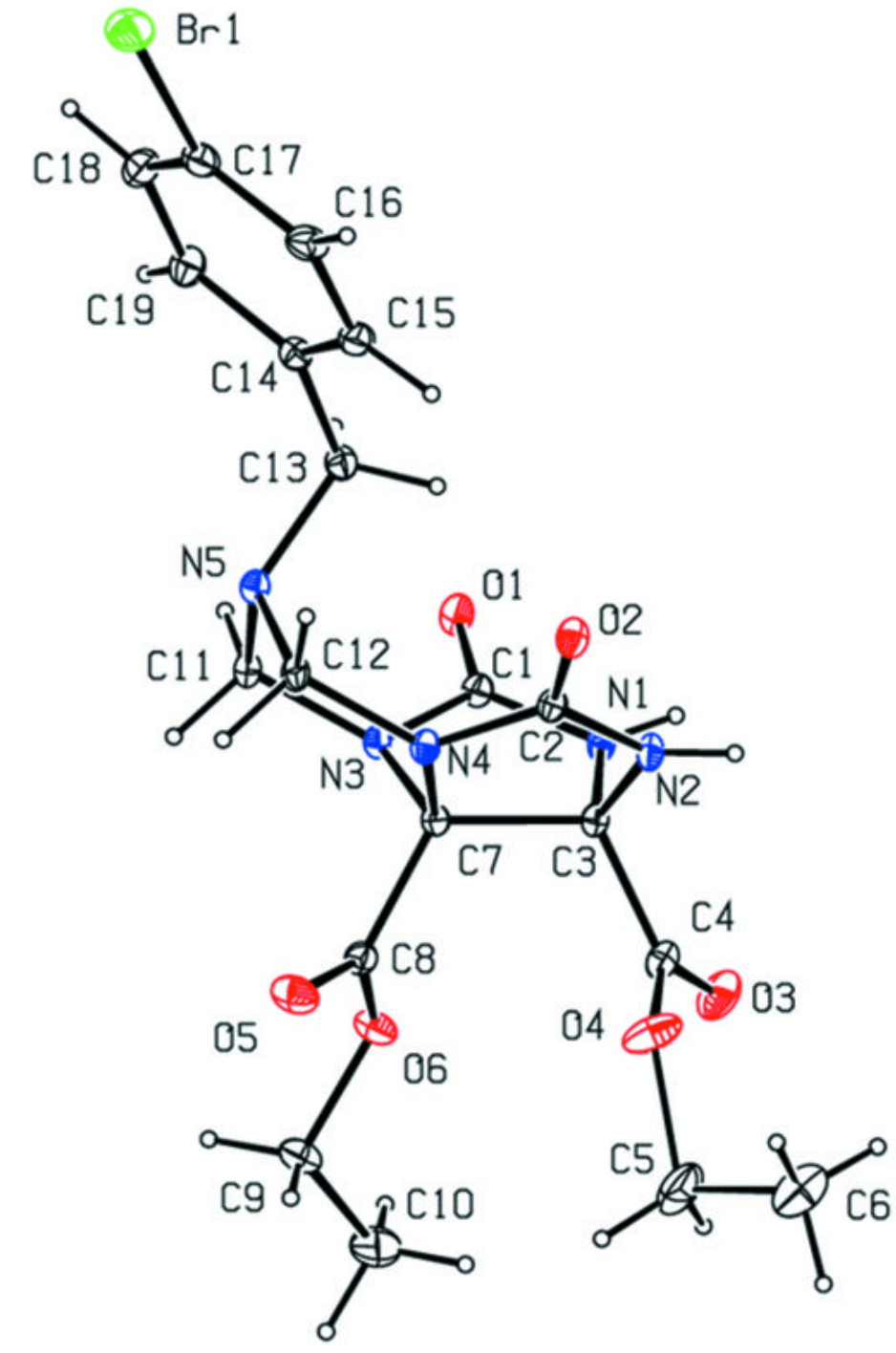
Symmetry codes: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O5^{ii}	0.93	2.54	3.222 (4)	130
C9—H9A···O2^{iii}	0.97	2.56	3.318 (5)	136
N2—H2···O2^{iv}	0.85 (4)	2.08 (4)	2.914 (3)	168 (3)
N1—H1···O1^{v}	0.852 (18)	1.98 (1)	2.828 (3)	171 (3)

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

Fig. 1


Br1
C18
C17
C16
C19
C15
C14
C13
N5
O1
O2
C11
C12
C1
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N3
N4
C2
N2
C7
C3
C4
C8
O3
O5
O4
O6
C5
C9
C10
C6

Fig. 2