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Dipropyl 4,8-dioxo-1*H*,5*H*-2,6-dioxa-3a,4a,7a,8a-tetraazacyclopenta[*def*]-fluorene-8b,8c-dicarboxylate

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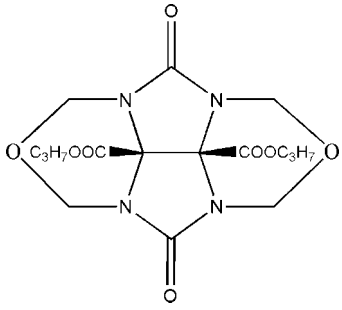
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.050; wR factor = 0.123; data-to-parameter ratio = 8.8.

The title compound, $C_{16}H_{22}N_4O_8$, is a glycoluril derivative with two propoxycarbonyl substituents on the convex face of the glycoluril system. The dihedral angle between the five-membered rings in the glycoluril unit is 72.70 (2)°. The oxadiazinane six-membered ring displays a normal chair conformation. One of the propyl groups is disordered over two positions with site occupancies of 0.557 (7) and 0.443 (7). Intermolecular C—H···O hydrogen bonds are effective in the stabilization of the crystal structure.

Related literature

For related structures, see: Branda *et al.* (1995); Elemans *et al.* (1999); Gao & Sun (2007); Isaacs & Witt (2002); Isaacs *et al.* (1999); Li *et al.* (2007); Rebek (1999); Rowan *et al.* (1999); She & Xi (2007); Witt *et al.* (2000); Wu *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{22}N_4O_8$
M_r = 398.38
Orthorhombic, $P2_12_12_1$
a = 8.6399 (4) Å
b = 13.401 (7) Å
c = 16.0445 (8) Å
V = 1857.7 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 294 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
17272 measured reflections
2315 independent reflections
1967 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.123
S = 1.07
2315 reflections
263 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O2^{i}	0.97	2.58	3.482 (5)	155
C9—H9B···O4^{ii}	0.97	2.58	3.388 (4)	141
C5—H5B···O7^{iii}	0.97	2.45	3.310 (4)	148
C9—H9A···O1^{iv}	0.97	2.50	3.364 (4)	149
C2—H2A···O3^{iv}	0.97	2.46	3.368 (4)	155
C1—H1B···O7^{v}	0.97	2.55	3.231 (4)	128

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

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).

The authors thank Dr She Neng-Fang and Meng Xiang-Gao for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2262).

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supplementary materials

Acta Cryst. (2008). E**64**, o265 [**doi:10.1107/S1600536807065853**]

Dipropyl 4,8-dioxo-1*H*,5*H*-2,6-dioxa-3a,4a,7a,8a-tetraazacyclopenta[*def*]fluorene-8b,8c-dicarboxylate

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Comment

Glycolurils are a topic of numerous reports; these compounds are of use in supramolecular chemistry as building blocks for molecular clips (Rowan *et al.*, 1999; Isaacs & Witt, 2002) and molecular capsules (Rebek, 1999). The derivatives of glycoluril have been employed in many applications, including polymer cross-linking, explosives and combinational chemistry (Witt *et al.*, 2000). The widespread interest in glycolurils has led to a variety of crystal structures reported for a number of its derivatives. Here we report the structure of the title glycoluril derivative, (I) (Fig. 1), which is an important intermediate for the preparation glycoluril receptors (Wu *et al.*, 2002) and in which the bond lengths and angles present no unusual features and are similar to those found in other similar compounds (Gao & Sun, 2007; She & Xi, 2007; Li *et al.*, 2007).

The oxadiazinane six-membered ring displays a normal chair conformation. The weak intermolecular C—H···O hydrogen bonds cause the formation of a three-dimensional network structure (Fig. 2).

Experimental

The title compound was synthesized according to the procedure reported (Isaacs *et al.*, 1999). Crystals appropriate for X-ray data collection were obtained by slow evaporation of a dichloromethane solution at 283 K.

Refinement

All H atoms were positioned geometrically, with C—H = 0.97 and 0.96 Å for methylene and methyl groups, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). One of the propyl groups is disordered over two positions and the final occupancies refined to 0.557 (7) and 0.443 (7). C15—C16 and C15'-C16' bond lengths were restrained to be 1.54 (1) Å, and C14—C15 and C14'-C15' to be 1.45 (1) Å. Same displacement parameters were used for atoms C15 and C15', and for atoms C16 and C16'. In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

Figures

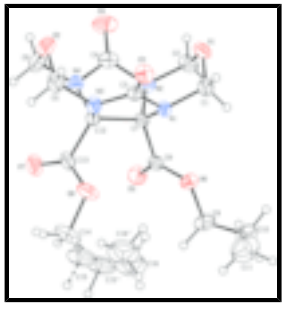

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

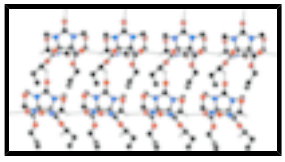

Fig. 2. A packing diagram of the title compound. C—H···O hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen-bonds have been omitted for clarity.

Dipropyl 4,8-dioxo-1H,5*H*-2,6-dioxa-3a,4a,7a,8a- tetraazacyclopenta[def]fluorene-8 b,8c-dicarboxylate

Crystal data

$C_{16}H_{22}N_4O_8$	F_{000} = 840
M_r = 398.38	D_x = 1.424 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3466 reflections
a = 8.6399 (4) Å	θ = 2.5–21.5º
b = 13.401 (7) Å	μ = 0.12 mm^{-1}
c = 16.0445 (8) Å	T = 294 (2) K
V = 1857.7 (10) Å^3	Block, colorless
Z = 4	0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1967 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.041
Monochromator: graphite	θ_{max} = 27.0º
T = 294(2) K	θ_{min} = 2.5º
φ and ω scans	h = −11→9
Absorption correction: none	k = −16→17
17272 measured reflections	l = −19→20
2315 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.2494P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.123	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.07	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
2315 reflections	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
263 parameters	Extinction correction: none
4 restraints	
Primary atom site location: structure-invariant direct methods	
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 $F^2 > 2\text{sigma}(F^2)$ 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)
C1	0.4167 (4)	0.2297 (2)	0.2432 (2)	0.0501 (8)	
H1A	0.3680	0.2906	0.2239	0.060*	
H1B	0.3651	0.1739	0.2165	0.060*	
C2	0.4676 (4)	0.1327 (2)	0.3595 (2)	0.0539 (9)	
H2A	0.4169	0.0760	0.3338	0.065*	
H2B	0.4532	0.1278	0.4193	0.065*	
C3	0.6597 (3)	0.31946 (18)	0.22249 (17)	0.0357 (6)	
C4	0.7391 (4)	0.1724 (2)	0.3941 (2)	0.0484 (8)	
C5	0.9211 (4)	0.3717 (2)	0.2653 (3)	0.0605 (10)	
H5A	1.0236	0.3599	0.2427	0.073*	
H5B	0.8884	0.4380	0.2487	0.073*	
C6	0.9781 (5)	0.2689 (3)	0.3785 (3)	0.0685 (11)	
H6A	0.9851	0.2662	0.4388	0.082*	
H6B	1.0804	0.2564	0.3559	0.082*	
C7	0.6721 (3)	0.14966 (18)	0.25409 (16)	0.0325 (6)	
C8	0.6681 (3)	0.05026 (18)	0.20526 (17)	0.0356 (6)	
C9	0.5560 (5)	−0.0444 (2)	0.0969 (2)	0.0569 (9)	
H9A	0.5447	−0.1000	0.1352	0.068*	
H9B	0.6490	−0.0549	0.0642	0.068*	
C10	0.4189 (6)	−0.0385 (3)	0.0411 (3)	0.0821 (13)	
H10A	0.3995	−0.1045	0.0187	0.098*	
H10B	0.3299	−0.0202	0.0746	0.098*	
C11	0.4299 (8)	0.0317 (5)	−0.0287 (4)	0.116 (2)	
H11A	0.4263	0.0988	−0.0080	0.174*	
H11B	0.3448	0.0209	−0.0662	0.174*	
H11C	0.5256	0.0212	−0.0578	0.174*	
C12	0.8373 (3)	0.19597 (19)	0.26092 (18)	0.0350 (6)	
C13	0.9645 (4)	0.1474 (2)	0.2084 (2)	0.0426 (7)	
C14	1.0282 (6)	0.1060 (3)	0.0683 (3)	0.0803 (14)	
H14A	1.0758	0.0455	0.0893	0.096*	0.557 (7)
H14B	1.1091	0.1541	0.0566	0.096*	0.557 (7)
H14C	1.0186	0.0339	0.0664	0.096*	0.443 (7)
H14D	1.1337	0.1222	0.0838	0.096*	0.443 (7)

C15	0.9389 (12)	0.0845 (6)	−0.0080 (5)	0.094 (3)	0.557 (7)
H15A	0.8691	0.0294	0.0033	0.113*	0.557 (7)
H15B	1.0101	0.0630	−0.0511	0.113*	0.557 (7)
C16	0.844 (2)	0.1729 (11)	−0.0413 (12)	0.130 (5)	0.557 (7)
H16A	0.7547	0.1828	−0.0065	0.194*	0.557 (7)
H16B	0.8109	0.1589	−0.0973	0.194*	0.557 (7)
H16C	0.9064	0.2320	−0.0410	0.194*	0.557 (7)
C15'	0.9949 (14)	0.1470 (8)	−0.0134 (5)	0.094 (3)	0.443 (7)
H15C	1.0705	0.1237	−0.0537	0.113*	0.443 (7)
H15D	0.9984	0.2193	−0.0117	0.113*	0.443 (7)
C16'	0.833 (2)	0.1113 (16)	−0.0377 (16)	0.130 (5)	0.443 (7)
H16D	0.8219	0.0418	−0.0245	0.194*	0.443 (7)
H16E	0.8173	0.1209	−0.0964	0.194*	0.443 (7)
H16F	0.7566	0.1490	−0.0074	0.194*	0.443 (7)
N1	0.5792 (3)	0.23013 (15)	0.21957 (15)	0.0347 (5)	
N2	0.6315 (3)	0.13036 (17)	0.34032 (15)	0.0426 (6)	
N3	0.8136 (3)	0.29806 (16)	0.23239 (16)	0.0390 (6)	
N4	0.8717 (3)	0.1929 (2)	0.34926 (16)	0.0471 (6)	
O1	0.3994 (3)	0.22198 (15)	0.33015 (16)	0.0533 (6)	
O2	0.9261 (3)	0.36549 (18)	0.35326 (18)	0.0682 (7)	
O3	0.6044 (3)	0.40152 (14)	0.21303 (15)	0.0511 (6)	
O4	0.7212 (4)	0.1841 (2)	0.46809 (15)	0.0726 (8)	
O5	0.7493 (3)	−0.01743 (15)	0.22604 (17)	0.0561 (6)	
O6	0.5676 (3)	0.05015 (13)	0.14370 (13)	0.0442 (5)	
O7	1.0852 (3)	0.11865 (18)	0.23472 (19)	0.0643 (7)	
O8	0.9201 (3)	0.14622 (19)	0.12965 (14)	0.0631 (7)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0357 (17)	0.0445 (16)	0.070 (2)	0.0030 (13)	−0.0040 (16)	−0.0090 (14)
C2	0.058 (2)	0.0406 (15)	0.063 (2)	−0.0100 (15)	0.0269 (18)	−0.0051 (14)
C3	0.0426 (17)	0.0291 (12)	0.0355 (14)	0.0015 (11)	0.0017 (13)	0.0004 (10)
C4	0.066 (2)	0.0432 (16)	0.0356 (17)	0.0154 (15)	−0.0011 (16)	0.0018 (12)
C5	0.046 (2)	0.0427 (16)	0.093 (3)	−0.0112 (14)	0.003 (2)	−0.0124 (17)
C6	0.054 (2)	0.075 (2)	0.076 (3)	0.0048 (19)	−0.026 (2)	−0.023 (2)
C7	0.0319 (15)	0.0294 (12)	0.0362 (14)	0.0018 (10)	0.0036 (12)	−0.0029 (10)
C8	0.0348 (15)	0.0306 (12)	0.0413 (16)	0.0015 (11)	0.0051 (13)	−0.0027 (11)
C9	0.076 (3)	0.0388 (15)	0.056 (2)	−0.0057 (17)	0.0001 (19)	−0.0154 (14)
C10	0.076 (3)	0.081 (3)	0.089 (3)	−0.009 (2)	−0.007 (3)	−0.036 (2)
C11	0.133 (5)	0.117 (4)	0.099 (4)	0.021 (4)	−0.035 (4)	−0.004 (3)
C12	0.0306 (15)	0.0321 (12)	0.0421 (15)	0.0002 (11)	−0.0016 (12)	−0.0021 (11)
C13	0.0306 (16)	0.0367 (13)	0.060 (2)	0.0026 (12)	0.0053 (14)	−0.0013 (13)
C14	0.085 (3)	0.081 (3)	0.075 (3)	0.026 (2)	0.043 (3)	−0.002 (2)
C15	0.126 (7)	0.078 (5)	0.079 (5)	−0.022 (4)	0.064 (5)	−0.015 (4)
C16	0.151 (8)	0.170 (15)	0.068 (4)	0.011 (13)	0.023 (5)	−0.002 (12)
C15'	0.126 (7)	0.078 (5)	0.079 (5)	−0.022 (4)	0.064 (5)	−0.015 (4)
C16'	0.151 (8)	0.170 (15)	0.068 (4)	0.011 (13)	0.023 (5)	−0.002 (12)

N1	0.0300 (12)	0.0312 (10)	0.0429 (13)	0.0033 (9)	−0.0012 (11)	−0.0021 (9)
N2	0.0523 (16)	0.0358 (11)	0.0397 (13)	−0.0004 (11)	0.0086 (12)	0.0027 (10)
N3	0.0336 (13)	0.0293 (10)	0.0542 (14)	−0.0028 (9)	0.0012 (11)	−0.0011 (10)
N4	0.0431 (15)	0.0521 (14)	0.0461 (15)	0.0096 (12)	−0.0126 (13)	−0.0050 (12)
O1	0.0414 (13)	0.0464 (11)	0.0721 (16)	−0.0010 (10)	0.0205 (11)	−0.0158 (10)
O2	0.0538 (16)	0.0610 (14)	0.0899 (19)	−0.0064 (12)	−0.0132 (15)	−0.0325 (13)
O3	0.0548 (15)	0.0308 (9)	0.0676 (14)	0.0080 (9)	−0.0019 (12)	0.0057 (9)
O4	0.098 (2)	0.0832 (17)	0.0361 (13)	0.0226 (17)	−0.0014 (14)	0.0007 (12)
O5	0.0535 (13)	0.0359 (10)	0.0788 (16)	0.0124 (10)	−0.0078 (13)	−0.0107 (10)
O6	0.0537 (13)	0.0353 (9)	0.0435 (11)	−0.0003 (9)	−0.0031 (11)	−0.0082 (8)
O7	0.0360 (14)	0.0604 (14)	0.0966 (19)	0.0096 (11)	−0.0057 (14)	−0.0154 (13)
O8	0.0587 (16)	0.0793 (16)	0.0515 (14)	0.0268 (13)	0.0173 (13)	−0.0024 (12)

Geometric parameters (Å, °)

C1—O1	1.407 (4)	C9—H9B	0.9700
C1—N1	1.454 (4)	C10—C11	1.467 (7)
C1—H1A	0.9700	C10—H10A	0.9700
C1—H1B	0.9700	C10—H10B	0.9700
C2—O1	1.414 (4)	C11—H11A	0.9600
C2—N2	1.449 (4)	C11—H11B	0.9600
C2—H2A	0.9700	C11—H11C	0.9600
C2—H2B	0.9700	C12—N4	1.449 (4)
C3—O3	1.209 (3)	C12—N3	1.457 (3)
C3—N3	1.369 (4)	C12—C13	1.530 (4)
C3—N1	1.385 (3)	C13—O7	1.189 (4)
C4—O4	1.207 (4)	C13—O8	1.321 (4)
C4—N4	1.381 (5)	C14—C15'	1.450 (8)
C4—N2	1.388 (4)	C14—O8	1.460 (4)
C5—O2	1.414 (5)	C14—C15	1.475 (8)
C5—N3	1.455 (4)	C14—H14A	0.9700
C5—H5A	0.9700	C14—H14B	0.9700
C5—H5B	0.9700	C14—H14C	0.9700
C6—O2	1.429 (5)	C14—H14D	0.9700
C6—N4	1.450 (4)	C15—C16	1.538 (9)
C6—H6A	0.9700	C15—H15A	0.9700
C6—H6B	0.9700	C15—H15B	0.9700
C7—N2	1.451 (4)	C16—H16A	0.9600
C7—N1	1.454 (3)	C16—H16B	0.9600
C7—C8	1.546 (3)	C16—H16C	0.9600
C7—C12	1.561 (4)	C15'—C16'	1.532 (10)
C8—O5	1.194 (3)	C15'—H15C	0.9700
C8—O6	1.315 (3)	C15'—H15D	0.9700
C9—O6	1.477 (3)	C16'—H16D	0.9600
C9—C10	1.486 (6)	C16'—H16E	0.9600
C9—H9A	0.9700	C16'—H16F	0.9600
O1—C1—N1	111.2 (3)	N3—C12—C13	109.1 (2)
O1—C1—H1A	109.4	N4—C12—C7	104.2 (2)
N1—C1—H1A	109.4	N3—C12—C7	102.9 (2)

O1—C1—H1B	109.4	C13—C12—C7	116.7 (2)
N1—C1—H1B	109.4	O7—C13—O8	126.1 (3)
H1A—C1—H1B	108.0	O7—C13—C12	124.9 (3)
O1—C2—N2	110.8 (3)	O8—C13—C12	108.9 (3)
O1—C2—H2A	109.5	C15'—C14—O8	110.1 (5)
N2—C2—H2A	109.5	O8—C14—C15	107.3 (5)
O1—C2—H2B	109.5	C15'—C14—H14A	135.6
N2—C2—H2B	109.5	O8—C14—H14A	110.3
H2A—C2—H2B	108.1	C15—C14—H14A	110.3
O3—C3—N3	126.1 (3)	C15'—C14—H14B	73.5
O3—C3—N1	125.7 (3)	O8—C14—H14B	110.3
N3—C3—N1	108.1 (2)	C15—C14—H14B	110.3
O4—C4—N4	126.4 (4)	H14A—C14—H14B	108.5
O4—C4—N2	125.3 (4)	C15'—C14—H14C	109.4
N4—C4—N2	108.2 (3)	O8—C14—H14C	109.5
O2—C5—N3	110.0 (3)	C15'—C14—H14D	109.4
O2—C5—H5A	109.7	O8—C14—H14D	110.2
N3—C5—H5A	109.7	H14C—C14—H14D	108.1
O2—C5—H5B	109.7	C14—C15—C16	114.7 (9)
N3—C5—H5B	109.7	C16—C15—H14C	148.2
H5A—C5—H5B	108.2	C14—C15—H15A	108.6
O2—C6—N4	110.2 (3)	C16—C15—H15A	108.6
O2—C6—H6A	109.6	H14C—C15—H15A	78.3
N4—C6—H6A	109.6	C14—C15—H15B	108.6
O2—C6—H6B	109.6	C16—C15—H15B	108.6
N4—C6—H6B	109.6	H14C—C15—H15B	98.0
H6A—C6—H6B	108.1	H15A—C15—H15B	107.6
N2—C7—N1	111.2 (2)	C14—C15'—C16'	107.1 (12)
N2—C7—C8	108.9 (2)	C14—C15'—H15C	110.3
N1—C7—C8	115.7 (2)	C16'—C15'—H15C	110.3
N2—C7—C12	103.0 (2)	C14—C15'—H15D	110.3
N1—C7—C12	103.7 (2)	C16'—C15'—H15D	110.3
C8—C7—C12	113.5 (2)	H15C—C15'—H15D	108.6
O5—C8—O6	126.6 (2)	C15'—C16'—H16D	109.5
O5—C8—C7	120.0 (3)	C15'—C16'—H16E	109.5
O6—C8—C7	113.3 (2)	H16D—C16'—H16E	109.5
O6—C9—C10	108.3 (3)	C15'—C16'—H16F	109.5
O6—C9—H9A	110.0	H16D—C16'—H16F	109.5
C10—C9—H9A	110.0	H16E—C16'—H16F	109.5
O6—C9—H9B	110.0	C3—N1—C1	118.7 (2)
C10—C9—H9B	110.0	C3—N1—C7	110.5 (2)
H9A—C9—H9B	108.4	C1—N1—C7	115.5 (2)
C11—C10—C9	116.3 (4)	C4—N2—C2	120.9 (3)
C11—C10—H10A	108.2	C4—N2—C7	111.0 (2)
C9—C10—H10A	108.2	C2—N2—C7	115.8 (3)
C11—C10—H10B	108.2	C3—N3—C5	121.3 (2)
C9—C10—H10B	108.2	C3—N3—C12	111.7 (2)
H10A—C10—H10B	107.4	C5—N3—C12	115.7 (2)
C10—C11—H11A	109.5	C4—N4—C12	110.2 (3)

C10—C11—H11B	109.5	C4—N4—C6	119.8 (3)
H11A—C11—H11B	109.5	C12—N4—C6	115.3 (3)
C10—C11—H11C	109.5	C1—O1—C2	110.4 (2)
H11A—C11—H11C	109.5	C5—O2—C6	110.2 (3)
H11B—C11—H11C	109.5	C8—O6—C9	115.3 (2)
N4—C12—N3	111.3 (2)	C13—O8—C14	117.6 (3)
N4—C12—C13	112.3 (2)		
N2—C7—C8—O5	−59.0 (4)	O1—C2—N2—C7	−51.7 (3)
N1—C7—C8—O5	174.8 (3)	N1—C7—N2—C4	−101.6 (3)
C12—C7—C8—O5	55.1 (4)	C8—C7—N2—C4	129.7 (2)
N2—C7—C8—O6	119.5 (3)	C12—C7—N2—C4	8.9 (3)
N1—C7—C8—O6	−6.6 (3)	N1—C7—N2—C2	41.1 (3)
C12—C7—C8—O6	−126.4 (3)	C8—C7—N2—C2	−87.6 (3)
O6—C9—C10—C11	69.0 (5)	C12—C7—N2—C2	151.7 (2)
N2—C7—C12—N4	2.4 (2)	O3—C3—N3—C5	25.0 (5)
N1—C7—C12—N4	118.5 (2)	N1—C3—N3—C5	−158.9 (3)
C8—C7—C12—N4	−115.2 (2)	O3—C3—N3—C12	167.0 (3)
N2—C7—C12—N3	−113.8 (2)	N1—C3—N3—C12	−17.0 (3)
N1—C7—C12—N3	2.3 (3)	O2—C5—N3—C3	88.6 (3)
C8—C7—C12—N3	128.6 (2)	O2—C5—N3—C12	−52.0 (4)
N2—C7—C12—C13	126.8 (2)	N4—C12—N3—C3	−102.3 (3)
N1—C7—C12—C13	−117.1 (2)	C13—C12—N3—C3	133.3 (3)
C8—C7—C12—C13	9.2 (3)	C7—C12—N3—C3	8.7 (3)
N4—C12—C13—O7	−6.8 (4)	N4—C12—N3—C5	42.0 (4)
N3—C12—C13—O7	117.0 (3)	C13—C12—N3—C5	−82.4 (3)
C7—C12—C13—O7	−127.0 (3)	C7—C12—N3—C5	153.0 (3)
N4—C12—C13—O8	175.3 (2)	O4—C4—N4—C12	−164.3 (3)
N3—C12—C13—O8	−60.8 (3)	N2—C4—N4—C12	19.2 (3)
C7—C12—C13—O8	55.1 (3)	O4—C4—N4—C6	−27.0 (5)
C15'—C14—C15—C16	−47.2 (12)	N2—C4—N4—C6	156.5 (3)
O8—C14—C15—C16	53.5 (10)	N3—C12—N4—C4	97.1 (3)
O8—C14—C15'—C16'	−63.6 (12)	C13—C12—N4—C4	−140.3 (2)
C15—C14—C15'—C16'	29.3 (12)	C7—C12—N4—C4	−13.1 (3)
O3—C3—N1—C1	−28.7 (4)	N3—C12—N4—C6	−42.3 (4)
N3—C3—N1—C1	155.2 (3)	C13—C12—N4—C6	80.3 (3)
O3—C3—N1—C7	−165.5 (3)	C7—C12—N4—C6	−152.5 (3)
N3—C3—N1—C7	18.4 (3)	O2—C6—N4—C4	−82.6 (4)
O1—C1—N1—C3	−83.7 (3)	O2—C6—N4—C12	52.7 (4)
O1—C1—N1—C7	51.1 (3)	N1—C1—O1—C2	−60.0 (3)
N2—C7—N1—C3	97.6 (3)	N2—C2—O1—C1	60.1 (4)
C8—C7—N1—C3	−137.4 (2)	N3—C5—O2—C6	60.9 (4)
C12—C7—N1—C3	−12.4 (3)	N4—C6—O2—C5	−61.5 (4)
N2—C7—N1—C1	−40.6 (3)	O5—C8—O6—C9	0.3 (4)
C8—C7—N1—C1	84.4 (3)	C7—C8—O6—C9	−178.1 (3)
C12—C7—N1—C1	−150.7 (2)	C10—C9—O6—C8	169.5 (3)
O4—C4—N2—C2	25.2 (5)	O7—C13—O8—C14	0.0 (5)
N4—C4—N2—C2	−158.2 (3)	C12—C13—O8—C14	177.8 (3)
O4—C4—N2—C7	165.8 (3)	C15'—C14—O8—C13	−155.8 (6)
N4—C4—N2—C7	−17.6 (3)	C15—C14—O8—C13	163.3 (4)

O1—C2—N2—C4	87.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14A···O2^{i}	0.97	2.58	3.482 (5)	155
C9—H9B···O4^{ii}	0.97	2.58	3.388 (4)	141
C5—H5B···O7^{iii}	0.97	2.45	3.310 (4)	148
C9—H9A···O1^{iv}	0.97	2.50	3.364 (4)	149
C2—H2A···O3^{iv}	0.97	2.46	3.368 (4)	155
C1—H1B···O7^{v}	0.97	2.55	3.231 (4)	128

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

Fig. 1

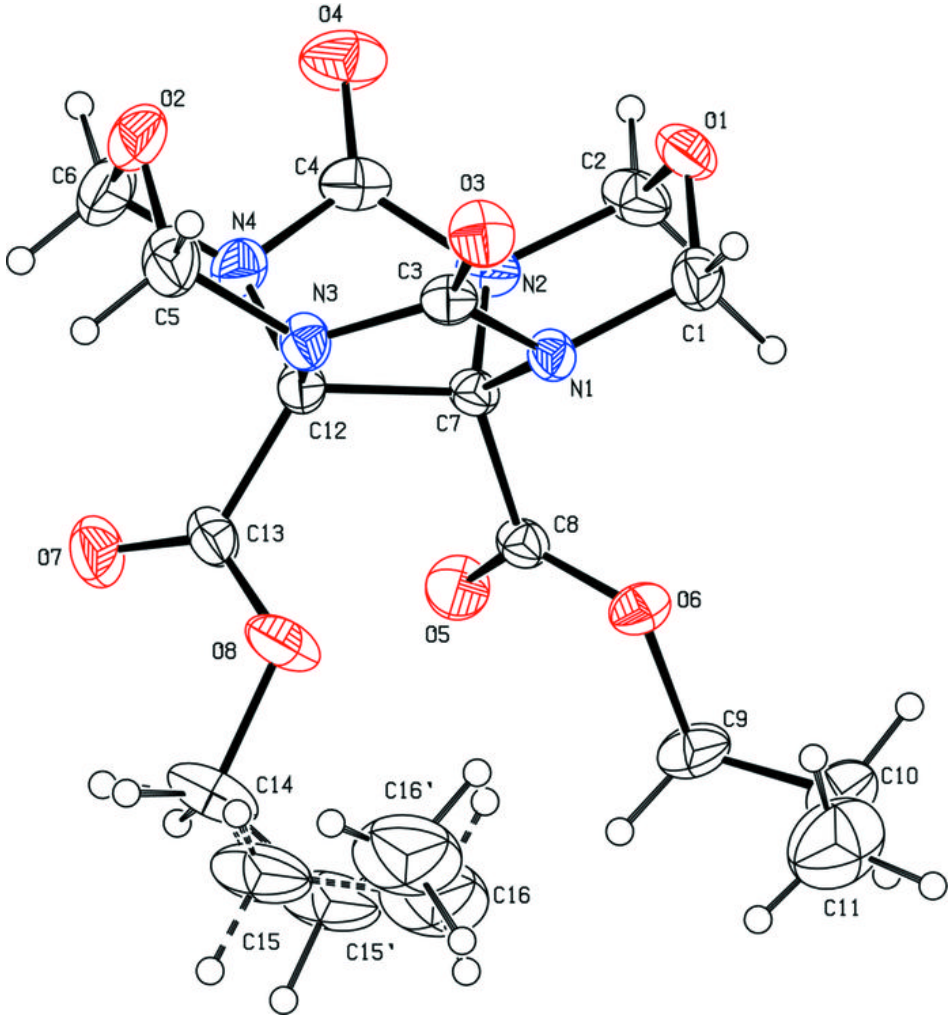
O4
O2
O1
C6
C4
C2
O3
N4
C3
N2
N3
C5
C1
N1
C12
C7
C13
C8
O7
O5
O6
O8
C9
C14
C16'
C10
C16
C11
C15
C15'

Fig. 2