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

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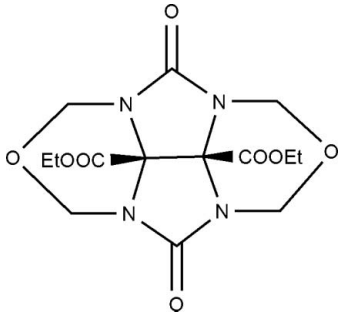
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.042; wR factor = 0.105; data-to-parameter ratio = 8.2.

The title compound, $C_{14}H_{18}N_4O_8$, is a glycoluril derivative, composed of two five-membered rings in envelope conformations and two six-membered rings in chair conformations. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules into a three-dimensional network. One methyl group and the H atoms on the adjacent C atom are disordered over two positions; the site-occupancy factors are *ca* 0.62 and 0.38.

Related literature

For related literature, see: Wu *et al.* (2002); Hof *et al.* (2002); van Nunen & Nolte (1997); Rowan *et al.* (1999); Burnett *et al.* (2003); Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Li *et al.* (2007); Cao *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{18}N_4O_8$
M_r = 370.32
Monoclinic, $P2_1$
a = 8.3182 (11) Å
b = 12.4523 (17) Å
c = 9.0479 (12) Å
β = 111.429 (2)°
V = 872.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4 K CCD area-detector diffractometer
Absorption correction: none
5178 measured reflections
1983 independent reflections
1764 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.105
S = 1.11
1983 reflections
242 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 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
C1—H1A···O8^{i}	0.97	2.56	3.223 (4)	125
C1—H1B···O2^{ii}	0.97	2.55	3.404 (4)	147
C2—H2A···O3^{iii}	0.97	2.38	3.183 (4)	140
C8—H8A···O1^{iv}	0.96	2.59	3.345 (12)	136

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

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: HK2272).

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supplementary materials

Acta Cryst. (2007). E**63**, o3495 [**doi:10.1107/S1600536807033739**]

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

N.-F. She and H.-L. Xi

Comment

The glycoluril skeleton has served as an important building block for the preparation of a wide variety of supramolecular assemblies (Wu *et al.*, 2002; Hof *et al.*, 2002). The title compound is a glycoluril derivative, and is an important intermediate for the preparation of molecular clips (van Nunen *et al.*, 1997). The molecular clip is a molecule with a rigid U-shaped cavity in which small aromatic guest molecules can be complexed by hydrogen bonding and aromatic stacking interactions (Rowan *et al.*, 1999). We report herein the crystal structure of the title glycoluril derivative, (I).

In the molecule of (I), (Fig. 1) the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987) and are in accordance with the corresponding values in similar compounds (Li *et al.*, 2007; Cao *et al.*, 2006). The rings A (O1/N1/N2/C1/C2/C5) and B (O4/N3/N4/C9/C13/C14) are not planar having total puckering amplitudes, Q_T of 1.844 (3) Å and 2.367 (3) Å, respectively, and chair conformations [φ = −28.16 (2)°, θ = 58.42 (4)° and φ = −90.10 (3)°, θ = 91.04 (3)°] (Cremer & Pople, 1975). Rings C (N1/N3/C3/C5/C9) and D (N2/N4/C4/C5/C9) have pseudo twofold axis passing through atom C9 and the mid-point of C3—N1 bond (for ring C) and atom C5 and the mid-point of C4—N4 bond (for ring D), as can be deduced from the torsion angles (Table 1). The conformations of rings C and D are envelopes, with atoms C3 and C4 at the flap positions, 0.241 (4) Å and 0.231 (3) Å from the mean planes through the other four atoms, respectively.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 2) link the molecules into a three dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

The title compound was synthesized according to the reported procedure (Burnett *et al.*, 2003). Crystals appropriate for X-ray data collection were obtained by slow evaporation of the dichloromethane solution at 283 K.

Refinement

When the crystal structure was solved, the atoms H7A, H7B, C8, H8A, H8B and H8C were found to be disordered. During refinement with isotropic thermal parameters, the occupancies of disordered H atoms were refined as H7A = 0.379 (16), H7C = 0.621 (16), H7B = 0.379 (16), H7D = 0.621 (16), H8A = 0.621 (16), H8D = 0.379 (16), H8B = 0.621 (16), H8E = 0.379 (16), H8C = 0.621 (16) and H8F = 0.379 (16). The remaining site occupancy factors were also refined as C8 = 0.621 (16) and C8' = 0.379 (16) during anisotropic refinement. H atoms were positioned geometrically with C—H = 0.97 and 0.96 Å for 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 methylene H atoms.

Figures

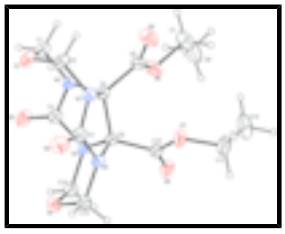

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

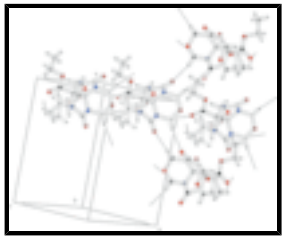

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

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

Crystal data

$C_{14}H_{18}N_4O_8$	F_{000} = 388
M_r = 370.32	D_x = 1.410 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1973 reflections
a = 8.3182 (11) Å	θ = 2.4–24.9º
b = 12.4523 (17) Å	μ = 0.12 mm^{-1}
c = 9.0479 (12) Å	T = 292 (2) K
β = 111.429 (2)º	Block, colourless
V = 872.4 (2) Å^3	0.30 × 0.20 × 0.20 mm
Z = 2	

Data collection

Bruker SMART 4 K CCD area-detector diffractometer	1764 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.024
Monochromator: graphite	θ_{max} = 27.0º
T = 292(2) K	θ_{min} = 2.4º
φ and ω scans	h = −10→9
Absorption correction: none	k = −15→15
5178 measured reflections	l = −8→11
1983 independent reflections	

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.105$

$S = 1.11$

1983 reflections

242 parameters

2 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

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.0447P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.15$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ 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)
O1	1.0609 (3)	0.93348 (19)	0.0366 (3)	0.0581 (6)	
O2	0.7355 (3)	1.1226 (2)	−0.1321 (3)	0.0696 (7)	
O3	0.7473 (3)	0.72136 (17)	0.0037 (3)	0.0610 (6)	
O4	0.4210 (3)	0.9097 (2)	−0.1716 (3)	0.0572 (6)	
O5	1.0440 (4)	1.0452 (3)	0.4466 (3)	0.0810 (8)	
O6	0.8671 (3)	0.9150 (2)	0.4617 (3)	0.0637 (6)	
O7	0.6082 (3)	1.08850 (19)	0.3175 (3)	0.0602 (6)	
O8	0.4464 (3)	0.9393 (2)	0.2830 (3)	0.0627 (7)	
N1	0.8814 (3)	1.04901 (19)	0.1154 (3)	0.0444 (6)	
N2	0.8885 (3)	0.86029 (18)	0.1682 (3)	0.0422 (5)	
N3	0.5932 (3)	1.0376 (2)	0.0105 (3)	0.0430 (5)	
N4	0.6002 (3)	0.85184 (18)	0.0845 (3)	0.0417 (5)	
C1	1.0532 (4)	0.8542 (3)	0.1461 (4)	0.0549 (8)	
H1A	1.1469	0.8651	0.2471	0.066*	
H1B	1.0666	0.7836	0.1069	0.066*	
C2	1.0460 (4)	1.0368 (3)	0.0943 (4)	0.0561 (8)	

H2A	1.0551	1.0905	0.0201	0.067*	
H2B	1.1399	1.0483	0.1951	0.067*	
C3	0.7366 (4)	1.0736 (2)	−0.0172 (4)	0.0465 (7)	
C4	0.7467 (3)	0.8029 (2)	0.0757 (4)	0.0427 (6)	
C5	0.8418 (3)	0.9662 (2)	0.2081 (3)	0.0384 (6)	
C6	0.9305 (4)	0.9837 (3)	0.3875 (4)	0.0495 (7)	
C7	0.9423 (6)	0.9107 (4)	0.6352 (4)	0.0835 (12)	
H7A	1.0499	0.9505	0.6737	0.100*	0.379 (16)
H7B	0.8637	0.9424	0.6799	0.100*	0.379 (16)
H7C	0.9169	0.9763	0.6799	0.100*	0.621 (16)
H7D	1.0655	0.9008	0.6717	0.100*	0.621 (16)
C8'	0.973 (4)	0.8013 (13)	0.681 (2)	0.120 (5)	0.379 (16)
H8D	0.8643	0.7644	0.6539	0.179*	0.379 (16)
H8E	1.0342	0.7970	0.7939	0.179*	0.379 (16)
H8F	1.0400	0.7684	0.6272	0.179*	0.379 (16)
C8	0.864 (2)	0.8187 (11)	0.6818 (12)	0.120 (5)	0.621 (16)
H8B	0.7419	0.8306	0.6512	0.179*	0.621 (16)
H8A	0.9146	0.8095	0.7949	0.179*	0.621 (16)
H8C	0.8831	0.7553	0.6302	0.179*	0.621 (16)
C9	0.6399 (3)	0.9613 (2)	0.1413 (3)	0.0371 (6)	
C10	0.5515 (3)	0.9931 (2)	0.2571 (3)	0.0437 (6)	
C11	0.5389 (6)	1.1295 (4)	0.4340 (5)	0.0843 (13)	
H11A	0.4160	1.1428	0.3836	0.101*	
H11B	0.5569	1.0774	0.5184	0.101*	
C12	0.6285 (8)	1.2279 (4)	0.4977 (6)	0.1070 (18)	
H12A	0.7496	1.2135	0.5494	0.161*	
H12B	0.5841	1.2575	0.5730	0.161*	
H12C	0.6116	1.2783	0.4130	0.161*	
C13	0.4298 (4)	1.0173 (3)	−0.1184 (4)	0.0540 (8)	
H13A	0.3354	1.0311	−0.0823	0.065*	
H13B	0.4179	1.0657	−0.2058	0.065*	
C14	0.4365 (4)	0.8373 (3)	−0.0448 (4)	0.0513 (7)	
H14A	0.4276	0.7640	−0.0832	0.062*	
H14B	0.3430	0.8497	−0.0071	0.062*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0501 (11)	0.0574 (15)	0.0793 (15)	0.0006 (10)	0.0384 (11)	0.0035 (12)
O2	0.0736 (15)	0.0679 (17)	0.0735 (16)	0.0006 (12)	0.0340 (13)	0.0305 (14)
O3	0.0601 (13)	0.0372 (12)	0.0925 (16)	0.0007 (10)	0.0360 (12)	−0.0151 (12)
O4	0.0512 (11)	0.0601 (14)	0.0532 (12)	−0.0013 (11)	0.0106 (9)	−0.0125 (11)
O5	0.0752 (17)	0.089 (2)	0.0687 (16)	−0.0352 (16)	0.0146 (13)	−0.0135 (15)
O6	0.0672 (13)	0.0738 (16)	0.0426 (11)	−0.0190 (12)	0.0113 (10)	0.0056 (11)
O7	0.0700 (14)	0.0540 (13)	0.0698 (14)	−0.0068 (11)	0.0413 (11)	−0.0195 (12)
O8	0.0542 (12)	0.0740 (17)	0.0763 (15)	−0.0143 (11)	0.0431 (11)	−0.0126 (13)
N1	0.0419 (12)	0.0373 (13)	0.0579 (14)	−0.0052 (10)	0.0230 (11)	0.0045 (11)
N2	0.0368 (11)	0.0353 (12)	0.0565 (14)	0.0021 (9)	0.0195 (10)	0.0033 (11)

N3	0.0409 (12)	0.0391 (12)	0.0492 (13)	0.0033 (10)	0.0166 (10)	0.0024 (11)
N4	0.0365 (11)	0.0354 (12)	0.0566 (14)	−0.0031 (9)	0.0209 (10)	−0.0043 (11)
C1	0.0400 (15)	0.0481 (18)	0.080 (2)	0.0081 (13)	0.0256 (14)	0.0042 (17)
C2	0.0431 (16)	0.0531 (18)	0.081 (2)	−0.0059 (14)	0.0326 (15)	0.0050 (18)
C3	0.0519 (16)	0.0347 (14)	0.0573 (17)	0.0012 (12)	0.0251 (13)	0.0034 (13)
C4	0.0446 (15)	0.0310 (14)	0.0572 (17)	0.0017 (11)	0.0241 (13)	0.0035 (13)
C5	0.0369 (12)	0.0335 (13)	0.0476 (15)	−0.0021 (10)	0.0188 (11)	−0.0002 (11)
C6	0.0389 (14)	0.0524 (18)	0.0527 (17)	−0.0025 (13)	0.0113 (12)	−0.0023 (14)
C7	0.098 (3)	0.096 (3)	0.046 (2)	−0.007 (3)	0.0133 (19)	0.004 (2)
C8'	0.172 (13)	0.127 (7)	0.055 (3)	−0.058 (9)	0.037 (7)	0.011 (4)
C8	0.172 (13)	0.127 (7)	0.055 (3)	−0.058 (9)	0.037 (7)	0.011 (4)
C9	0.0358 (12)	0.0320 (13)	0.0447 (15)	−0.0019 (10)	0.0159 (11)	−0.0020 (11)
C10	0.0383 (13)	0.0459 (16)	0.0485 (15)	−0.0005 (11)	0.0177 (11)	−0.0038 (13)
C11	0.095 (3)	0.084 (3)	0.091 (3)	−0.003 (2)	0.055 (3)	−0.036 (2)
C12	0.172 (5)	0.074 (3)	0.084 (3)	0.002 (3)	0.057 (3)	−0.025 (3)
C13	0.0450 (16)	0.0562 (19)	0.0551 (17)	0.0096 (13)	0.0113 (13)	0.0024 (15)
C14	0.0438 (15)	0.0459 (17)	0.064 (2)	−0.0068 (13)	0.0195 (13)	−0.0103 (14)

Geometric parameters (Å, °)

C1—O1	1.416 (4)	C8'—H8D	0.9600
C1—N2	1.458 (4)	C8'—H8E	0.9600
C1—H1A	0.9700	C8'—H8F	0.9600
C1—H1B	0.9700	C8—H8B	0.9600
C2—O1	1.411 (4)	C8—H8A	0.9600
C2—N1	1.458 (4)	C8—H8C	0.9600
C2—H2A	0.9700	C9—N4	1.451 (4)
C2—H2B	0.9700	C9—N3	1.455 (4)
C3—O2	1.203 (4)	C9—C10	1.536 (4)
C3—N3	1.380 (4)	C10—O8	1.191 (3)
C3—N1	1.389 (4)	C10—O7	1.320 (4)
C4—O3	1.208 (4)	C11—C12	1.441 (7)
C4—N2	1.371 (4)	C11—O7	1.466 (4)
C4—N4	1.390 (3)	C11—H11A	0.9700
C5—N1	1.442 (4)	C11—H11B	0.9700
C5—N2	1.457 (3)	C12—H12A	0.9600
C5—C6	1.533 (4)	C12—H12B	0.9600
C5—C9	1.565 (3)	C12—H12C	0.9600
C6—O5	1.181 (4)	C13—O4	1.417 (4)
C6—O6	1.311 (4)	C13—N3	1.454 (4)
C7—C8'	1.420 (16)	C13—H13A	0.9700
C7—C8	1.455 (10)	C13—H13B	0.9700
C7—O6	1.463 (4)	C14—O4	1.427 (4)
C7—H7A	0.9700	C14—N4	1.447 (4)
C7—H7B	0.9700	C14—H14A	0.9700
C7—H7C	0.9692	C14—H14B	0.9700
C7—H7D	0.9628		
C2—O1—C1	110.1 (2)	C8—C7—H7C	110.7
C13—O4—C14	110.3 (2)	O6—C7—H7C	110.1

C6—O6—C7	118.6 (3)	H7A—C7—H7C	74.4
C10—O7—C11	115.8 (3)	C8'—C7—H7D	74.0
C3—N1—C5	110.5 (2)	C8—C7—H7D	109.5
C3—N1—C2	118.0 (3)	O6—C7—H7D	110.9
C5—N1—C2	115.3 (2)	H7B—C7—H7D	134.8
C4—N2—C5	112.0 (2)	H7C—C7—H7D	109.4
C4—N2—C1	122.1 (3)	C7—C8'—H8D	109.5
C5—N2—C1	115.5 (2)	H7D—C8'—H8D	147.9
C3—N3—C13	121.8 (3)	C7—C8'—H8E	109.5
C3—N3—C9	111.6 (2)	H7D—C8'—H8E	91.2
C13—N3—C9	115.6 (2)	C7—C8'—H8F	109.4
C4—N4—C14	119.0 (2)	H7D—C8'—H8F	84.9
C4—N4—C9	109.9 (2)	C7—C8—H8B	109.5
C14—N4—C9	115.4 (2)	C7—C8—H8A	109.5
O1—C1—N2	110.2 (2)	H8B—C8—H8A	109.5
O1—C1—H1A	109.6	C7—C8—H8C	109.4
N2—C1—H1A	109.6	H8B—C8—H8C	109.5
O1—C1—H1B	109.6	H8A—C8—H8C	109.5
N2—C1—H1B	109.6	N4—C9—N3	111.5 (2)
H1A—C1—H1B	108.1	N4—C9—C10	112.4 (2)
O1—C2—N1	110.9 (2)	N3—C9—C10	109.5 (2)
O1—C2—H2A	109.5	N4—C9—C5	104.3 (2)
N1—C2—H2A	109.5	N3—C9—C5	102.6 (2)
O1—C2—H2B	109.5	C10—C9—C5	116.1 (2)
N1—C2—H2B	109.5	O8—C10—O7	127.0 (3)
H2A—C2—H2B	108.0	O8—C10—C9	123.9 (3)
O2—C3—N3	126.0 (3)	O7—C10—C9	109.1 (2)
O2—C3—N1	126.1 (3)	C12—C11—O7	107.5 (4)
N3—C3—N1	107.8 (3)	C12—C11—H11A	110.2
O3—C4—N2	126.5 (3)	O7—C11—H11A	110.2
O3—C4—N4	125.2 (3)	C12—C11—H11B	110.2
N2—C4—N4	108.3 (2)	O7—C11—H11B	110.2
N1—C5—N2	111.4 (2)	H11A—C11—H11B	108.5
N1—C5—C6	113.0 (2)	C11—C12—H12A	109.5
N2—C5—C6	108.8 (2)	C11—C12—H12B	109.5
N1—C5—C9	104.1 (2)	H12A—C12—H12B	109.5
N2—C5—C9	102.4 (2)	C11—C12—H12C	109.5
C6—C5—C9	116.6 (2)	H12A—C12—H12C	109.5
O5—C6—O6	126.5 (3)	H12B—C12—H12C	109.5
O5—C6—C5	124.6 (3)	O4—C13—N3	110.7 (2)
O6—C6—C5	108.8 (2)	O4—C13—H13A	109.5
C8'—C7—O6	108.1 (9)	N3—C13—H13A	109.5
C8—C7—O6	106.2 (5)	O4—C13—H13B	109.5
C8'—C7—H7A	110.1	N3—C13—H13B	109.5
C8—C7—H7A	138.7	H13A—C13—H13B	108.1
O6—C7—H7A	110.1	O4—C14—N4	110.7 (2)
C8'—C7—H7B	110.0	O4—C14—H14A	109.5
C8—C7—H7B	75.9	N4—C14—H14A	109.5
O6—C7—H7B	110.1	O4—C14—H14B	109.5

H7A—C7—H7B	108.4	N4—C14—H14B	109.5
C8'—C7—H7C	136.8	H14A—C14—H14B	108.1
N2—C1—O1—C2	60.7 (3)	N1—C5—C9—N4	120.5 (2)
O1—C1—N2—C4	90.1 (3)	N2—C5—C9—N4	4.3 (3)
O1—C1—N2—C5	−52.1 (4)	C6—C5—C9—N4	−114.3 (3)
N1—C2—O1—C1	−60.9 (3)	N1—C5—C9—N3	4.1 (3)
O1—C2—N1—C3	−81.6 (3)	N2—C5—C9—N3	−112.1 (2)
O1—C2—N1—C5	52.0 (4)	C6—C5—C9—N3	129.3 (2)
O2—C3—N1—C5	−164.3 (3)	N1—C5—C9—C10	−115.4 (3)
N3—C3—N1—C5	19.3 (3)	N2—C5—C9—C10	128.5 (2)
O2—C3—N1—C2	−28.6 (5)	C6—C5—C9—C10	9.9 (3)
N3—C3—N1—C2	155.0 (3)	O5—C6—O6—C7	−0.7 (6)
O2—C3—N3—C13	24.5 (5)	C5—C6—O6—C7	175.2 (3)
N1—C3—N3—C13	−159.0 (3)	C8'—C7—O6—C6	−132.7 (13)
O2—C3—N3—C9	167.1 (3)	C8—C7—O6—C6	−172.3 (9)
N1—C3—N3—C9	−16.5 (3)	N4—C9—N3—C3	−103.8 (3)
O3—C4—N2—C5	167.0 (3)	C10—C9—N3—C3	131.2 (2)
N4—C4—N2—C5	−15.9 (3)	C5—C9—N3—C3	7.3 (3)
O3—C4—N2—C1	23.7 (5)	N4—C9—N3—C13	41.3 (3)
N4—C4—N2—C1	−159.2 (3)	C10—C9—N3—C13	−83.8 (3)
O3—C4—N4—C14	−27.8 (4)	C5—C9—N3—C13	152.4 (2)
N2—C4—N4—C14	155.0 (2)	N3—C9—N4—C4	96.1 (2)
O3—C4—N4—C9	−164.1 (3)	C10—C9—N4—C4	−140.5 (2)
N2—C4—N4—C9	18.7 (3)	C5—C9—N4—C4	−13.9 (3)
N2—C5—N1—C3	95.5 (3)	N3—C9—N4—C14	−41.8 (3)
C6—C5—N1—C3	−141.7 (2)	C10—C9—N4—C14	81.6 (3)
C9—C5—N1—C3	−14.2 (3)	C5—C9—N4—C14	−151.9 (2)
N2—C5—N1—C2	−41.5 (3)	N4—C9—C10—O8	−8.5 (4)
C6—C5—N1—C2	81.3 (3)	N3—C9—C10—O8	116.1 (3)
C9—C5—N1—C2	−151.1 (2)	C5—C9—C10—O8	−128.3 (3)
N1—C5—N2—C4	−104.0 (3)	N4—C9—C10—O7	173.0 (2)
C6—C5—N2—C4	130.8 (2)	N3—C9—C10—O7	−62.5 (3)
C9—C5—N2—C4	6.7 (3)	C5—C9—C10—O7	53.1 (3)
N1—C5—N2—C1	41.9 (3)	O8—C10—O7—C11	2.9 (5)
C6—C5—N2—C1	−83.3 (3)	C9—C10—O7—C11	−178.6 (3)
C9—C5—N2—C1	152.6 (2)	C12—C11—O7—C10	174.1 (4)
N1—C5—C6—O5	−13.6 (4)	N3—C13—O4—C14	59.8 (3)
N2—C5—C6—O5	110.6 (4)	O4—C13—N3—C3	90.0 (4)
C9—C5—C6—O5	−134.3 (4)	O4—C13—N3—C9	−51.1 (3)
N1—C5—C6—O6	170.4 (2)	O4—C14—N4—C4	−81.8 (3)
N2—C5—C6—O6	−65.4 (3)	O4—C14—N4—C9	52.2 (3)
C9—C5—C6—O6	49.7 (3)	N4—C14—O4—C13	−60.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O8^{i}	0.97	2.56	3.223 (4)	125
C1—H1B···O2^{ii}	0.97	2.55	3.404 (4)	147

C2—H2A···O3^{iii}	0.97	2.38	3.183 (4)	140
C8—H8A···O1^{iv}	0.96	2.59	3.345 (12)	136

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

Fig. 1

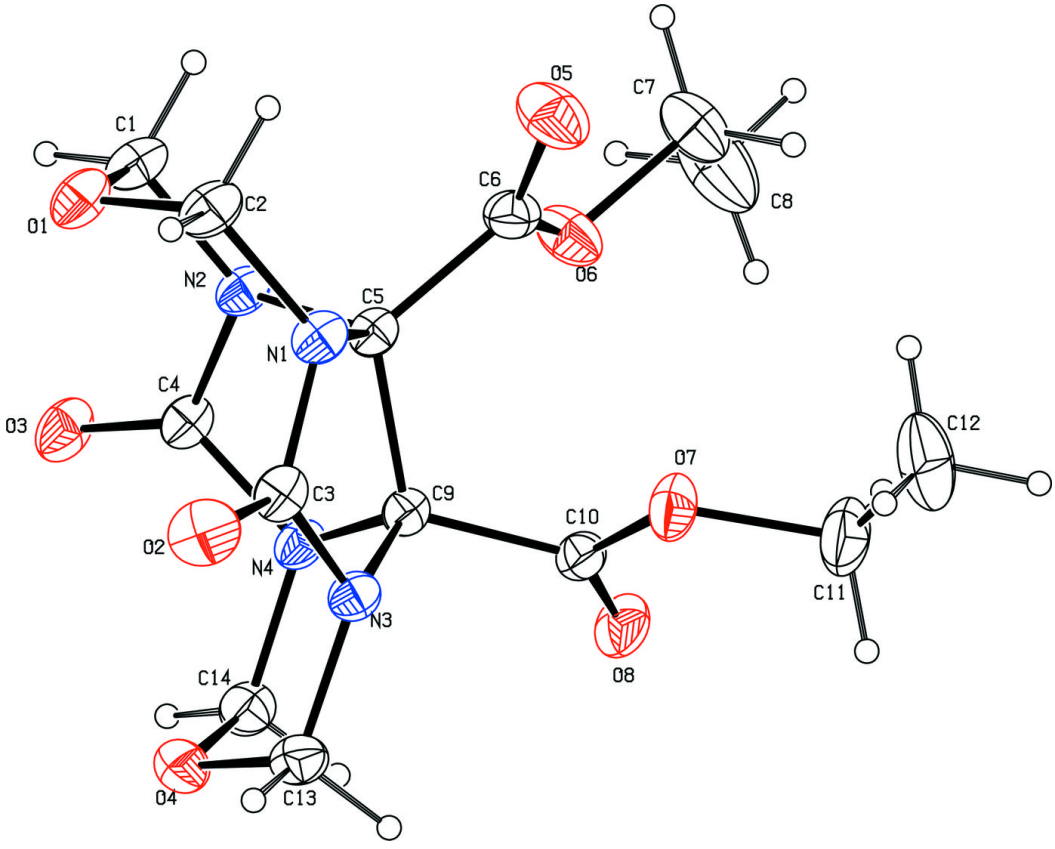
O5
C7
C1
C6
C8
O1
C2
O6
N2
C5
N1
C4
O3
C12
O7
C3
C9
C10
O2
N4
C11
N3
O8
C14
O4
C13

Fig. 2