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Diethyl 6-(2-hydroxyethyl)-1,4-dioxo-2,2a,3,4,6,7-hexahydro-1*H*,5*H*-2,3,4a,6,7a-pentaazacyclopenta-[*cd*]indene-2a,7b-dicarboxylate

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Key indicators

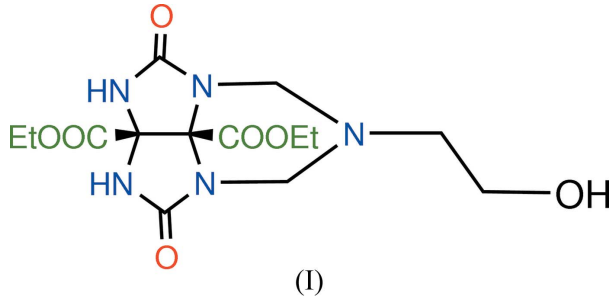
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.152
Data-to-parameter ratio = 6.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{14}H_{21}N_5O_7$, is a glycoluril derivative. The molecule is built up from three fused rings. The triazine six-membered ring displays a normal chair conformation.

Comment

Glycoluril derivatives have shown applications in many fields such as explosives, slow-release fertilizers, cross-linkers, iodogen stabilisers of organic compounds against photodegradation and as reagents in combinatorial chemistry (Wu *et al.*, 2002). As a part of our ongoing investigation of glycoluril derivatives (Li *et al.*, 2005), we present here the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The triazine six-membered ring displays a normal chair conformation. Within the imidazole rings, the N—$C_{carbonyl}$ bond distances are much shorter than the other N—C bond distances (Table 1). The crystal packing is stabilized by intermolecular O—H···O, N—H···O and C—H···O hydrogen bonding (Table 2).

Experimental

2-Aminoethanol (0.61 g, 10 mmol) and formaldehyde (1.2 g, 40 mmol) were added to a stirred solution of diethyl 2,5-dioxotetrahydroimidazo[4,5-*d*]imidazole-3a,6a-dicarboxylate (1.43 g, 5 mmol) in acetonitrile (50 ml) under a nitrogen atmosphere. The mixture was stirred overnight at room temperature. The solvent was evaporated to dryness and the compound was purified by column chromatography to give (I) (yield 0.31 g, 20%). Single crystals of (I) were obtained by slow evaporation of an ethyl acetate solution at 283 K.

Crystal data

$C_{14}H_{21}N_5O_7$
M_r = 371.36
Monoclinic, *Cc*
a = 17.867 (4) Å
b = 11.871 (2) Å
c = 8.8912 (17) Å
β = 115.793 (3)°
V = 1698.0 (6) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
Mo *Kα* radiation
μ = 0.12 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
5110 measured reflections
1659 independent reflections
1354 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 26.0^{\circ}$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.152$
$S = 1.06$
1659 reflections
261 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C2—N1	1.473 (7)	C6—N5	1.361 (6)
C3—N1	1.448 (7)	C6—N3	1.361 (6)
C3—N2	1.468 (6)	C7—N3	1.452 (6)
C4—N1	1.439 (7)	C7—N2	1.454 (6)
C4—N3	1.471 (6)	C11—N5	1.437 (6)
C5—N4	1.344 (6)	C11—N4	1.439 (6)
C5—N2	1.392 (6)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82 (2)	2.16 (4)	2.911 (6)	152 (8)
N4—H4···O1^{ii}	0.86	2.03	2.860 (5)	161
N5—H5···O3^{iii}	0.86	2.12	2.877 (5)	147
C3—H3A···O2^{i}	0.97	2.59	3.260 (6)	126
C13—H13A···O2^{iv}	0.97	2.54	3.511 (11)	176

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

The hydroxy H atom was located in a difference Fourier map and refined with a restraint of O—H = 0.82 (2) Å, and with U_{iso}(H) = 1.5U_{eq}(O). Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions, with N—H = 0.86 Å and C—H = 0.97 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(N,C). One of the ethyl groups (C13 and C14 and attached H atoms) is disordered over two sites; the site-occupancy factors refined to 0.77 (3) and 0.23 (3). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

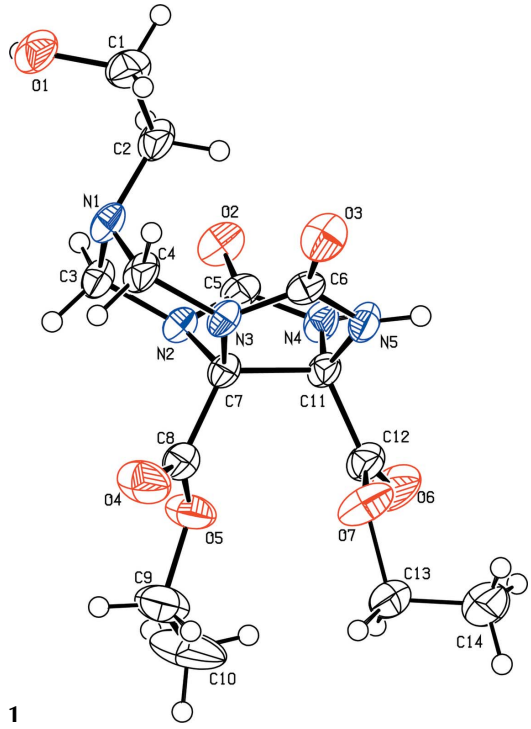


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Only one disorder component is shown.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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