Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-Bis(hydroxymethyl)-6-isopropylperhydro-2,3,4a,6,7a-pentaazacyclopenta[*cd*]indene-1,4-dione

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Received 13 December 2006
Accepted 18 December 2006

The molecule of the title compound, $C_{11}H_{19}N_5O_4$, contains three fused rings, namely two nearly planar imidazole rings and one non-planar triazine ring. The latter ring displays a chair conformation. Two hydroxylmethylene groups are linked to two N atoms from separate rings of the glycoluril system.

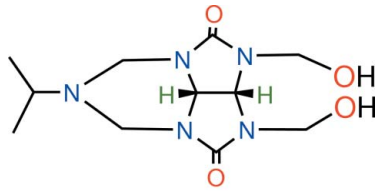
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.127
Data-to-parameter ratio = 17.0

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

Comment

Glycoluril derivatives have applications in many fields such as explosives, slow-release fertilizers, cross-linkers, iodogen stabilizers of organic compounds against photodegradation and 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. Within the nearly planar five-membered ring, the N—C(carbonyl) bond

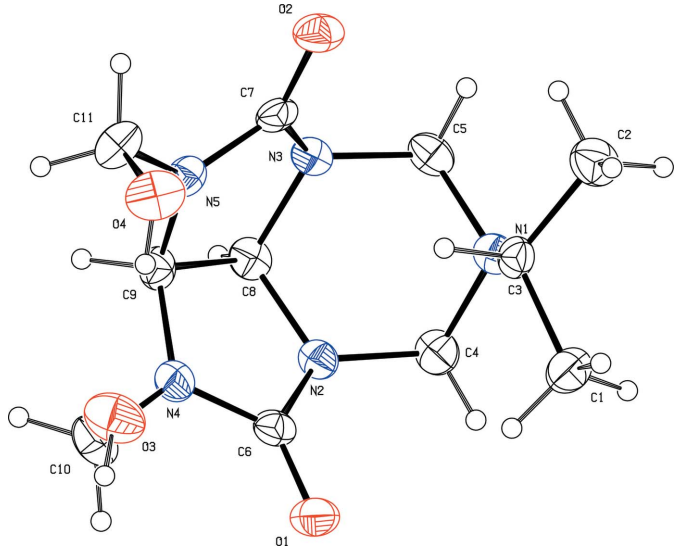


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary radius.

distances are much shorter than the other N—C distances (Table 1), indicating electron delocalization. The six-membered ring displays a chair conformation.

The crystal structure is stabilized by inter- and intramolecular O—H···O and C—H···O hydrogen-bonding interactions (Table 2).

Experimental

Isopropylamine (0.58 g, 10 mmol) and formaldehyde (2.4 g, 40 mmol) were added to a stirred solution of tetrahydroimidazo[4,5-*d*]imidazole-2,5-dione (0.71 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 obtain the title compound (yield 0.28 g, 20%) as a colorless soid. Crystals of (I) suitable for data collection were obtained by slow evaporation of a methanol solution at 283 K.

Crystal data

$C_{11}H_{19}N_5O_4$
M_r = 285.31
Orthorhombic, *Pbca*
a = 12.778 (3) Å
b = 12.655 (3) Å
c = 15.926 (4) Å
V = 2575.2 (11) Å^3
Z = 8
D_x = 1.472 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 292 (2) K
Plate, colorless
0.30 × 0.20 × 0.04 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
28542 measured reflections
3154 independent reflections
1428 reflections with $I > 2\sigma(I)$
R_{int} = 0.143
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.127
S = 0.83
3154 reflections
185 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—N1	1.486 (3)	C7—N5	1.367 (3)
C4—N1	1.454 (3)	C7—N3	1.370 (3)
C4—N2	1.461 (3)	C8—N2	1.451 (3)
C6—N2	1.365 (3)	C8—N3	1.452 (3)
C6—N4	1.373 (3)		
N2—C6—N4	107.7 (2)	N5—C7—N3	109.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5*B*···O2	0.97	2.59	2.925 (3)	101
O4—H4···O3	0.82	1.92	2.728 (3)	168
C10—H10*B*···O4^{i}	0.97	2.58	3.494 (3)	157
O3—H3*A*···O2^{i}	0.82	1.90	2.713 (2)	173

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z$.

All H atoms were positioned geometrically and treated as riding, with C—H = 0.96 (CH_3), 0.97 (CH_2) or 0.98 Å (CH) and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C,O).

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 (Central China Normal University, Wuhan, China) for the X-ray data collection.

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