

Yi-Tao Li, Yun-Feng Chen,
Neng-Fang She and An-Xin Wu*Key Laboratory of Pesticides and Chemical
Biology, Ministry of Education, College of
Chemistry, Central China Normal University,
Wuhan 430079, People's Republic of ChinaCorrespondence e-mail:
chwuax@mail.ccnu.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 292\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.054
 wR factor = 0.155
Data-to-parameter ratio = 12.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Diethyl 2,6-bis(2-hydroxyethyl)-4,8-dioxo-
1,2,5,6-tetrahydrohydro-2,3a,4a,6,7a,8a-
hexaazacyclopenta[def]fluorene-8b,8c-
dicarboxylateIn the title glycoluril derivative, $\text{C}_{18}\text{H}_{28}\text{N}_6\text{O}_8$, the $\text{N}-\text{C}_{\text{carbonyl}}$ bond distances are much shorter than the other $\text{N}-\text{C}$ bond distances in the same five-membered ring, indicating electron delocalization within the nearly planar ring. Both six-membered rings display chair conformations.Received 24 August 2005
Accepted 5 September 2005
Online 21 September 2005

Comment

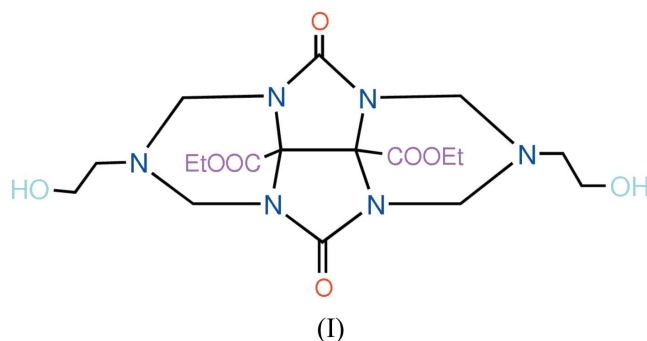
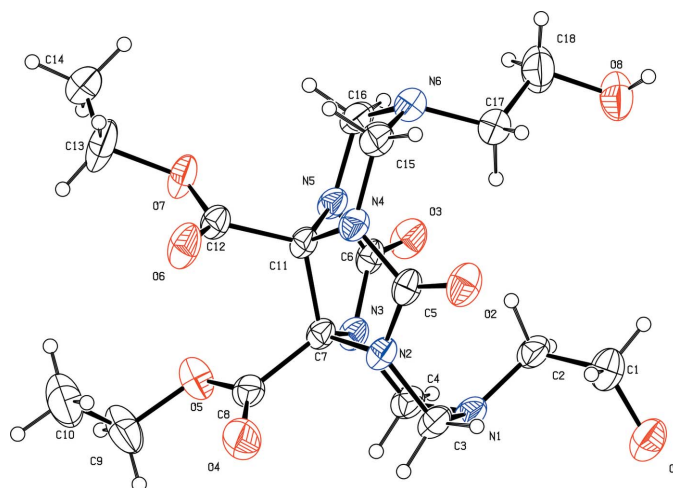
Glycoluril derivatives have shown applications in many fields, such as explosives, slow-release fertilizers, crosslinkers, iodogens, stabilisers of organic compounds against photo-degradation and reagents in combinatorial chemistry (Wu *et al.*, 2002). As a part of our ongoing investigation of glycoluril derivatives (Li & Wu, 2005), we present here the structure of the title compound, (I).The molecular structure of (I) is shown in Fig. 1. Within the nearly planar five-membered ring, the $\text{N}-\text{C}_{\text{carbonyl}}$ bond

Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

distances are much shorter than the other N—C distances (Table 1), indicating electron delocalization. Both six-membered rings display chair conformations.

Classical O—H···O and weaker C—H···O intermolecular hydrogen bonds occur in the crystal structure of (I) (Table 2).

Experimental

2-Aminoethanol (0.61 g, 10 mmol) and formaldehyde (1.2 g, 40 mmol) were added to a stirred solution of 2,5-dioxotetrahydroimidazo[4,5-*d*]imidazole-3a,6a-dicarboxylic acid diethyl ester (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 yield solid (I) (yield 2.05 g, 90%). Single crystals of (I) were obtained by recrystallization from an ethyl acetate solution at 283 K.

Crystal data

$C_{18}H_{28}N_6O_8$	$D_x = 1.408 \text{ Mg m}^{-3}$
$M_r = 456.46$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3098 reflections
$a = 10.116 (2) \text{ \AA}$	$\theta = 2.5\text{--}24.7^\circ$
$b = 8.7131 (18) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 24.608 (5) \text{ \AA}$	$T = 292 (2) \text{ K}$
$\beta = 96.763 (4)^\circ$	Block, colourless
$V = 2153.9 (8) \text{ \AA}^3$	$0.30 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2819 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.029$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
10431 measured reflections	$h = -12 \rightarrow 10$
3758 independent reflections	$k = -10 \rightarrow 9$
	$l = -28 \rightarrow 29$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.5776P]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.155$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$
3758 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
293 parameters	
H-atom parameters constrained	

Table 1

Selected bond lengths (\AA).

C1—O1	1.404 (3)	C8—O4	1.185 (3)
C5—O2	1.215 (3)	C8—O5	1.321 (3)
C5—N2	1.362 (3)	C9—O5	1.474 (3)
C5—N4	1.382 (3)	C11—N4	1.448 (3)
C6—O3	1.214 (3)	C11—N5	1.438 (3)
C6—N5	1.373 (3)	C12—O6	1.188 (3)
C6—N3	1.377 (3)	C12—O7	1.312 (3)
C7—N2	1.444 (3)	C13—O7	1.464 (3)
C7—N3	1.455 (3)	C18—O8	1.416 (3)
C7—C11	1.565 (3)		

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H1···O3 ⁱ	0.82	2.13	2.832 (3)	144
O8—H8···O2 ⁱⁱ	0.82	1.96	2.760 (3)	164
C16—H16A···O4 ⁱⁱⁱ	0.97	2.43	3.302 (3)	149

Symmetry codes: (i) $-x, -y + 2, -z$; (ii) $-x + 1, -y + 2, -z$; (iii) $x, y + 1, z$.

Hydroxyl H and methyl H atoms were placed in calculated positions, with C—H = 0.96 \AA and O—H = 0.82 \AA , and torsion angles were refined to fit the electron density, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{carrier})$. Methylene H atoms were placed in calculated positions, with C—H = 0.97 \AA , and refined in riding mode, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

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.

The authors are grateful to the Central China Normal University, the National Natural Science Foundation of China (grant No.20472022) and the Hubei Province Natural Science Fund (grant Nos. 2004ABA085 and 2004ABC002) for financial support.

References

- Bruker (2000). SMART (Version 5.618), SAINT (Version 6.02) and SHELXTL (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Li, Y. & Wu, A. (2005). *Acta Cryst.* **E61**, o2374–o2375.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Wu, A., Fetting, J. C. & Isaacs, L. (2002). *Tetrahedron*, **58**, 9769–9777.