

2,6-Diisopropyl-2,3,6,7-tetrahydro-2,3a,4a,6,7a,8a-hexaaza-1*H*,5*H*-cyclopenta-[*def*]fluorene-4,8-dioneShu-Qi Qin^a and Yi-Tao Li^{b*}^aCollege of Chemistry and Engineering, Northwest Normal University, Lanzhou 730070, People's Republic of China, and ^bKey Laboratory of Pesticides & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of ChinaCorrespondence e-mail:
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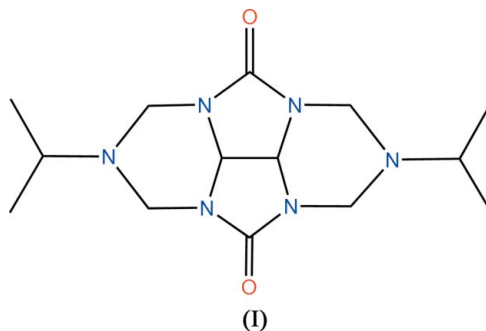
Key indicators

Single-crystal X-ray study
T = 292 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.052
wR factor = 0.136
Data-to-parameter ratio = 17.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{14}\text{H}_{24}\text{N}_6\text{O}_2$, is a glycoluril derivative. The molecule is built up from four fused rings, two nearly planar imidazole five-membered rings and two non-planar triazine six-membered rings. Both six-membered rings display chair conformations.

Comment

Glycoluril derivatives have shown applications in many fields such as explosives, slow-release fertilizers, cross-linkers, iodogen stabilizers of organic compounds against photo-degradation and reagents in combinatorial chemistry (Wu *et al.*, 2002). As part of our ongoing investigations 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 rings, the N—C(carbonyl) bond distances are much shorter than the other N—C distances (Table 1) due to conjugation.

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, giving the title compound (yield 1.38 g, 90%) as a colorless solid. Crystals suitable for data collection were obtained by slow evaporation of a methanol solution at 283 K.

Crystal data

 $\text{C}_{14}\text{H}_{24}\text{N}_6\text{O}_2$
 $M_r = 308.39$
Monoclinic, $P2_1/n$
 $a = 13.1492 (17) \text{ \AA}$
 $b = 7.7672 (10) \text{ \AA}$
 $c = 14.9337 (19) \text{ \AA}$
 $\beta = 94.934 (2)^\circ$
 $V = 1519.6 (3) \text{ \AA}^3$ $Z = 4$
 $D_x = 1.348 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 292 (2) \text{ K}$
Block, colorless
 $0.30 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer
 φ and ω scans
Absorption correction: none
17239 measured reflections

3627 independent reflections
2602 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.068$
 $\theta_{\text{max}} = 28.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.136$
 $S = 0.99$
3627 reflections
203 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0744P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$

Table 1Selected geometric parameters (\AA , $^\circ$).

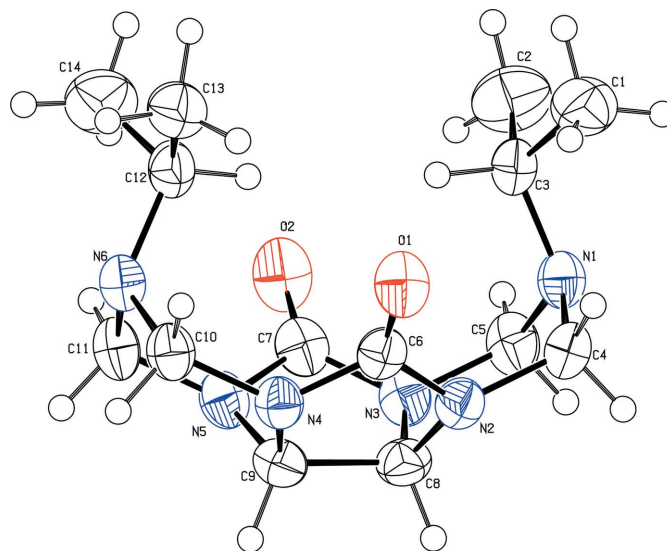
C3–N1	1.4787 (19)	C7–N3	1.383 (2)
C5–N1	1.4535 (19)	C7–N5	1.3851 (19)
C5–N3	1.4688 (19)	C8–N2	1.4482 (18)
C6–N4	1.3796 (18)	C8–N3	1.451 (2)
C6–N2	1.3835 (18)		
N4–C6–N2	107.58 (12)	N3–C7–N5	107.45 (13)

Table 2Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
C13–H13C \cdots O1	0.96	2.60	3.442 (2)	147

The methyl H atoms were constrained to an ideal geometry, with C–H distances of 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Methylene H atoms were placed in calculated positions, with C–H = 0.97 \AA , and refined in riding mode, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Methine H atoms were placed in calculated positions, with C–H = 0.98 \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: *SMART*; data reduction: *SAINTE* (Bruker, 2000); program(s) used to solve

**Figure 1**

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

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 Northwest Normal University and Gansu Province Natural Science Fund (No. 3ZS051-A25-002) for financial support.

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