

Diethyl 2,6-diisobutyl-4,8-dioxo-2,3,6,7-tetrahydro-1*H*,5*H*-2,3*a*,4*a*,6,7*a*,8*a*-hexaazacyclopenta[*def*]-fluorene-8*b*,8*c*-dicarboxylate**Yi-Tao Li* and Qian-Ni Guo**

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Correspondence e-mail:
chliyt@mail.ccnu.edu.cn**Key indicators**Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.057
 wR factor = 0.176
Data-to-parameter ratio = 13.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

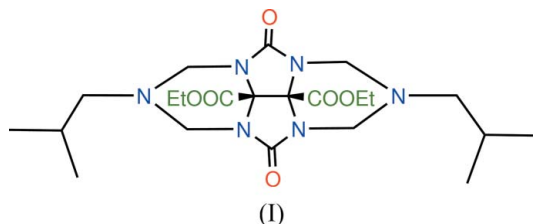
The molecule of the title compound, $\text{C}_{22}\text{H}_{36}\text{N}_6\text{O}_6$, exhibits normal geometric parameters. Both imidazole rings adopt envelope conformations with the $\text{C}=\text{O}$ group at the flap position. The triazinane rings adopt chair conformations.

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Comment

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



The molecular structure of (I) is shown in Fig. 1. It has four fused rings, two imidazole and two triazinane rings. Both imidazole rings adopt envelope conformations with the $\text{C}=\text{O}$ group at the flap position. The six-membered triazinane rings adopt chair conformations, and the isobutyl and ethyl-carboxylate groups are axially attached to them. The Csp^2-N and Csp^3-N distances lie in the range 1.373 (3)–1.380 (3) Å and 1.443 (3)–1.479 (3) Å, respectively. The crystal packing is stabilized by $\text{C}-\text{H}\cdots\text{O}$ interactions (Table 1).

Experimental

Isobutylamine (0.73 g, 10 mmol) and formaldehyde (2.4 g, 40 mmol) were added to a stirred solution of 2,5-dioxo-tetrahydro-imidazo-[4,5-*d*]imidazole-3*a*,6*a*-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 (I) (2.16 g, 90%) as a colourless solid. Colourless, block-like crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of an acetic acid–ethyl ester solution (0.004 mol l^{-1}) at 283 K.

Crystal data

C₂₂H₃₆N₆O₆
M_r = 480.57
 Monoclinic, *P*2₁/*c*
a = 13.7596 (9) Å
b = 7.8469 (5) Å
c = 24.4168 (15) Å
 β = 103.476 (1)°
V = 2563.7 (3) Å³

Z = 4
D_x = 1.245 Mg m⁻³
 Mo *K*α radiation
 μ = 0.09 mm⁻¹
T = 292 (2) K
 Block, colourless
 0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.973, *T_{max}* = 0.982

19253 measured reflections
 5044 independent reflections
 3644 reflections with *I* > 2σ(*I*)
R_{int} = 0.030
 θ_{max} = 26.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.057
wR(*F*²) = 0.176
S = 1.09
 5044 reflections
 380 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0971P)^2 + 0.2921P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.30 e Å⁻³
 Δρ_{min} = -0.33 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6—H6 <i>B</i> ...O3	0.97	2.57	3.109 (3)	115
C20—H20 <i>A</i> ...O1 ⁱ	0.98	2.40	3.340 (8)	160

Symmetry code: (i) *x*, *y* + 1, *z*.

Atoms C1–C3 and C20–C22 of the two isopropyl groups are disordered over two positions with site occupation factors of 0.605 (16) and 0.395 (16), respectively, for the major and minor components of atoms C1–C3, and 0.541 (11) and 0.458 (11), respectively, for the major and minor components of atoms C20–C22. Atom C12 is also disordered over two positions with site occupation factors of 0.687 (10) and 0.313 (10). The C–C bond distances involving the disordered atoms were restrained to 1.51 (1) Å and the *U*^{*ij*} components of atoms C1', C2', C22, C2 and C3 were approximated to isotropic behaviour. Methyl H atoms were constrained to an ideal geometry, with C–H distances of 0.96 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C), but each group was allowed to rotate freely about its C–C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.95–0.98 Å and with *U*_{iso}(H) = 1.2*U*_{eq}(C).

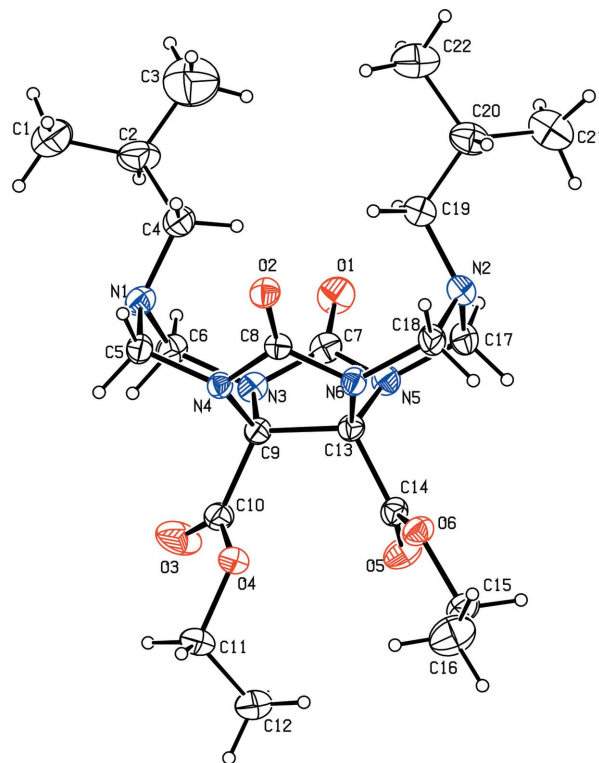


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Only the major components of the disordered atoms are shown.

Data collection: *SMART* (Bruker, 2000); cell refinement: *S SAINT* (Bruker, 2000); data reduction: *S 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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