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Diethyl 2,6-dibenzyl-4,8-dioxotetrahydro-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]-fluorene-8b,8c-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.072
wR factor = 0.204
Data-to-parameter ratio = 13.1

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

The molecule of the title compound, $C_{28}H_{32}N_6O_6$, shows normal geometrical parameters. The crystal packing is stabilized by a C—H···O interaction.

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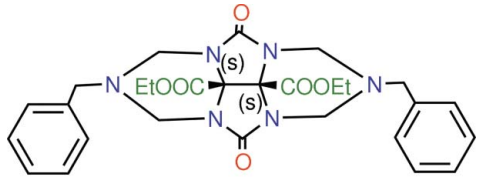
Comment

Glycoluril derivatives have many areas of applications, such as explosives, slow-release fertilizers, cross-linkers, iodogens, stabilizers of organic compounds against photodegradation, and reagents in combinatorial chemistry (Wu *et al.*, 2002). In this paper, we present the structure of the title compound, (I) (Fig. 1), as a continuation of our previous studies in this area (Wei & Wu, 2005).



(I)

All the geometrical parameters for (I) are normal. The crystal packing is stabilized by a C—H···O interaction (Table 1 and Fig. 2), leading to chains of (I) along [100]. The shortest distance between ring centroids of 4.38 Å indicates that any π–π effects are very weak.

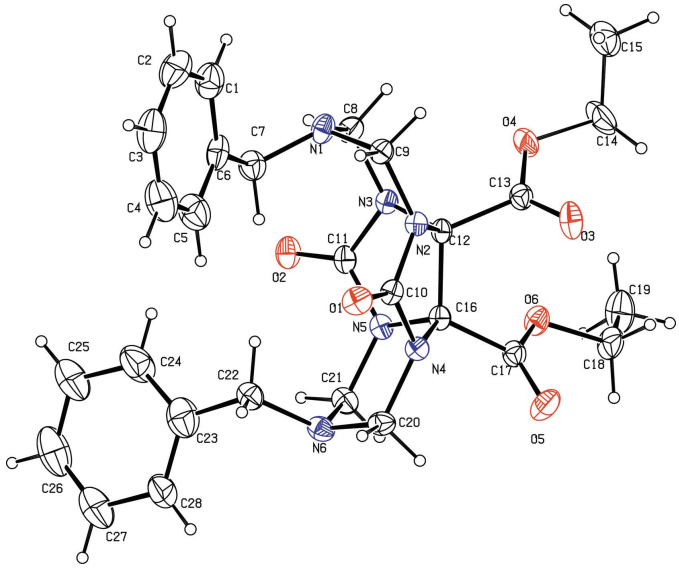


Figure 1
View of (I) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Only the major component of the disordered C14/C15 ethyl group is shown.

Experimental

Benzylamine (1.07 g, 10 mmol) and formaldehyde (2.4 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 yield (I) (2.46 g, 90%) as a colorless solid. Colorless block-like crystals of (I) suitable for data collection were obtained by slow evaporation of a solution in ethyl acetate at 283 K.

Crystal data

$C_{28}H_{32}N_6O_6$
M_r = 548.60
Triclinic, $P\overline{1}$
a = 7.6177 (6) Å
b = 12.2882 (9) Å
c = 14.9197 (11) Å
α = 95.008 (1)°
β = 101.415 (1)°
γ = 92.755 (1)°
V = 1360.69 (18) Å^3
Z = 2
D_x = 1.339 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4320 reflections
θ = 2.7–26.4°
μ = 0.10 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.981
9666 measured reflections
4689 independent reflections
3734 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°
$h = -8 \rightarrow 9$
$k = -14 \rightarrow 13$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.204
S = 1.04
4689 reflections
359 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1005P)^2 + 1.0611P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1^{i}	0.97	2.44	3.261 (3)	142

Symmetry codes: (i) $x + 1, y, z$.

The C14/C15 ethyl group is disordered over two positions in a 0.657 (6):0.343 (6) ratio. All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, allowing for free rotation of methyl groups. The constraint U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C) was applied.

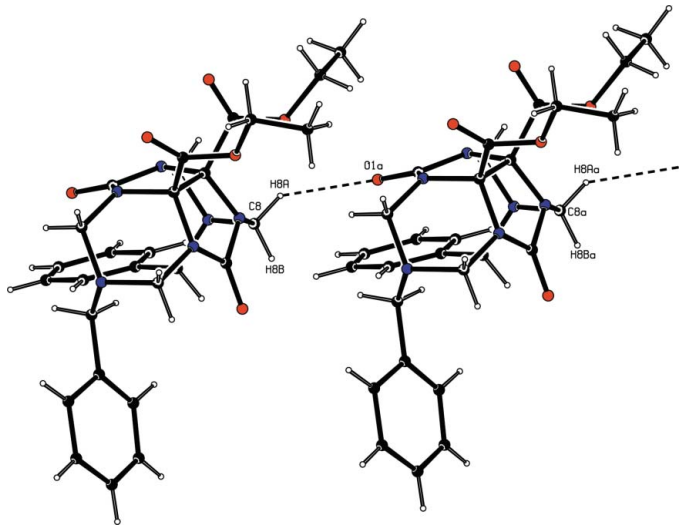


Figure 2
The C—H···O interaction (dashed lines) in (I). [Symmetry code: (*a*) 1 + *x*, *y*, *z*.]

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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