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Diethyl 2,6-bis(4-methylbenzyl)-4,8-dioxo-2,3,6,7-tetrahydro-1*H*,5*H*-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]fluorene-8b,8c-dicarboxylate

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The title compound, $C_{30}H_{36}N_6O_6$, is a glycoluril derivative with two ethyl acetate substituents on the convex face of the glycouril system. Two equivalent six-membered triazine rings bind the N atoms from separate rings of the glycouril unit to form the flexible sidewalls of a molecular clip. One N atom from each ring caries a 4-methylbenzyl substituent. The crystal structure is stabilized by intermolecular C—H···O interactions.

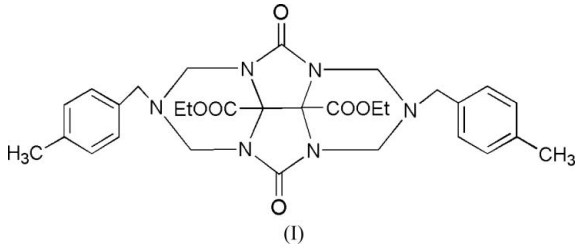
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.064
wR factor = 0.197
Data-to-parameter ratio = 14.3

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

Comment

Derivatives of glycoluril have been employed in many applications, including polymer cross-linking, explosives, stabilization of organic compounds against photo-degradation, textile waste, stream purification, and combinational chemistry. (Witt *et al.*, 2000). They are also used as building blocks for self-assembly, molecular recognition, and catalysis (Rebek, 1999; Rowan *et al.*, 1999). We report here the structure of the title glycoluril derivative, (I) (Fig. 1), in which the bond lengths and angles present no unusual features and are similar to those found in other comparable compounds (Li *et al.*, 2006). The dihedral angle between the imidazolone rings of the glycouril unit is 71.26 (3)° and the six-membered triazine rings each adopt a chair conformation.



(I)

In the crystal structure, C—H···O hydrogen bonds (Table 1) link the molecules into rows along the *c* axis (Fig. 2).

Experimental

The title compound was synthesized according to the procedure of Liu *et al.* (2004). Crystals appropriate for data collection were obtained by slow evaporation of a CH_2Cl_2/CH_3OH (1:1 *v*/*v*) solution at 283 K.

Crystal data

$C_{30}H_{36}N_6O_6$
M_r = 576.65
Monoclinic, $P2_1/c$
a = 16.9160 (8) Å
b = 13.6411 (6) Å
c = 12.9597 (2) Å
β = 94.160 (1)°
V = 2982.6 (2) Å^3
Z = 4
D_x = 1.284 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
33094 measured reflections
6504 independent reflections
3764 reflections with $I > 2\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 27.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.197$
$S = 1.01$
6504 reflections
455 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1054P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22B···O1^{i}	0.97	2.40	3.356 (3)	169
C19—H19B···O3^{i}	0.97	2.47	3.179 (5)	129

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

One ethyl group and one 4-methylbenzyl group were found to be disordered over two orientations. The occupancies of the disordered positions C19/C19′ and C20/C20′ refined to 0.68 (1)/0.32 (1), and C23/C23′, C24/C24′, C25/C25′, C26/C26′, C27/C27′, C28/C28′, C29/C29′, and C30/C30′ refined to 0.60 (1)/0.40 (1). All H atoms bound to carbon were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2, and C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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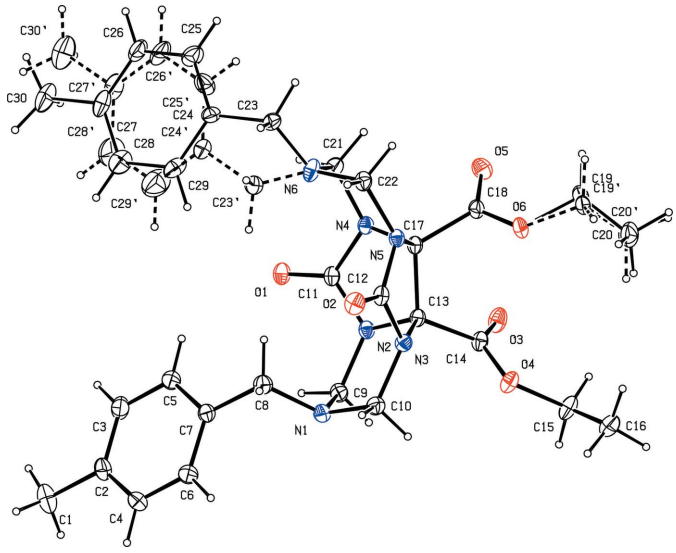

Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Bonds in the minor disorder component are shown as dashed lines.

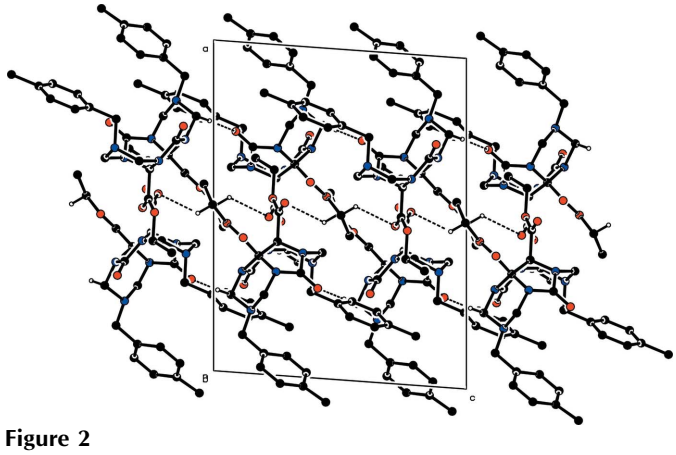

Figure 2
The molecular packing of (I), viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. Only one disorder component is shown.

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