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N,*N*′-Bis(*N*,*N*-dimethyl-*p*-toluidine)bis(ethoxycarbonyl)glycoluril

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Key indicators

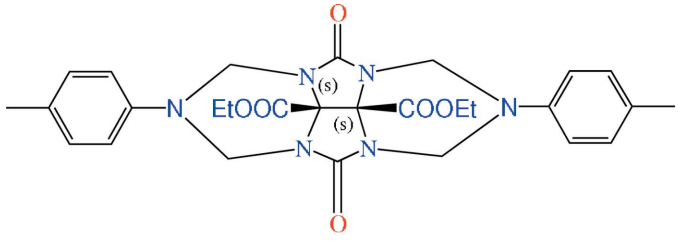
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.132
Data-to-parameter ratio = 15.7

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

In the title compound (systematic name: diethyl 4,8-dioxo-2,6-di-*p*-tolyl-1,3,5,7-tetrahydro-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]fluorene-8b,8c-dicarboxylate), $C_{28}H_{32}N_6O_6$, the dihedral angles between the two fused five-membered rings in the glycoluril unit and between the two terminal benzene rings are 71.8 (2) and 88.5 (1)°, respectively.

Comment

Since Mock and co-workers first characterized the chemical nature and structure of cucurbit[6]uril (CB[6]; Freeman *et al.*, 1981), many receptors based on glycoluril have been reported, including Nolte's molecular clips and molecular baskets (Rowan *et al.*, 1999), Rebek's molecular capsules (Hof *et al.*, 2002), CB[*n*] homologues (*n* = 5,7 and 8; Lee *et al.*, 2003) and CB[*n*] (*n* = 5–7) derivatives (Lee *et al.*, 2003; Lagona *et al.*, 2003). In this paper, we report the crystal structure of the title compound, (I), a new type of receptor based on glycoluril (Fig. 1).



(I)

Selected bond lengths and angles are listed in Table 1. The crystal packing is mainly governed by intermolecular C—H···O interactions (Table 2 and Fig. 2). The dihedral angle between the two fused five-membered rings in the glycoluril unit is 71.8 (2)° and that between the two terminal benzene rings is 88.5 (1)°.

Experimental

p-Tolylamine (2.14 g, 20 mmol) and formaldehyde (4.8 g, 80 mmol) were added to a solution of diethoxycarbonyl glycoluril (2.86 g, 10 mmol) in dimethyformamide (50 ml) under a nitrogen atmosphere. The mixture was stirred overnight. The solvent was then evaporated to dryness and the residue purified by column chromatography (hexane–ethyl acetate, 4:1), to obtain the title compound as a white solid (yield 60%, 3.27 g). Crystals of (I) suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane–methanol (2:1) solution of the title compound under ambient conditions.

Crystal data

$C_{28}H_{32}N_6O_6$
M_r = 548.60
Monoclinic, $P2_1/n$
a = 9.7963 (7) Å
b = 11.3007 (8) Å
c = 24.0399 (17) Å
β = 93.9220 (10)° **[OR 93.922 (1)°]**
V = 2655.1 (3) Å^3
Z = 4
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5116 reflections
θ = 2.3–21.9°
μ = 0.10 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
21931 measured reflections
5746 independent reflections
4052 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.0°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -28 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.132
S = 1.02
5746 reflections
365 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.6281P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C5—N1	1.406 (3)	C16—N4	1.446 (2)
C8—N1	1.440 (3)	C16—N5	1.449 (2)
C8—N3	1.467 (2)	C20—N4	1.444 (2)
C9—N1	1.442 (3)	C20—N6	1.475 (2)
C9—N2	1.464 (2)	C22—N6	1.436 (2)
N1—C8—N3	110.60 (16)	N4—C20—N6	110.61 (14)
N1—C9—N2	111.20 (16)	N5—C21—N6	109.79 (14)
N3—C8—N1—C5	−120.5 (2)	N5—C21—N6—C22	176.08 (14)
N2—C9—N1—C5	120.8 (2)	N4—C20—N6—C22	−175.80 (14)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18B···O5^{i}	0.97	2.53	3.336 (2)	141

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

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, allowing for free rotation of the methyl groups. The constraint U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq} (methyl C) was applied.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); 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, 2001); software used to prepare material for publication: *SHELXTL*.

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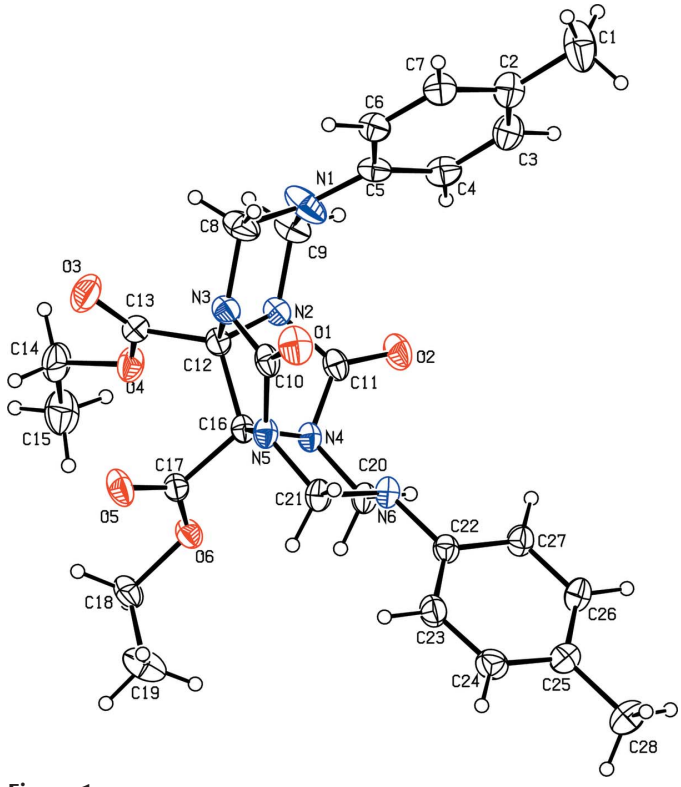

Figure 1
A view of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 50% probability level.

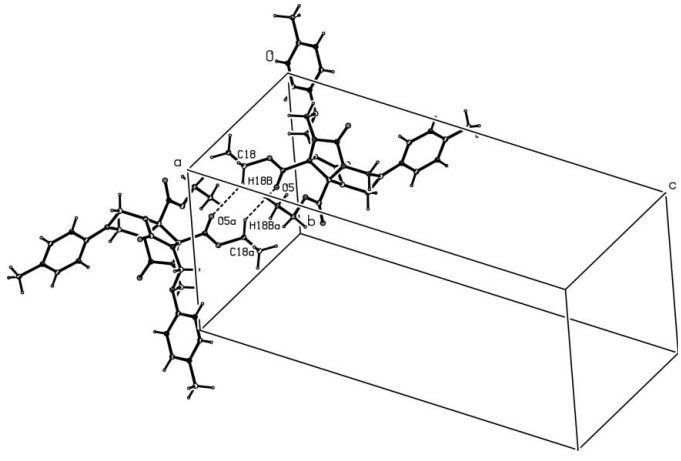

Figure 2
The C—H···O intermolecular interactions (dashed lines) in the crystal structure of (I). The suffix a corresponds to symmetry code (i) in Table 2.

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