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Diethyl 4,8-dioxo-2,6-diphenyl-1,3,5,7-tetrahydro-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) = 0.005 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.181
Data-to-parameter ratio = 12.7

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

In the title compound, $C_{26}H_{28}N_6O_6$, the dihedral angle between the two five-membered rings is 73.1 (2); that between the two phenyl rings is 60.1 (1)°.

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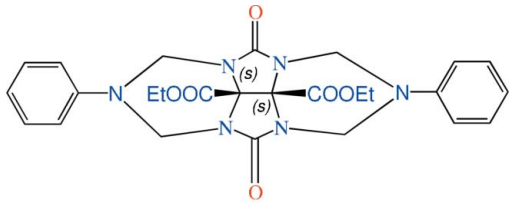
Comment

Glycoluril and its derivatives are widely used as building blocks in supramolecular chemistry. The title compound, (I) (Fig. 1), is a new kind of supramolecular building block and it can bind some guest molecules as the molecular clips receptor. We present the crystal structure of (I) as a continuation of our previous studies in this area (Wei & Wu, 2005).



(I)

One of the ethyl groups (C17/C18) shows positional disorder. The molecules are connected mainly by C—H···O

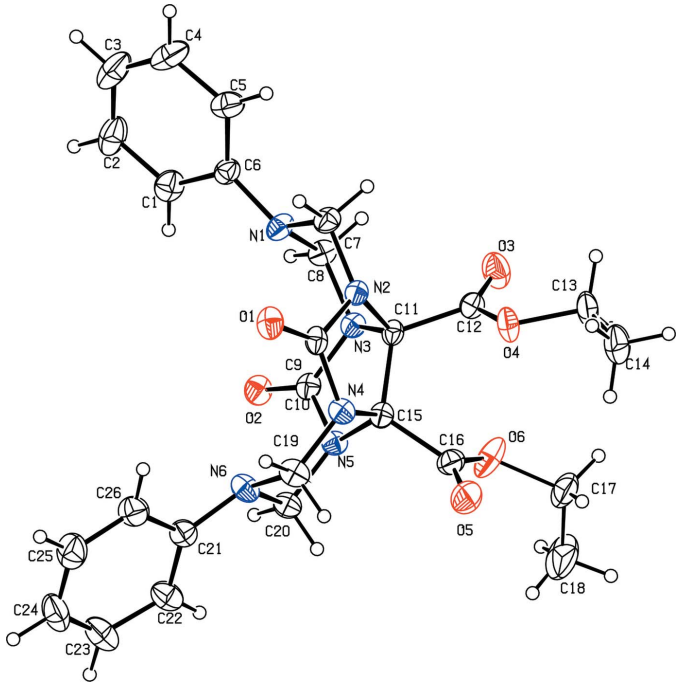


Figure 1
View of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Only the major component of the disordered C17/C18 ethyl group is shown.

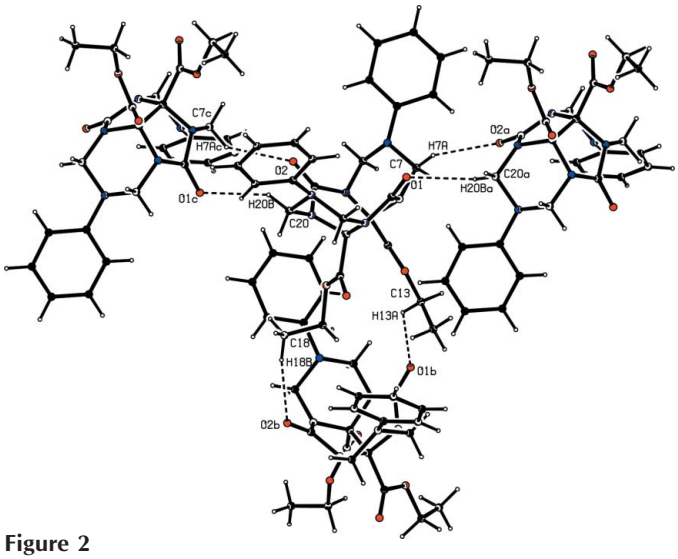


Figure 2
The C—H···O intermolecular interactions (dashed lines) in the crystal structure.

intermolecular interactions (Fig. 2). The C···O distances of these carbonyl groups are similar to those reported in the literature (Desiraju, 1996). Selected bond lengths and angles are listed in Table 1. The dihedral angle between two five-membered rings in glycoluril is 73.1 (2)°, and that between the two terminal phenyl rings is 60.1 (1)°.

Experimental

Phenylamine (1.86 g, 20 mmol) and formaldehyde (4.8 g, 80 mmol) were added to a stirred solution of diethoxycarbonyl glycoluril (2.86 g, 10 mmol) in *N*,*N*-dimethylformamide (50 ml) under a dinitrogen atmosphere. The mixture was stirred overnight and the solvent was evaporated to dryness and purified by column chromatography (hexane–EtOAc = 8:1) to obtain the title compound (yield 3.64 g, 70%) as a white solid. Crystals suitable for X-ray diffraction were grown by slow evaporation of $CDCl_3$ solutions of the title compound under ambient conditions after ^{13}C NMR analysis. ^1H NMR ($CDCl_3$, 400 MHz): δ 7.12 (*t*, 4H), 6.94 (*d*, 4H), 6.88 (*t*, 2H), 5.94 (*d*, 4H, *J* = 13.2 Hz), 4.56 (*d*, 4H, *J* = 13.2 Hz), 4.33 (*q*, 4H), 1.35 (*t*, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): 165.13, 157.32, 145.86, 129.24, 121.71, 117.73, 76.13, 63.33, 58.31, 13.84.

Crystal data

$C_{26}H_{28}N_6O_6$
M_r = 520.54
Orthorhombic, *Pbca*
a = 18.0634 (16) Å
b = 15.1108 (13) Å
c = 18.8377 (17) Å
V = 5141.8 (8) Å^3
Z = 8
D_x = 1.345 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 5199 reflections
θ = 2.6–22.1°
μ = 0.10 mm^{-1}
T = 292 (2) K
Block, colorless
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.962, T_{max} = 0.971
27955 measured reflections
4521 independent reflections
3607 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25.0°
h = −21 → 21
k = −17 → 17
l = −22 → 22

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
S = 1.18
4521 reflections
355 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 1.3901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C7—N1	1.449 (3)	C15—N4	1.457 (3)
C7—N2	1.458 (3)	C19—N4	1.451 (3)
C8—N3	1.463 (3)	C19—N6	1.462 (3)
C8—N1	1.465 (3)	C20—N5	1.455 (3)
C15—N5	1.456 (3)	C20—N6	1.462 (3)
N1—C7—N2	108.44 (19)	N5—C15—N4	110.9 (2)
N3—C8—N1	109.2 (2)	N4—C19—N6	109.7 (2)
N3—C11—N2	111.86 (19)	N5—C20—N6	109.1 (2)
N1—C7—N2—C11	52.8 (3)	N6—C19—N4—C15	−51.6 (3)
N1—C8—N3—C11	−50.3 (3)	N6—C20—N5—C15	52.0 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O1^{i}	0.97	2.49	3.169 (4)	127
C18—H18*B*···O2^{i}	0.96	2.60	3.533 (7)	164
C20—H20*B*···O1^{ii}	0.97	2.51	3.340 (3)	144
C7—H7*A*···O2^{iii}	0.97	2.50	3.326 (3)	143

Symmetry codes: (i) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, z$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$.

One of the ethyl groups (C17/C18) is found to be disordered over two orientations. The occupancies of the disordered positions C17/C17′ and C18/C18′ were refined to 0.817 (15) and 0.183 (15), respectively. 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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