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Diethyl 2,13-dioxo-16,18-dioxa-1,3,12,14-tetraazapentacyclo[10.7.2.$0^{3,21}$.$0^{5,10}$.$0^{14,20}$]-undeca-5,7,9-triene-20,21-dicarboxylate

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The title compoud, $C_{21}H_{24}N_4O_8$, is an important intermediate for the synthesis of more complex glycoluril derivatives. The crystal structure is stabilized mostly by intermolecular C—H···O and C—H···π interactions.

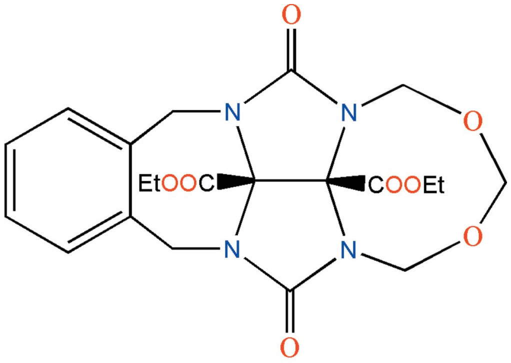
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.064
wR factor = 0.196
Data-to-parameter ratio = 11.5

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

Comment

The glycoluril skeleton has served as an important building block for the preparation of a wide variety of supramolecular systems, including molecular clips (Rowan *et al.*, 1999; Chakraborty *et al.*, 2002; Yin *et al.*, 2006; Li *et al.*, 2006), the cucurbit[*n*]uril family (Freeman *et al.*, 1981) and molecular capsules (Hof *et al.*, 2002; Rebek, 2005). Supramolecular building blocks based on diethoxycarbonyl glycoluril have been the subject of recent interest (Chakraborty *et al.* 2002; Wu *et al.*, 2002). Diethoxycarbonyl glycoluril derivatives bearing potentially electrophilic cyclic ether groups have been shown to be important intermediates in the synthesis of more complex glycoluril derivatives (Wu *et al.*, 2002). As a part of our research on glycoluril derivatives (Chen *et al.*, 2006), we report here the structure of the title compound, (I).



(I)

The bowl-shaped system of five fused rings in (I) is shown in Fig. 1. The ethoxycarbonyl groups are disordered over two positions. The crystal structure is stabilized by C—H···O and C—H···π interactions (Table 1).

Experimental

The title compound was synthesized according to the procedure of Wu *et al.* (2002) in 14% isolated yield. Crystals appropriate for data collection were obtained by slow evaporation of a dichloroethane–methanol solution (4:1 *v*/*v*) at 293 K.

Crystal data

$C_{21}H_{24}N_4O_8$
$M_r = 460.44$
Monoclinic, $P2_1/c$
$a = 8.4954$ (8) Å
$b = 15.0557$ (13) Å
$c = 17.2379$ (16) Å
$\beta = 91.541$ (2)°
$V = 2204.0$ (3) Å^3

$Z = 4$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
12355 measured reflections

4325 independent reflections
3087 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\rm max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.196$
$S = 1.01$
4325 reflections
376 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1286P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.84$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

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

Cg is the centroid of the benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2$^{\rm i}$	0.93	2.41	3.282 (3)	157
C3—H3···O7$^{\rm ii}$	0.93	2.53	3.434 (3)	165
C13—H13*B*···O7$^{\rm iii}$	0.97	2.56	3.374 (10)	141
C8—H8*A*···*Cg*$^{\rm iv}$	0.97	2.79	3.679 (3)	153

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{5}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x, -y, -z+2$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, and methyl groups were allowed to rotate freely about their C—C bonds. The ethoxycarbonyl groups are disordered over two orientations, with refined occupancy factors of 0.67 (2) and 0.33 (2) for the ethoxycarbonyl groups containing atoms O3 and O3′, and 0.514 (11) and 0.486 (11) for the groups containing atoms O5 and O5′. Restraints were imposed on the geometry and displacement parameters of the ethoxycarbonyl groups.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

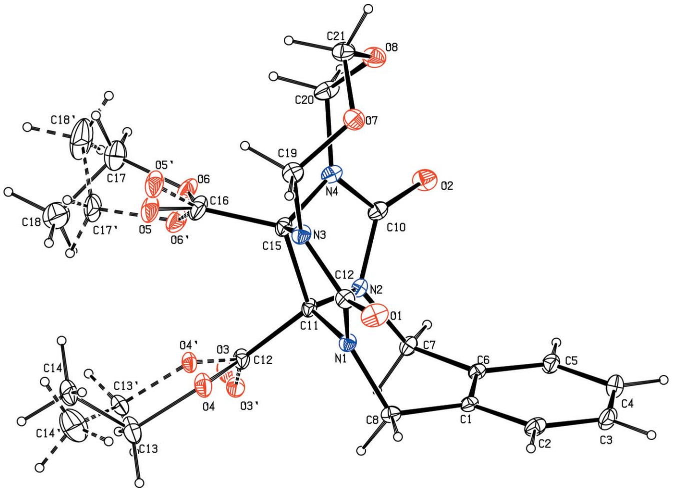


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 10% probability level and H atoms are represented by circles of arbitrary size. Atoms of the minor disorder components are linked by dashed lines.

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