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Key indicators

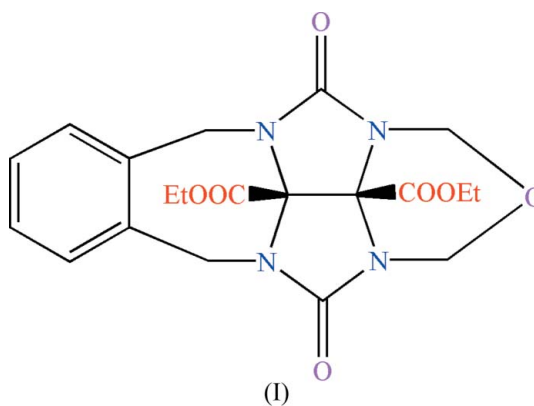
Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.052
 wR factor = 0.145
Data-to-parameter ratio = 13.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Diethyl *cis*-4,5,10,11-tetrahydro-4,11-
dioxo-1*H*,3*H*-2-oxa-3*a*,4*a*,10*a*,11*a*-tetra-
azabenz[*f*]indeno[2,1,7-*ija*]azulene-
11*b*,11*c*-dicarboxylate

The title compound, $\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_7$, is a glycoluril derivative with five fused rings. A benzene ring is fused to a seven-membered ring which includes two of the N atoms from separate rings of the glycoluril system, and the other two N atoms are linked to an O atom by two methylene groups. The structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

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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). In addition, Isaacs and co-workers have synthesized many methylene-bridged glycoluril dimers from the title compound (Wu, Fettinger *et al.*, 2002). As a part of our ongoing investigation of glycoluril derivatives (Zhou *et al.*, 2005), we report here the structure of the title compound, (I) (Fig. 1).



The crystal packing is stabilized by inversion-related intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming dimers. These dimers are linked by additional $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds involving the carbonyl O atoms of the glycoluril system (Table 1 and Fig. 2).

Experimental

The title compound was synthesized according to the procedure of Wu, Chakraborty *et al.* (2002), using paraformaldehyde (0.18 g, 6 mmol), 1,2-bis(chloromethyl)benzene (0.18 g, 10 mmol), and glycoluril (5.72 g, 20 mmol) as starting materials. Crystals appropriate for data collection were obtained by slow evaporation of a dichloroethane–methanol (4:1 *v/v*) solution at 293 K.

Crystal data

C₂₀H₂₂N₄O₇
M_r = 430.42
 Triclinic, *P*1̄
a = 8.1503 (7) Å
b = 11.1548 (9) Å
c = 11.9935 (10) Å
 α = 95.016 (1)°
 β = 104.297 (1)°
 γ = 111.285 (1)°

V = 965.42 (14) Å³
Z = 2
D_x = 1.481 Mg m⁻³
 Mo *K*α radiation
 μ = 0.11 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
 9092 measured reflections

4167 independent reflections
 3560 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.057
 θ_{max} = 27.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.052
wR(*F*²) = 0.145
S = 1.05
 4167 reflections
 321 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0791P)^2 + 0.1548P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.24 e Å⁻³
 Δρ_{min} = -0.36 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i> ⋯ <i>A</i>
C17—H17B⋯O2 ⁱ	0.97	2.51	3.373 (3)	148
C19—H19B⋯O1 ⁱⁱ	0.97	2.55	3.457 (2)	156
C20—H20A⋯O2 ⁱⁱ	0.97	2.47	3.217 (2)	133

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) -*x* + 1, -*y* + 2, -*z* + 1.

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C), and methyl groups were allowed to rotate freely about their C—C bonds. All other 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*_{iso}(H) = 1.2*U*_{eq}(C). The O4 ethoxy group and the O5 atom of the carbonyl group of the other ester are disordered over two orientations. The occupancies of the disordered atoms C13/C13', C14/C14' and O4/O4' refined to 0.650 (11)/0.350 (11), and O5/O5' refined to 0.536 (19)/0.464 (19).

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*.

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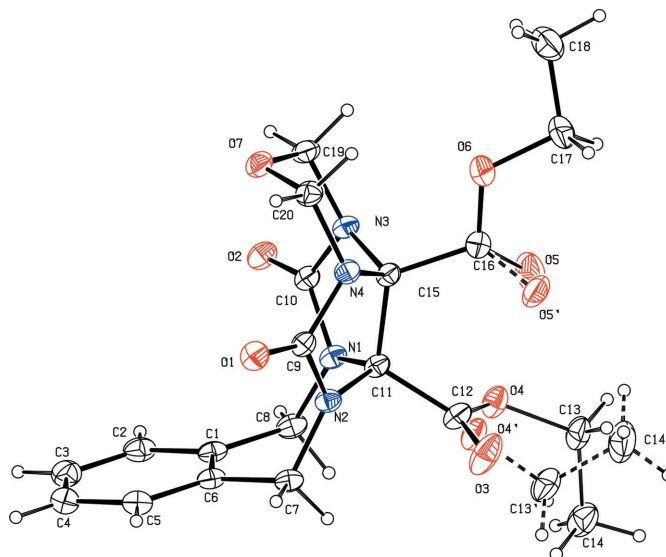


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary size. Bonds to atoms of the minor disorder components are drawn as dashed lines.

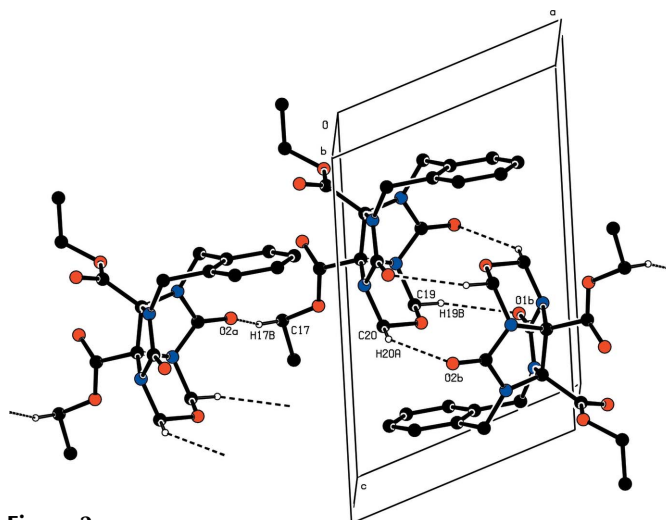


Figure 2

Packing of (I) with hydrogen bonds drawn as dashed lines. For the sake of clarity, H atoms not involved in these interactions are omitted. [Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) -*x* + 1, -*y* + 2, -*z* + 1.] Only one disorder component is shown.

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