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Diethyl *cis*-1,2,3,4,5,10-hexahydro-7,8-dimethyl-1,4-dioxo-2,3,4a,10a-tetraazabenzo[*g*]cyclopent-[*cd*]azulene-2a,10b-dicarboxylate chloroform disolvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.119
Data-to-parameter ratio = 17.3

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

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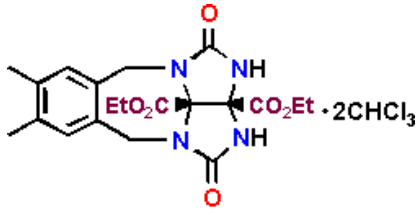
The crystal structure of the title compound, 4,5-dimethyl-1,2-xylylene-derived diethoxycarbonyl glycoluril, $C_{20}H_{24}N_4O_6\cdot 2CHCl_3$, is reported. In the crystal structure, there are not only intermolecular hydrogen bonds, but also π–π stacking interactions.

Comment

Glycoluril is a bisurea, formed in the condensation reaction of two equivalents of urea with one equivalent of glyoxal. Glycoluril derivatives have a wide range of applications, such as explosives, slow-release fertilizers, crosslinkers and iodogens, and in the stabilization of organic compounds against photodegradation, combinatorial chemistry, *etc*. (Wu, Fettinger *et al.*, 2002). In 1981, Mock and co-workers established and named the structure of cucurbit[*n*]uril (CB[*n*], *n* = 6), which is a remarkable macrocyclic compound comprising six glycoluril rings and 12 methylene bridges (Freeman *et al.*, 1981). Since then, 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, 8 and 10) and derivatives (Lee *et al.*, 2003), CB[*n*] analogues (Lagona *et al.*, 2003), and anion-binding receptors (Kang *et al.*, 2004). In additon, Isaacs and co-workers have synthesized many methylene-bridged glycoluril dimers from organic-soluble diethoxycarbonyl glycoluril derivatives (Witt *et al.*, 2000). On this basis, they also proposed the important self-sorting phenomenon (Wu & Isaacs, 2003). In this paper, we present the X-ray crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond distances are listed in Table 1. The molecules are connected by N3—H3*A*···O2^{i} and O1···H4^{i}—N4^{i} hydrogen bonds [Table 2; symmetry code: (i) $\frac{1}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$], forming a ribbon structure (Fig. 2).

In the crystal structure, there is a weak π–π stacking interaction. The distance between *Cg*1 and *Cg*1^{ii} is 3.692 Å, where *Cg*1 is the centroid of the benzene ring [symmetry code: (ii) $1-x$, y, $\frac{1}{2}-z$].

Experimental

The title compound was synthesized according to the literature procedure of Wu, Chakraborty *et al.* (2002) in 55% yield. Crystals of (I) appropriate for data collection were obtained by slow evaporation from a methanol–chloroform (1:25 *v*/*v*) solution at 283 K.

Crystal data

$C_{20}H_{24}N_4O_6\cdot 2CHCl_3$
$M_r = 655.17$
Monoclinic, $C2/c$
$a = 25.216$ (3) Å
$b = 9.9757$ (9) Å
$c = 25.566$ (3) Å
$\beta = 108.566$ (3)°
$V = 6096.5$ (12) Å^3
$Z = 8$
$D_x = 1.428$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3132 reflections
$\theta = 2.3$–21.7°
$\mu = 0.61$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART Apex CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.839$, $T_{max} = 0.868$
16 255 measured reflections
5990 independent reflections
3790 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 26.0°$
$h = -30 \rightarrow 31$
$k = -11 \rightarrow 12$
$l = -31 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.119$
$S = 1.02$
5990 reflections
347 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.22P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C4—C9	1.505 (5)	C15—O3	1.187 (4)
C9—N1	1.440 (4)	C16—O4	1.416 (4)
C10—N1	1.471 (4)	C16—C17	1.503 (5)
C10—C13	1.583 (5)	C22—Cl4	1.706 (4)
C12—O1	1.226 (4)	C22—Cl6	1.717 (4)
C12—N1	1.366 (4)	C22—Cl5	1.739 (4)
C13—C15	1.525 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O2^{i}	0.80	2.09	2.880 (4)	172
N4—H4···O1^{ii}	0.80	2.03	2.833 (4)	173

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

The amide H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(N)$ and N—H = 0.80 Å. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C—C bond. 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 0.95–1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

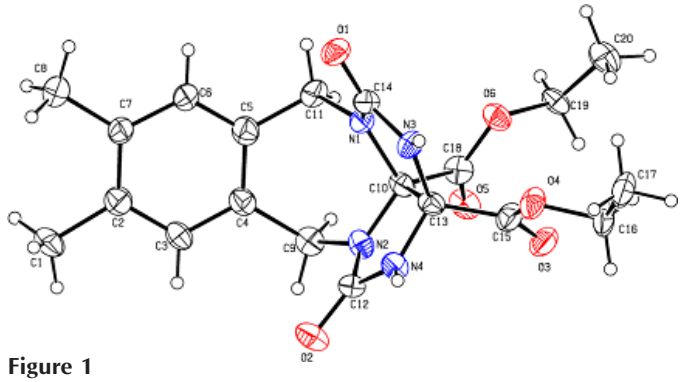


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 50% probability level. The chloroform molecules have been omitted for clarity.

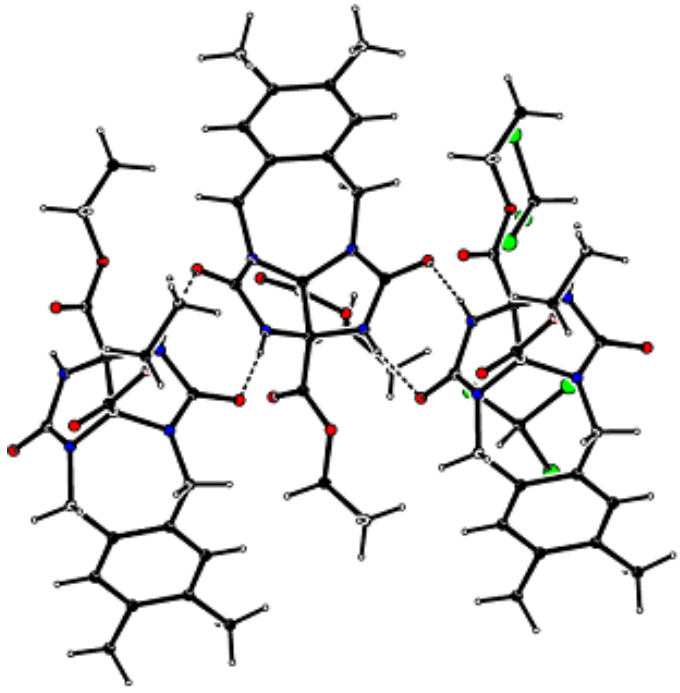


Figure 2
Intermolecular hydrogen bonding in the crystal structure of (I). Hydrogen-bonding interactions are indicated by dashed lines.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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