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Diethyl 5,10-dihydro-6-iodo-4,11-dioxo-*cis*-1*H*,3*H*,4*H*,11*H*-2-oxa-3a,4a,10a,11a-tetraazabenz[*f*]indeno[2,1,7-*ija*]azulene-11b,11c-dicarboxylate

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The title compound, $C_{20}H_{21}IN_4O_7$, is a glycoluril derivative which contains five fused rings. An iodobenzene ring is fused to a seven-membered ring linked to two of the N atoms from the separate rings of the glycoluril system, and the other two N atoms are linked to an O atom by two of the methylene groups. The structure of the compound possesses normal geometrical parameters. The crystal packing is consolidated by intermolecular hydrogen bonds, resulting in a two-dimensional network.

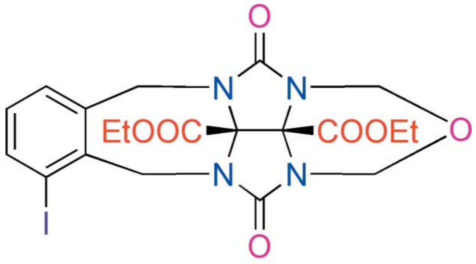
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.148
Data-to-parameter ratio = 16.1

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

Comment

Since Freeman *et al.* (1981) first 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, many glycoluril derivatives have been synthesized. Witt *et al.* (2000) have comprehensively synthesized many methylene-bridged glycoluril dimers from organic-soluble diethoxycarbonyl glycoluril derivatives and discuss many methods for the synthesis of glycoluril derivatives. In the present paper, we report the crystal structure of the title compound, (I) (Fig. 1), a useful intermediate for glycoluril derivatives of a more complex nature.



(I)

Selected bond lengths and angles are listed in Table 1. In the crystal structure, analysis using *PLATON* (Spek, 2003) shows that no obvious C—H···π and π–π interactions are observed. However, pairs of molecules are associated by intermolecular complementary hydrogen bonds; at the same time, each molecule also forms hydrogen bonds with another molecule (Table 2), resulting in a two-dimensional network (Fig. 2).

Experimental

Compound (I) was synthesized according to the procedure reported by Wu *et al.* (2002). Crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a dichloromethane and hexane (*v*/*v* 1:2) solution at 283 K.

Crystal data

$C_{20}H_{21}IN_4O_7$
$M_r = 556.31$
Orthorhombic, $Pbca$
$a = 15.533\ (6)$ Å
$b = 11.414\ (5)$ Å
$c = 24.006\ (10)$ Å
$V = 4256\ (3)$ Å^3
$Z = 8$
$D_x = 1.736$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.56$ mm^{-1}
$T = 292\ (2)$ K
Block, colorless
$0.10 \times 0.05 \times 0.02$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
46045 measured reflections
4856 independent reflections
2978 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.134$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.148$
$S = 0.94$
4856 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.92$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—I1	2.099 (5)	C9—N1	1.389 (5)
C6—C7	1.534 (6)	C19—O7	1.427 (6)
C7—N1	1.449 (5)	C19—N3	1.440 (6)
C9—O1	1.196 (5)		
C6—C1—I1	122.3 (3)	O7—C19—N3	111.0 (3)
C5—C6—C7	118.8 (4)	C9—N3—C19	121.1 (4)
N1—C7—C6	114.9 (3)	C19—N3—C15	116.3 (4)
N2—C11—N1	113.5 (3)	C20—O7—C19	110.6 (4)
N2—C11—C12	108.5 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C19—H19$B\cdots$O2$^{\rm i}$	0.97	2.49	3.251 (6)	135
C13—H13$A\cdots$O4$^{\rm ii}$	0.97	2.53	3.302 (6)	136
C8—H8$B\cdots$O2$^{\rm ii}$	0.97	2.35	3.246 (5)	153
C7—H7$A\cdots$O2$^{\rm ii}$	0.97	2.47	3.353 (5)	152

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

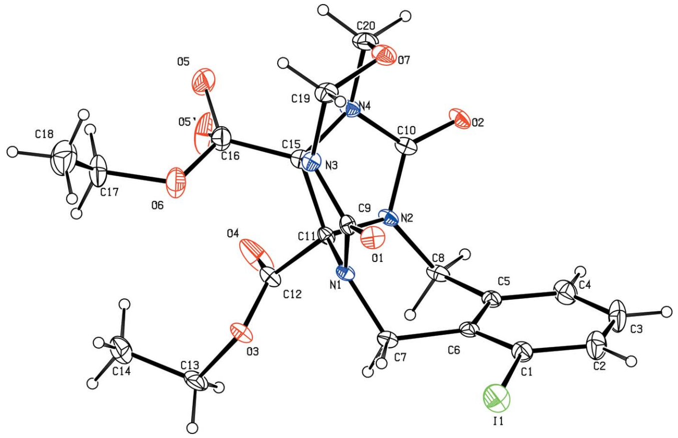


Figure 1
A view of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

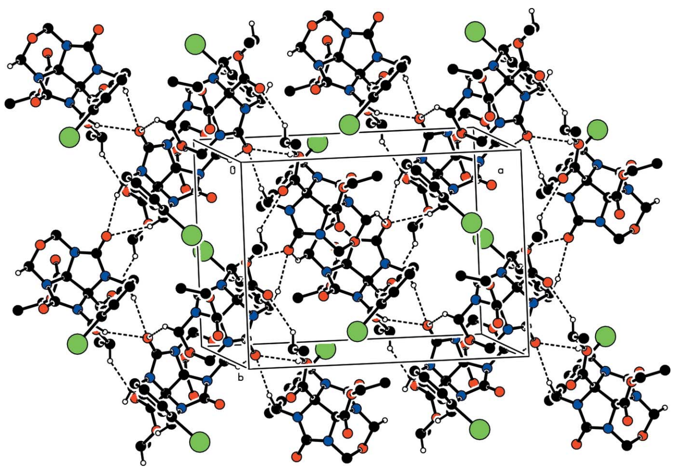


Figure 2
Plot of the crystal packing forming a two-dimensional network in the *ab* plane. Hydrogen bonds are shown as dashed lines.

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 $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. All other H atoms were placed in geometrically idealized positions, with C—H distances in the range 0.93–0.97 Å, and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Atom O5 is disordered over two sites; the site-occupancy factors for the two orientations were refined, giving 0.78 (2) and 0.22 (2) for the major and minor components, respectively. The high $R_{\rm int}$ was the result of the poor quality of the crystal; attempts to obtain a larger crystal were unsuccessful.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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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