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Diethyl *cis*-1,2,3,4,5,10-hexahydro-6-iodo-1,4-dioxo-2,3,4a,10a-tetraazabenzo[*g*]-cyclopent[*cd*]azulene-2a,10b-dicarboxylate

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The title compound, $C_{18}H_{19}IN_4O_6$, is a glycoluril derivative which contains four 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. The structure is stabilized by intermolecular hydrogen bonds and π–π-stacking interactions.

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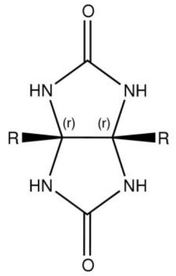
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
R factor = 0.042
wR factor = 0.117
Data-to-parameter ratio = 13.2

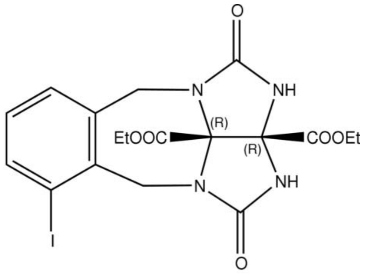
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Glycoluril derivatives (see scheme) have been widely used in molecular recognition, self-assembly, self-sorting and catalysis. Work by various groups has devised many practical synthetic methods for the preparation of such derivatives (Rowan *et al.*, 1999; Sijbesma & Nolte, 1995; Rebek, 1996, 1999; Wu *et al.*, 2002). An important step in many of these syntheses involves the nucleophilic addition of glycoluril anions to 1,2-bis-(halomethyl)aromatics to generate glycoluril derivatives bearing *o*-xylylene rings on one or both sides of the glycoluril skeleton. We have obtained a new glycoluril derivative, (I), (Fig. 1) by the reaction of 1,2-bis(bromomethyl)-3-iodobenzene with diethoxycarbonyl glycoluril.



Glycoluril dervatives, R = H, COOEt, Me, Ph, $(CH_2)_4$, 2-pyridyl



(I)

In the crystal structure, there are three hydrogen bonds (Fig. 3 and Table 2); two are strong N—H$\cdot\cdot\cdot$O bonds that link pairs of molecules from adjacent sheets. The sheets form as a result of the third, weaker C—H$\cdot\cdot\cdot$O bond. Additional stabilization comes from π–π stacking interactions (Janiak, 2000) between adjacent iodobenzene rings (Fig. 2). The distance between the centroids of adjacent iodobenzene rings is 3.624 (3) Å and the dihedral angle between them is 0.02 (1)°.

Experimental

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

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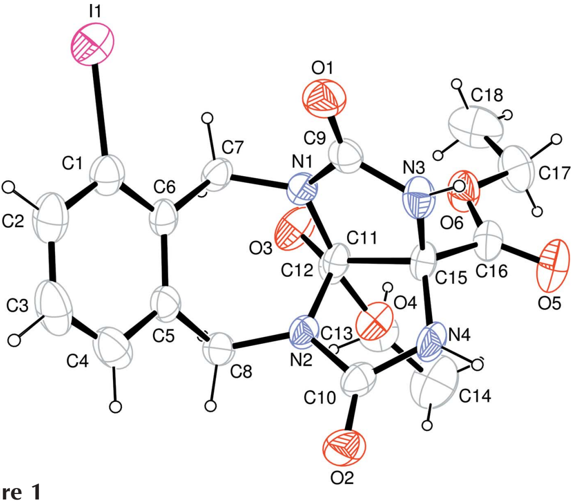

Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

Crystal data

$C_{18}H_{19}IN_4O_6$
$M_r = 514.27$
Triclinic, $P\overline{1}$
$a = 8.0414$ (8) Å
$b = 8.7014$ (8) Å
$c = 14.3196$ (14) Å
$\alpha = 87.205$ (2)°
$\beta = 88.374$ (2)°
$\gamma = 84.521$ (2)°
$V = 995.92$ (17) Å^3
$Z = 2$
$D_x = 1.715$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4277 reflections
$\theta = 2.6$–28.2°
$\mu = 1.65$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.637$, $T_{max} = 0.734$
5656 measured reflections
3485 independent reflections
3231 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 9$
$l = -17 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.117$
$S = 1.04$
3485 reflections
264 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 2.2689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.26$ e Å^{-3}
$\Delta\rho_{min} = -1.03$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—I1	2.087 (5)	C9—N1	1.384 (5)
C7—N1	1.463 (5)	C15—N4	1.434 (5)
C9—O1	1.203 (5)	C16—O6	1.320 (6)
C9—N3	1.378 (6)		
C6—C1—I1	123.6 (3)	N2—C11—C15	101.1 (3)
C1—C6—C7	122.2 (4)	N4—C15—N3	115.1 (3)
N1—C7—C6	114.8 (3)	N3—C15—C16	108.6 (3)
O1—C9—N1	126.1 (4)		
C6—C1—C2—C3	0.6 (7)	C4—C5—C8—N2	−121.3 (4)
I1—C1—C2—C3	179.0 (4)	N4—C15—N3—C9	−116.5 (4)
C5—C6—C7—N1	−59.8 (5)	N3—C15—N4—C10	92.9 (4)

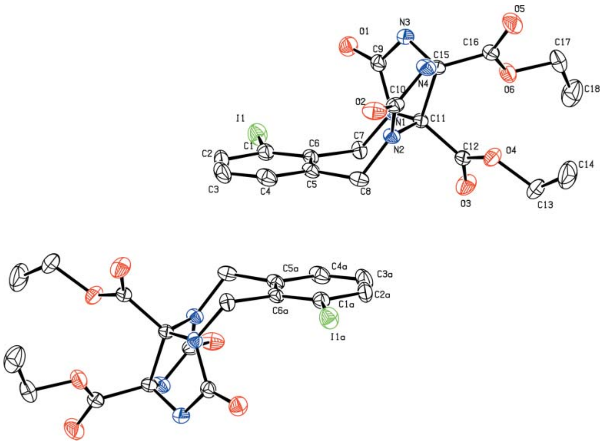

Figure 2
π–π stacking of the aromatic rings in (I). [Symmetry code: (*a*) $1 - x$, $2 - y$, $1 - z$.]

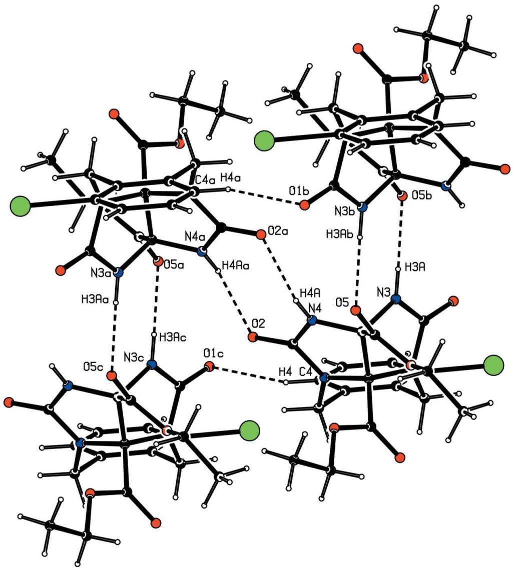

Figure 3
The intermolecular hydrogen bonding in (I). Hydrogen bonds are drawn as dashed lines. [Symmetry codes: (*a*) $1 + x$, y, z; (*b*) $-x$, $2 - y$, $-z$; (*c*) $1 - x$, $2 - y$, $-z$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^{i}	0.93	2.41	3.247 (6)	149
N3—H3A···O5^{ii}	0.86	2.16	3.020 (5)	173
N4—H4A···O2^{iii}	0.86	2.07	2.828 (4)	146

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

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 $U_{iso}(H) = 1.5U_{eq}(C)$. 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 $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak and deepest hole are located 0.84 and 0.72 Å from atoms I1 and I1, respectively.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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