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## Key indicators

Single-crystal X-ray study  
 $T = 292\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$   
Disorder in main residue  
 $R$  factor = 0.048  
 $wR$  factor = 0.125  
Data-to-parameter ratio = 12.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Diethyl 7,12-dihydro-8-iodo-6,13-dioxo-*cis*-7*H*,11*H*-  
2,4-dioxa-5*a*,6*a*,12*a*,13*a*-tetraazabenz[*f*]cycloocta-  
[*cd*]azulene-13*b*,13*c*-dicarboxylate

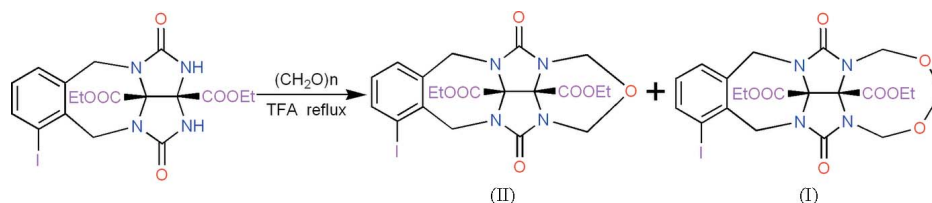
Received 23 May 2006

Accepted 2 June 2006

The title compound,  $\text{C}_{21}\text{H}_{23}\text{I}\text{N}_4\text{O}_8$ , is a glycoluril derivative with five fused rings. An iodobenzene ring is fused to a seven-membered ring which binds two of the N atoms from separate rings of the glycoluril system. The other N atoms are linked *via* methylene groups to form an eight-membered ring with two ether O atoms separated by an additional methylene group. The crystal structure is stabilized by inversion-related  $\text{C}-\text{H}\cdots\pi$  interactions together with  $\text{C}-\text{H}\cdots\text{O}$  non-classical hydrogen bonds.

## Comment

The glycoluril skeleton has served as an important building block for the preparation of a wide variety of supramolecular assemblies, including molecular clips and baskets (Rowan *et al.*, 1999), molecular capsules (Hof *et al.*, 2002), cucurbituril[*n*] homologues ( $n = 5, 7, 8$  and 10) and their derivatives (Lee *et al.*, 2003), and anion-binding receptors (Kang *et al.*, 2004). Supramolecular building blocks based on diethoxycarbonyl glycoluril are more soluble and have been the subject of recent research (Chakraborty *et al.* 2002; Wu, Chakraborty, Fettingner *et al.* 2002). Diethoxycarbonyl glycoluril derivatives bearing potentially electrophilic cyclic ether groups were shown to be important intermediates for more complex glycoluril derivatives. These were prepared *via* condensation reactions in refluxing trifluoroacetic acid containing paraformaldehyde (Wu, Chakraborty, Witt *et al.*, 2002) with (I) as the kinetic product. The more stable cyclic ether (II) is also produced and its structure was reported previously (Chen *et al.*, 2006). Here we report the structure of the title compound (I) (Fig. 1 and Table 1).



In the crystal structure, inversion-related  $\text{C8}-\text{H8B}\cdots\pi$  interactions to the iodobenzene ring link pairs of molecules (Table 2;  $\text{Cg}$  is the centroid of the iodobenzene ring).  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds link these pairs into chains along [100] (Fig. 2).

## Experimental

The title compound was synthesized according to the literature procedure (Wu, Chakraborty, Witt *et al.*, 2002) in 10% isolated yield.

Crystals of (I) suitable for data collection were obtained by slow evaporation of a dichloromethane and hexane (1:4 v/v) solution at 283 K.

#### Crystal data

$C_{21}H_{23}IN_4O_8$	$V = 1096.5 (3) \text{ \AA}^3$
$M_r = 570.33$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.727 \text{ Mg m}^{-3}$
$a = 8.2832 (12) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.7080 (14) \text{ \AA}$	$\mu = 1.51 \text{ mm}^{-1}$
$c = 14.872 (2) \text{ \AA}$	$T = 292 (2) \text{ K}$
$\alpha = 99.300 (2)^\circ$	Irregular, colorless
$\beta = 97.076 (2)^\circ$	$0.30 \times 0.20 \times 0.15 \text{ mm}$
$\gamma = 108.921 (2)^\circ$	

#### Data collection

Bruker SMART Apex CCD area-detector diffractometer	3785 independent reflections
$\omega$ scans	3405 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.019$
6102 measured reflections	$\theta_{\text{max}} = 25.0^\circ$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 1.3642P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.125$	$(\Delta\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.50 \text{ e \AA}^{-3}$
3785 reflections	$\Delta\rho_{\text{min}} = -0.61 \text{ e \AA}^{-3}$
313 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C8—N2	1.474 (6)	C11—N2	1.425 (6)
C9—O1	1.202 (6)	C15—C16	1.540 (8)
C9—N2	1.375 (6)	C20—N4	1.432 (7)
O1—C9—N2	126.3 (5)	N4—C15—N3	113.5 (4)
N2—C9—N4	107.0 (4)	C9—N2—C8	120.1 (4)

**Table 2**

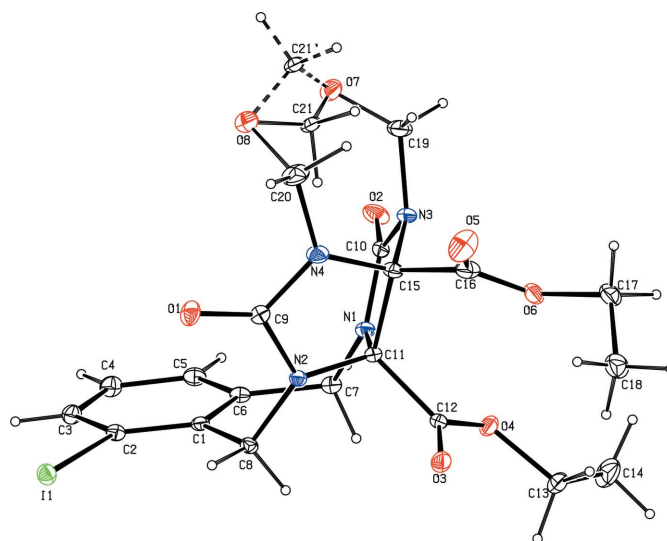
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8A $\cdots$ I1	0.97	2.72	3.363 (5)	124
C5—H5 $\cdots$ O1 <sup>i</sup>	0.93	2.45	3.290 (7)	151
C8—H8B $\cdots$ Cg1 <sup>ii</sup>	0.97	2.91	3.716 (5)	141

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

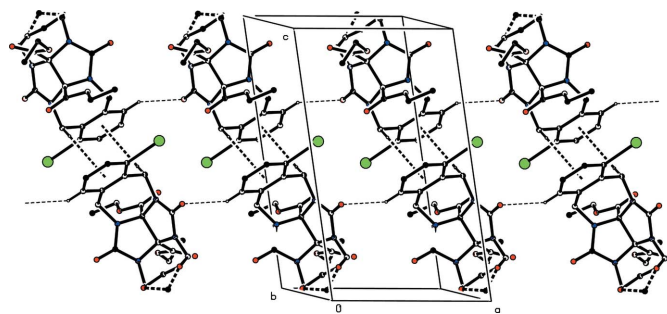
All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of  $0.96 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . All other H atoms were placed in geometrically idealized positions, with C—H distances in the range  $0.93\text{--}0.97 \text{ \AA}$ , and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . C21 is disordered over two sites; the site-occupancy factors for the two components were refined to 0.64 (1) and 0.36 (1) for the major and minor components, respectively. The highest peak is located  $0.94 \text{ \AA}$  from I1.

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



**Figure 1**

View of the molecular structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Both disorder components are shown.



**Figure 2**

Crystal packing for (I), with hydrogen bonds and C—H $\cdots\pi$  interactions drawn as dashed lines. For the sake of clarity, H atoms not involved in these interactions have been omitted. Both disorder components are shown.

structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

We thank Professor Anxin Wu (Central China Normal University, Wuhan, China) for helpful discussions, and Dr Xiang-Gao Meng for the X-ray data collection. This work was supported by the Hubei Province Natural Science Fund (No. 2004ABA085 and No. 2004ABC002).

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