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#### Key indicators

Single-crystal X-ray study  
T = 292 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
R factor = 0.045  
wR factor = 0.135  
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>.

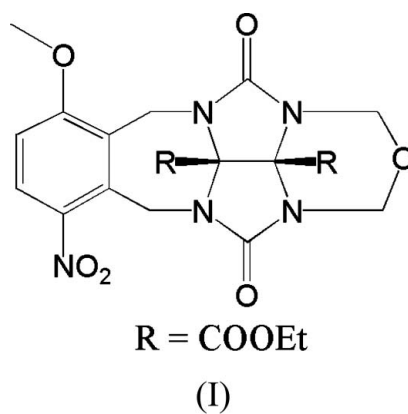
## Diethyl 5,10-dihydro-9-methoxy-6-nitro-4,11-dioxo-cis-1H,3H,4H,11H-2-oxa-3a,4a,10a,11a-tetraazabenz[*f*]indeno[2,1,7-*ija*]azulene-11b,11c-dicarboxylate

The crystal structure of the title compound,  $\text{C}_{21}\text{H}_{23}\text{N}_5\text{O}_{10}$ , an important intermediate for molecular tweezers, shows intermolecular hydrogen bonding.

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#### Comment

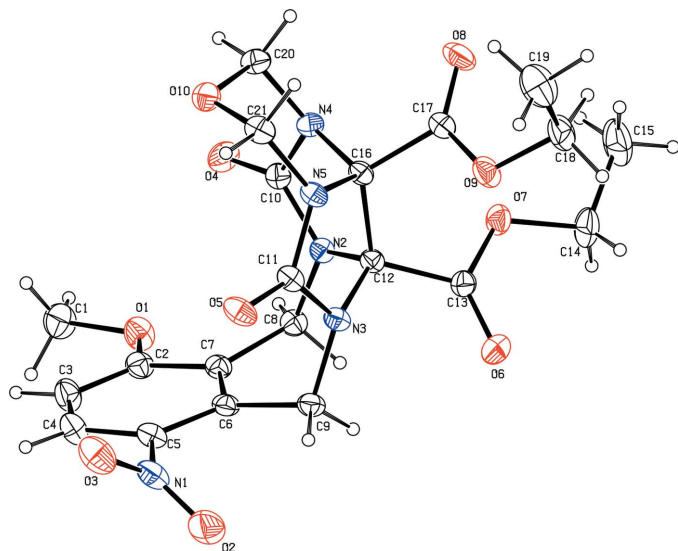
Derivatives of glycoluril have been employed in many applications, including polymer cross-linking, explosives, slow-release fertilizers, cross-linkers, assessment of drug reactivity (Jeon *et al.*, 2005), stabilization of organic compounds against photo-degradation and combinatorial chemistry (Wu, Fettinger & Isaacs, 2002; Wu, Chakraborty *et al.*, 2002). The use of glycoluril derivatives as building blocks for self-assembly, molecular recognition (Kang & Kim, 2005) and catalysis in chloroform has been reported (Conn & Rebek, 1997). Many methylene-bridged glycoluril dimers have been synthesized from soluble organic diethoxycarbonyl glycoluril derivatives (Witt *et al.*, 2000). In this paper, we present the X-ray crystal structure of the title compound (I).



The X-ray crystallographic structure of (I) is shown in Fig. 1. Selected bond distances and angles are listed in Table 1. The molecules are connected by intermolecular hydrogen bonding (Fig. 2, Table 2). In the crystal structure, the dihedral angle between the two five-membered rings of the glycoluril is  $70.00(10)^\circ$ .

#### Experimental

The title compound was synthesized according to the literature procedure of Wu, Chakraborty *et al.* (2002), using paraformaldehyde (0.18 g, 6 mmol), 2,3-bisbromomethyl-1-methoxy-4-nitrobenzene (4 mmol, 1.36 g) and glycoluril (5.72 g, 20 mmol) as starting materials. Crystals suitable for data collection were obtained by slow evaporation of a methanol–1,2-dichloroethane (1:4) solution at 293 K.



**Figure 1**

A view of the molecule of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

**Crystal data**

$C_{21}H_{23}N_5O_{10}$   
 $M_r = 505.44$   
 Triclinic,  $P\bar{1}$   
 $a = 9.756(2) \text{ \AA}$   
 $b = 10.608(2) \text{ \AA}$   
 $c = 11.716(3) \text{ \AA}$   
 $\alpha = 110.123(4)^\circ$   
 $\beta = 98.754(4)^\circ$   
 $\gamma = 92.119(4)^\circ$   
 $V = 1119.9(4) \text{ \AA}^3$

$Z = 2$   
 $D_x = 1.499 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 4159 reflections  
 $\theta = 2.6\text{--}28.3^\circ$   
 $\mu = 0.12 \text{ mm}^{-1}$   
 $T = 292(2) \text{ K}$   
 Block, yellow  
 $0.30 \times 0.20 \times 0.20 \text{ mm}$

**Data collection**

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 8700 measured reflections  
 4340 independent reflections

3412 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.019$   
 $\theta_{max} = 26.0^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -13 \rightarrow 13$   
 $l = -14 \rightarrow 14$

**Refinement**

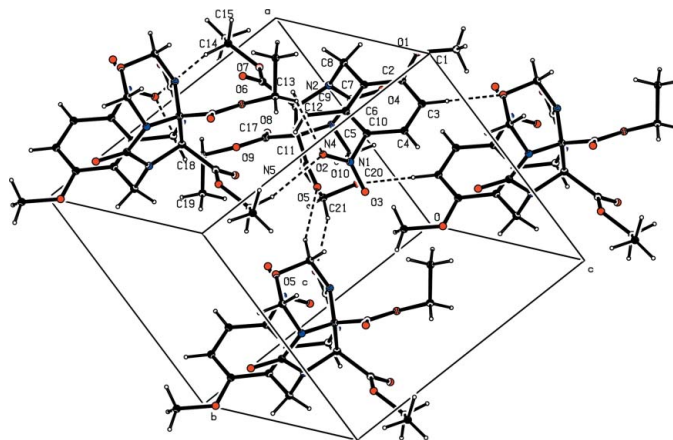
Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.135$   
 $S = 1.06$   
 4340 reflections  
 328 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 0.1853P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.26 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.17 \text{ e \AA}^{-3}$

**Table 1**

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

C2—C3	1.383 (3)	C10—N4	1.390 (2)
C2—C7	1.404 (3)	C12—N2	1.453 (2)
C7—C8	1.517 (3)	C12—C16	1.571 (2)
C8—N2	1.463 (2)	C16—N4	1.444 (2)
C10—O4	1.205 (2)	C20—N4	1.452 (2)
C10—N2	1.369 (2)	C21—O10	1.423 (2)
C2—C7—C8	119.94 (17)	N2—C12—C16	102.39 (13)
N2—C8—C7	112.31 (14)	O10—C20—N4	110.87 (15)
N2—C10—N4	108.27 (14)		



**Figure 2**

The intermolecular hydrogen bonding in the crystal structure of (I). Hydrogen-bonding interactions are indicated by dashed lines.

**Table 2**

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 $\cdots$ O10 <sup>i</sup>	0.93	2.50	3.342 (3)	150
C21—H21A $\cdots$ O5 <sup>ii</sup>	0.97	2.31	3.233 (2)	158
C18—H18A $\cdots$ O2 <sup>iii</sup>	0.97	2.56	3.343 (3)	138
C14—H14B $\cdots$ O2 <sup>iii</sup>	0.97	2.57	3.519 (3)	167

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

All H atoms were initially located in a difference Fourier map. Methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96  $\text{\AA}$  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  $\text{\AA}$ , and were constrained to ride on their parent atoms, with  $U_{iso}(H) = 1.2U_{eq}(C)$ .

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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