

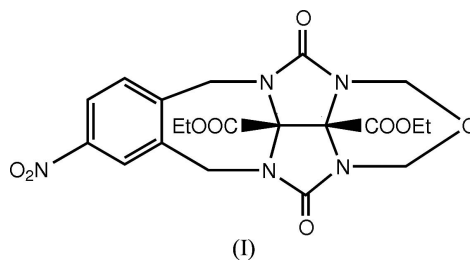
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## Key indicators

Single-crystal X-ray study  
 $T = 292$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.065  
 $wR$  factor = 0.172  
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***rel*-(11*bR*,11*cS*)-Diethyl 5,10-dihydro-7-nitro-4,11-dioxo-1*H*,3*H*,4*H*,11*H*-2-oxa-3*a*,4*a*,10*a*,11*a*-tetraazabenz[*f*]indeno[2,1,7-*ija*]azulene-11*b*,11*c*-dicarboxylate**In the title compound,  $\text{C}_{20}\text{H}_{21}\text{N}_5\text{O}_9$ , the dihedral angle between the two five-membered rings of the glycoluril system is  $68.79(14)^\circ$  and the crystal structure is stabilized by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions.Received 31 August 2005  
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## Comment

The design and synthesis of host molecules for binding of neutral guest molecules continues to be an area of interest in supramolecular chemistry. In recent years, many molecular clips based on glycoluril and its derivatives have been studied, which can bind dihydroxy-substituted aromatic guests by means of hydrogen bonds as well as  $\pi$ - $\pi$  interactions (Sijbesma & Nottle, 1991; Reek *et al.*, 1997; Rowan *et al.*, 1999). In this paper, we report the crystal structure of the title compound (I) (Fig. 1), a new half molecular clip based on diethoxycarbonyl glycoluril. Selected bond lengths and angles are listed in Table 1. The dihedral angle between the two five-membered rings of the glycoluril system is  $68.79(14)^\circ$  and the crystal packing is mainly governed by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions (Table 2 and Fig. 2).

## Experimental

1,2-Bis(chloromethyl)-4-nitrobenzene (1.1 g, 5 mmol), diethoxycarbonyl glycoluril (1.43 g, 5 mmol), formaldehyde (2.4 g, 40 mmol) and trifluoroacetic acid (30 ml) were used as starting materials to synthesize the title compound according to the literature (Wu *et al.*, 2002). Crystals suitable for X-ray diffraction were grown by slow evaporation of MeCN–MeOH(1:1) solutions at ambient conditions.

## Crystal data

 $\text{C}_{20}\text{H}_{21}\text{N}_5\text{O}_9$   
 $M_r = 475.42$   
Monoclinic,  $P2_1/c$   
 $a = 10.0454(18)$  Å  
 $b = 25.564(5)$  Å  
 $c = 8.4997(15)$  Å  
 $\beta = 99.427(3)^\circ$   
 $V = 2153.2(7)$  Å<sup>3</sup>  
 $Z = 4$  $D_x = 1.467$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 3635 reflections  
 $\theta = 2.2$ – $25.1^\circ$   
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 292(2)$  K  
Block, colorless  
 $0.30 \times 0.20 \times 0.10$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer	4225 independent reflections
$\varphi$ and $\omega$ scans	3284 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$R_{\text{int}} = 0.053$
$T_{\text{min}} = 0.966$ , $T_{\text{max}} = 0.988$	$\theta_{\text{max}} = 26.0^\circ$
11538 measured reflections	$h = -12 \rightarrow 11$
	$k = -31 \rightarrow 31$
	$l = -10 \rightarrow 6$

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 1.0059P]$
$R[F^2 > 2\sigma(F^2)] = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.172$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.48 \text{ e } \text{\AA}^{-3}$
4225 reflections	$\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$
309 parameters	
H-atom parameters constrained	

Table 1

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

C1–N1	1.462 (5)	C11–N2	1.441 (3)
C9–O3	1.205 (3)	C11–N3	1.444 (3)
C9–N2	1.374 (3)	C11–C15	1.566 (3)
C9–N4	1.384 (3)	C15–N4	1.443 (3)
C10–O4	1.211 (3)	C15–N5	1.458 (3)
C10–N3	1.372 (3)	C19–O9	1.415 (3)
C10–N5	1.385 (3)	C20–O9	1.417 (3)
N2–C7–C6	111.93 (19)	N2–C11–N3	114.3 (2)
N3–C8–C5	113.6 (2)	N4–C15–N5	111.5 (2)
N2–C9–N4	107.5 (2)	C19–O9–C20	110.9 (2)
N3–C10–N5	108.2 (2)		
C6–C7–N2–C9	−74.5 (3)	C5–C8–N3–C10	81.6 (3)
C6–C7–N2–C11	80.2 (3)	C5–C8–N3–C11	−73.9 (3)

Table 2

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

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
C20–H20A $\cdots$ O3 <sup>i</sup>	0.97	2.54	3.262 (4)	132
C19–H19B $\cdots$ O4 <sup>i</sup>	0.97	2.54	3.444 (4)	156
C17–H17A $\cdots$ O7 <sup>ii</sup>	0.97	2.52	3.293 (5)	136
C7–H7B $\cdots$ O3 <sup>iii</sup>	0.97	2.41	3.288 (3)	150

Symmetry codes: (i)  $-x + 1, -y + 1, -z + 2$ ; (ii)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (iii)  $-x, -y + 1, -z + 2$ .

After their location in a different Fourier map, H atoms were placed in calculated positions and allowed to ride on their parent atoms, with  $C\text{---}H = 0.93\text{--}0.97 \text{ \AA}$ , and  $U_{\text{iso}} = 1.2\text{--}1.5 U_{\text{eq}}(C)$ .

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); 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, 2001); software used to prepare material for publication: SHELXTL.

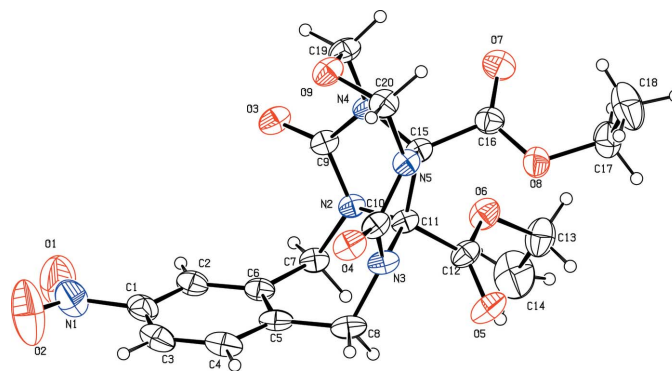


Figure 1

View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

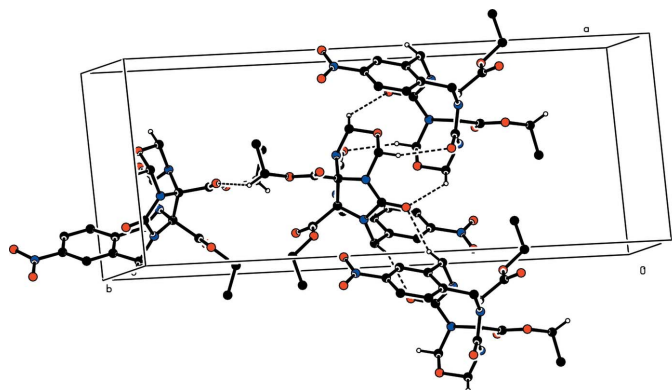


Figure 2

C–H $\cdots$ O intermolecular hydrogen-bonding (dashed lines).

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