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Diethyl 6,9-dibromo-1,4-dioxo-1,2,3,4,5,10-hexahydro-2,3,4a,10a-tetraazabenzo[*g*]cyclopenta[*cd*]azulene-2a,10b-dicarboxylate

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Key indicators

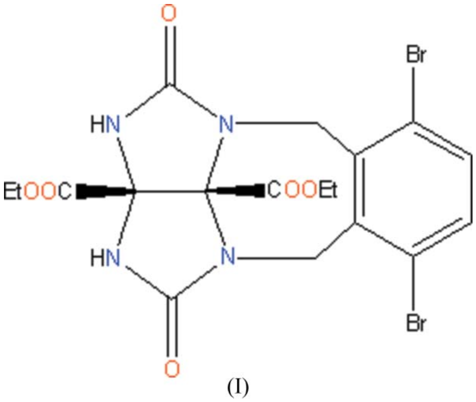
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.009 Å
R factor = 0.058
wR factor = 0.157
Data-to-parameter ratio = 13.1

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

The title compound, $C_{18}H_{18}Br_2N_4O_6$, is an important intermediate for the synthesis of molecular clips. Intermolecular N—H···O hydrogen bonds form eight-membered rings. No π–π stacking is observed.

Comment

The derivatives of glycoluril are well known in studies of organic supramolecular structures (Isaacs & Fettinger, 1999; Burnett *et al.*, 2003; Chakraborty *et al.*, 2002). In addition, glycoluril derivatives have significant potential in crystal engineering studies (Wu *et al.*, 2002; Johnson *et al.*, 2002). In this paper, we present the X-ray crystallographic analysis of the title compound, (I), which is an important intermediate for the preparation of S- and C-shaped methylene-bridged glycoluril dimers (Wu *et al.*, 2002).



(I)

The molecular structure of (I) is shown in Fig. 1. Selected bond lengths and angles are given in Table 1. The molecules

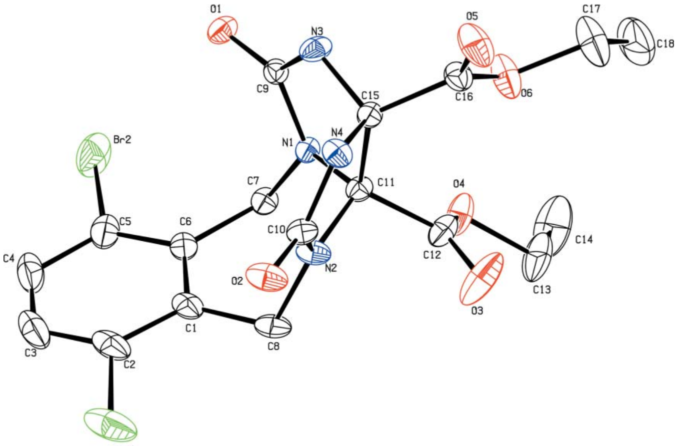


Figure 1
The molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

are linked by intermolecular N—H···O hydrogen bonds (Table 2 and Fig. 2), utilizing a carbonyl O atom as acceptor. As a result, an eight-membered ring is formed, the topological motif of which corresponds to the first level graph-set descriptor $R_2^2(8)$ (Bernstein *et al.*, 1995). No π–π stacking is observed.

Experimental

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

Crystal data

$C_{18}H_{18}Br_2N_4O_6$
$M_r = 546.18$
Monoclinic, $P2_1/c$
$a = 12.0665$ (11) Å
$b = 10.3050$ (10) Å
$c = 17.4969$ (17) Å
$\beta = 108.371$ (2)°
$V = 2064.8$ (3) Å^3
$Z = 4$
$D_x = 1.757$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6947 reflections
$\theta = 2.33$–23.98°
$\mu = 3.97$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.238$, $T_{\rm max} = 0.304$
14418 measured reflections
3656 independent reflections
2925 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 25.1$°
$h = -14 \rightarrow 14$
$k = -12 \rightarrow 12$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.157$
$S = 1.03$
3656 reflections
279 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 5.0818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 1.02$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.09$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.906 (7)	C13—C14	1.488 (8)
C1—C2	1.398 (7)	C17—C18	1.470 (8)
C1—C6	1.403 (8)		
N2—C11—C12	107.8 (4)	C16—C15—C11	119.9 (4)
N3—C15—C11	101.4 (3)		
C8—C1—C6—C7	−1.1 (6)	C12—C11—N2—C8	−69.9 (5)
C6—C7—N1—C9	−76.0 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O1$^{\rm i}$	0.86 (4)	2.13 (5)	2.965 (5)	166 (7)
N3—H3*A*···O2$^{\rm ii}$	0.81 (4)	1.95 (5)	2.750 (5)	170 (8)

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

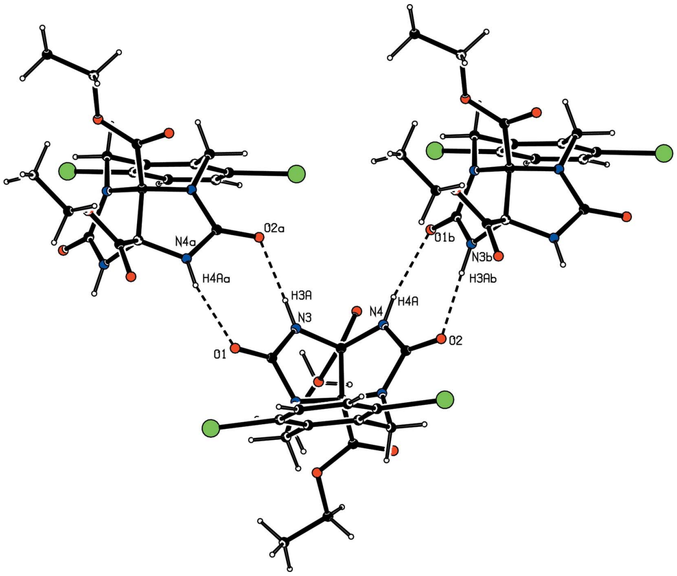


Figure 2
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

The N-bound H atoms were located in a difference map and refined freely [N—H = 0.81 (4)–0.86 (4) Å]; their displacement parameters were allowed for with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. The methyl H atoms were positioned geometrically (C—H = 0.96 Å). The constraint $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ was applied, but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions [0.93 (for CH) or 0.97 Å (for CH$_2$)], and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The highest peak and deepest hole are located 0.87 and 0.85 Å, respectively, from atom Br2.

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*.

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1997). *SMART*. Version 5.054. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Version 6.01. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, C. A., Lagona, J., Wu, A., Shaw, J. A., Coady, D., Fettinger, J. C., Day, A. I. & Isaacs, L. (2003). *Tetrahedron*, **59**, 1961–1970.
Chakraborty, A., Wu, A., Witt, D., Lagona, J., Fettinger, J. C. & Isaacs, L. (2002). *J. Am. Chem. Soc.* **124**, 8297–8306.
Isaacs, L. & Fettinger, J. C. (1999). *Chem. Commun.* pp. 2549–2550.
Johnson, D. W., Palmer, L. C., Hof, F., Iovine, P. M. & Rebek, J. (2002). *Chem. Commun.* pp. 2228–2229.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wu, A., Chakraborty, A., Witt, D., Lagona, J., Damkaci, F., Ofori, M. A., Chiles, J. K., Fettinger, J. C. & Isaacs, L. (2002). *J. Org. Chem.* **67**, 5817–5830.

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