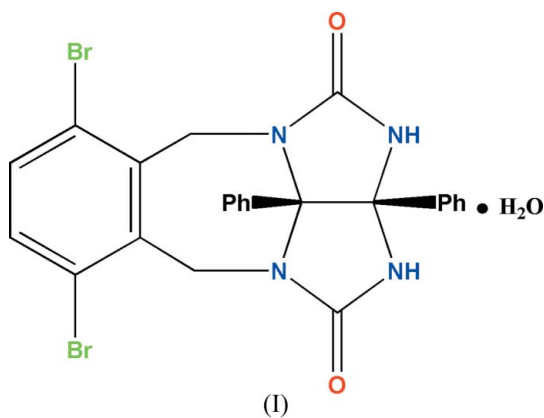


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## Key indicators

Single-crystal X-ray study  
 $T = 292$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.011$  Å  
 $R$  factor = 0.053  
 $wR$  factor = 0.136  
Data-to-parameter ratio = 16.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.6,9-Dibromo-2a,10b-diphenyl-2a,5,10,10b-  
tetrahydro-2H,3H-2,3,4a,10a-tetraaza-  
benzo[g]cyclopenta[cd]azulene-1,4-dione  
monohydrateThe title compound,  $\text{C}_{24}\text{H}_{18}\text{Br}_2\text{N}_4\text{O}_2 \cdot \text{H}_2\text{O}$ , forms a supra-  
molecular structure *via*  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{C}-\text{H} \cdots \text{O}$   
hydrogen bonds. In the crystal structure, the water molecule  
serves as a bifurcated hydrogen-bond acceptor and as a  
hydrogen-bond donor.Received 27 March 2006  
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## Comment

Glycoluril and its derivatives have been studied extensively  
for a variety of purposes. In particular, they have been  
investigated as building blocks for the construction of rigid  
host molecules (Rowan *et al.*, 1999; Wu *et al.*, 2002). The title  
compound, (I), is an important intermediate in the prepara-  
tion of methylene-bridged glycoluril dimers (Wu *et al.*, 2002)  
and its structure is reported here (Fig. 1 and Table 1). The  
crystal structure of an analog, 6,9-dibromo-1,4-dioxo-  
1,2,3,4,5,10-hexahydro-2,3,4a,10a-tetraaza-benzo[g]cyclo-  
penta[cd]azulene-2a,10b-dicarboxylic acid diethyl ester, was  
reported previously (She *et al.*, 2005).In the crystal structure, molecules are interlinked *via*  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds (Fig. 2 and Table 2). Each water molecule serves as a bifurcated hydrogen-bond acceptor and as a hydrogen-bond donor. No  $\pi-\pi$  stacking can be observed in the crystal structure.

## Experimental

The title compound was synthesized according to the procedure reported by Wu *et al.* (2002). Crystals appropriate for data collection were obtained by slow evaporation of a methanol–chloroform (1:30  $v/v$ ) solution at 283 K.

## Crystal data

 $C_{24}H_{18}Br_2N_4O_2 \cdot H_2O$  $M_r = 572.26$ Monoclinic,  $P2_1/c$  $a = 13.066$  (2) Å $b = 9.9260$  (15) Å $c = 18.351$  (3) Å $\beta = 106.539$  (3)° $V = 2281.5$  (6) Å<sup>3</sup> $Z = 4$  $D_x = 1.666$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation $\mu = 3.59$  mm<sup>-1</sup> $T = 292$  (2) K

Prism, colorless

0.30 × 0.20 × 0.20 mm

## Data collection

Bruker SMART 4K CCD area-detector diffractometer

 $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Sheldrick 1997)

 $T_{\min} = 0.412$ ,  $T_{\max} = 0.534$ 

(expected range = 0.377–0.488)

14118 measured reflections

5151 independent reflections

3218 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.043$  $\theta_{\text{max}} = 27.5^\circ$ 

## Refinement

Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.053$  $wR(F^2) = 0.136$  $S = 1.05$ 

5151 reflections

306 parameters

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0625P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} < 0.001$  $\Delta\rho_{\text{max}} = 0.68$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.56$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

C1–C8	1.521 (8)	C10–O2	1.236 (7)
C6–C7	1.520 (9)	C10–N4	1.333 (7)
C9–N3	1.367 (8)	C18–C19	1.509 (8)
N1–C7–C6	114.4 (5)	N3–C18–N4	111.9 (5)
O2–C10–N4	127.2 (5)	C19–C18–C11	117.8 (4)
Br2–C5–C6–C7	−3.0 (8)	O1–C9–N3–C18	168.2 (6)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C15–H15 <sup>i</sup> ⋯O1 <sup>i</sup>	0.93	2.58	3.432 (9)	152
N4–H4A⋯O2 <sup>ii</sup>	0.86	2.08	2.901 (6)	158
N3–H3C⋯O3 <sup>iii</sup>	0.86	2.46	2.950 (7)	117
O3–H3B⋯O1 <sup>iii</sup>	0.88 (5)	1.91 (5)	2.786 (6)	171 (5)

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

H atoms of the water molecule were located in a difference Fourier map and freely refined with fixed isotropic displacement parameters. All other H atoms were positioned geometrically and treated as riding, with  $N-H = 0.86$  Å and  $C-H = 0.93$  or  $0.97$  Å.  $U_{\text{iso}}(\text{H})$  values were set equal to  $xU_{\text{eq}}(\text{carrier atom})$ , where  $x = 1.2$  for  $\text{CH}_2$  and  $\text{CH}$  or  $x = 1.5$  for  $\text{N}$ .

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.

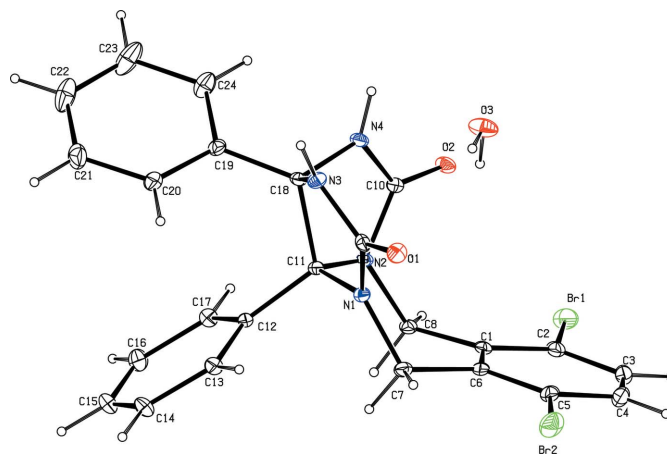


Figure 1

The asymmetric unit of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

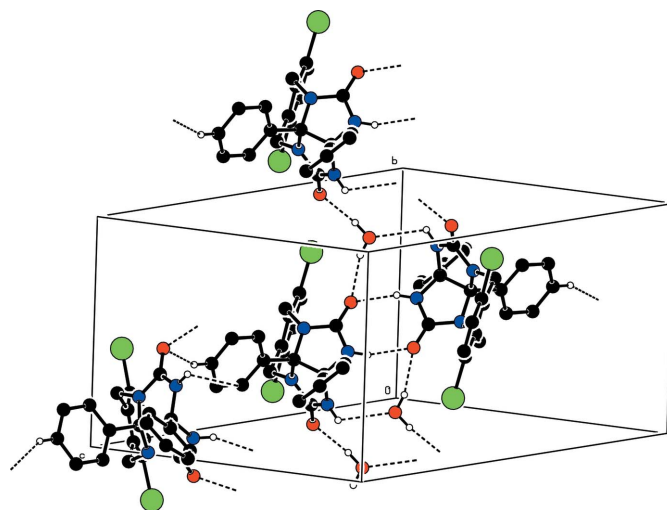


Figure 2

Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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