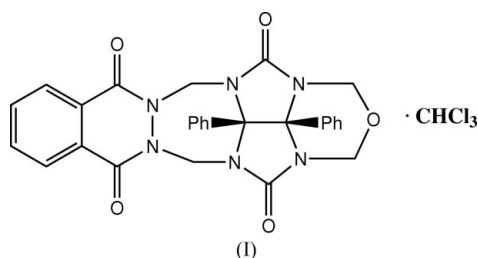


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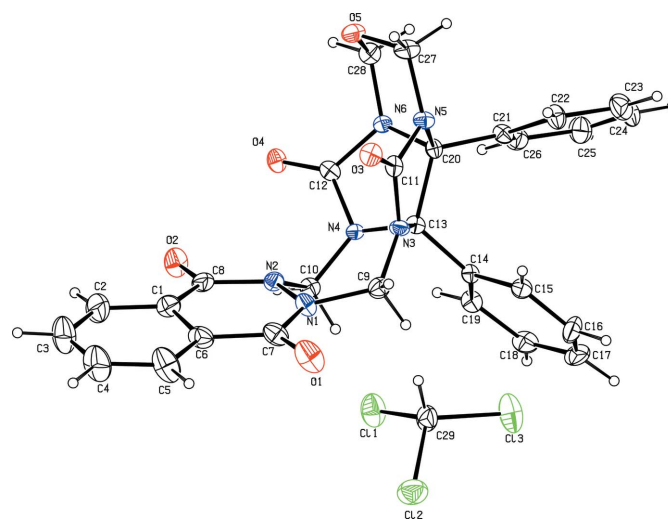
Key indicators

Single-crystal X-ray study
 $T = 292\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.013\text{ \AA}$
 R factor = 0.071
 wR factor = 0.203
Data-to-parameter ratio = 12.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(13*bR*,13*cS*)-*rel*-11*b*,11*c*-Diphenyl-6,11-dihydro-
1*H*,3*H*,4*H*,13*H*-2-oxa-3*a*,4*a*,5*a*,11*a*,12*a*,13*a*-hexa-
azabenz[*f*]indeno[2,1,8-*ija*]naphth[2,3-*f*]azulene-
4,6,11,13-tetrone chloroform solvate**The crystal structure of the title compound, $\text{C}_{28}\text{H}_{22}\text{N}_6\text{O}_5 \cdot \text{CHCl}_3$, an important intermediate for molecular tweezers, shows intermolecular hydrogen bonding.Received 3 May 2006
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Comment

Glycoluril derivatives have found widespread use as building blocks in supramolecular chemistry, including the preparation of molecular clips (Rowan *et al.*, 1999), molecular capsules (Hof *et al.*, 2002), the cucurbit[*n*]uril family (Lagona *et al.*, 2003) and anion-binding receptors (Kang *et al.*, 2004). Many methylene-bridged glycoluril dimers have been synthesized from dimerization reactions of glycoluril-derived cyclic ethers (Wu *et al.*, 2002). In this paper, we report the crystal structure of the title compound, (I).

The X-ray crystallographic structure of (I) is shown in Fig. 1. In the crystal structure, the interplanar angle between the two five-membered rings (C12/N4/C13/C20/N6 and C11/N3/C13/

**Figure 1**
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as circles of arbitrary radii.

C20/N5) is 73.1 (4)°, while the angle between the planes of the two phenyl rings (C14–C19 and C21–C26) is 43.9 (4)°. The aromatic ring C1–C6 is effectively coplanar with the six-membered C1/C6/C7/N1/N2/C8 ring, as seen in the dihedral angle between their least-squares planes of 3.1 (4)°. The molecules are connected by intermolecular hydrogen bonding (Fig. 2 and Table 1).

Experimental

The title compound was synthesized according to the literature procedure of Lagona *et al.* (2003). Crystals suitable for data collection were obtained by slow evaporation of a methanol–chloroform solution (1:25 *v/v*).

Crystal data

$C_{29}H_{23}Cl_3N_6O_5$	$V = 1418.4 (4) \text{ \AA}^3$
$M_r = 641.88$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.503 \text{ Mg m}^{-3}$
$a = 8.2200 (14) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 13.441 (2) \text{ \AA}$	$\mu = 0.38 \text{ mm}^{-1}$
$c = 13.883 (2) \text{ \AA}$	$T = 292 (2) \text{ K}$
$\alpha = 69.674 (3)^\circ$	Block, colourless
$\beta = 83.711 (3)^\circ$	$0.20 \times 0.10 \times 0.06 \text{ mm}$
$\gamma = 81.077 (4)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	4921 independent reflections
φ and ω scans	1549 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.106$
7437 measured reflections	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2]$
$wR(F^2) = 0.203$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.80$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4921 reflections	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
388 parameters	$\Delta\rho_{\text{min}} = -0.37 \text{ e \AA}^{-3}$

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C29–H29 \cdots O4 ⁱ	0.98	2.51	3.209 (9)	129
C29–H29 \cdots O2 ⁱ	0.98	2.44	3.256 (10)	140
C19–H19 \cdots O2 ⁱⁱ	0.93	2.56	3.337 (9)	141
C10–H10A \cdots O4 ⁱⁱ	0.97	2.57	3.072 (8)	113
C22–H22 \cdots O1 ⁱⁱⁱ	0.93	2.56	3.317 (9)	139
C15–H15 \cdots O3 ⁱⁱⁱ	0.93	2.56	3.484 (10)	172

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

H atoms were constrained to an ideal geometry and to ride on their parent atoms as follows: methylene C–H = 0.97 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$; methine C–H = 0.98 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$; aromatic C–H = 0.93 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

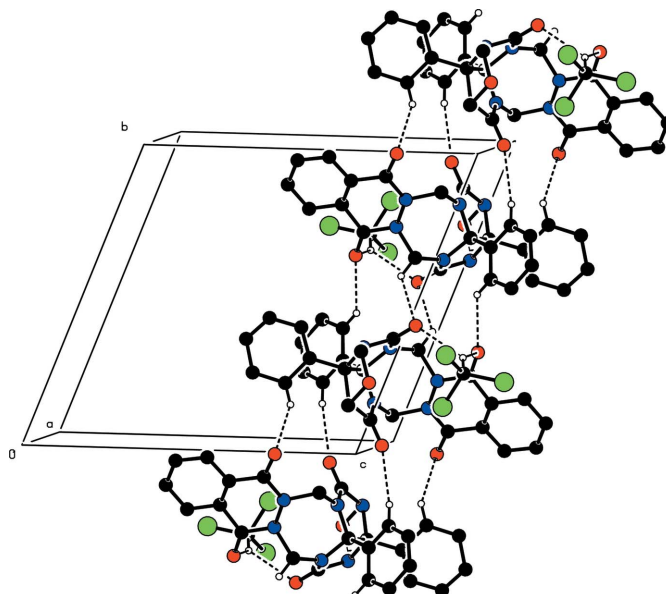


Figure 2

The molecular packing of (I), viewed approximately along the *b* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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