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Diethyl 4-iodo-1,22-dioxo-15-(4-pyridylethynyl)-1,20,12,21-tetraazahexacyclo[10.10.2.0^{3,8}.0^{10,23}.0^{14,20}.0^{21,24}]tetracosa-3,5,7,14,16,18-hexaene-23,24-dicarboxylate: a molecular clip based on bis(ethoxycarbonyl)glycoluril

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Key indicators

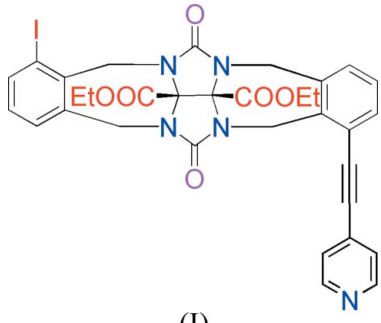
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.011 Å
Disorder in main residue
R factor = 0.094
wR factor = 0.278
Data-to-parameter ratio = 12.0

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

The title compound, $C_{33}H_{28}IN_5O_6$, is a molecular clip based on the glycoluril framework. A 4-pyridinylethynyl-substituted benzene ring is fused to one seven-membered ring, which binds two of the N atoms from separate rings of the glycoluril system. The second, similar, seven-membered ring is fused to an iodobenzene ring. The orientation of these fused-benzene-ring substituents is *trans* with respect to the glycouril framework. The crystal structure is stabilized by weak C—H···O and C—H···N hydrogen bonds and C—H···π interactions.

Comment

A molecular clip is a molecule with a rigid U-shaped cavity, in which small guest molecules can be complexed by intermolecular interactions (Reek *et al.*, 1997). The glycoluril skeleton has served as an important building block for research into molecular clips (Rowan *et al.*, 1999; Chakraborty *et al.*, 2002). The introduction of different functional groups into the clip has been of recent interest (Chen *et al.*, 2006) and we report here the structure of a glycouril derivative, (I) (Fig. 1), with 4-pyridinylethynyl- and iodo-substituted benzene rings fused to the side-wall of the molecular clip.



(I)

Weak C—H···O and C—H···N hydrogen bonds stabilize the structure, forming a two-dimensional network (Table 1 and Fig. 2). In addition, inversion-related C10—H10···π interactions to the C1–C5/N1^{i} rings [H10···ring centroid distance = 2.94 Å; symmetry code: (i) $1-x, 1-y, 1-z$] form a three-dimensional network.

Experimental

The title compound was synthesized according to a literature procedure (Wu *et al.*, 2002) in 10% isolated yield. Crystals of (I) appropriate for data collection were obtained by slow evaporation of a dichloromethane–methanol (1:4 *v*/*v*) solution at 283 K.

Crystal data

$C_{33}H_{28}IN_5O_6$
M_r = 717.50
Monoclinic, $C2/c$
a = 38.825 (3) Å
b = 8.5516 (6) Å
c = 18.8448 (14) Å
β = 108.7730 (10)°
V = 5923.9 (7) Å^3
Z = 8
D_x = 1.609 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.14 mm^{-1}
T = 298 (2) K
Block, colorless
0.08 × 0.06 × 0.02 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.915, T_{max} = 0.978
19410 measured reflections
5005 independent reflections
2882 reflections with $I > 2\sigma(I)$
R_{int} = 0.076
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.094
$wR(F^2)$ = 0.278
S = 1.01
5005 reflections
418 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1787P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.97 e Å^{-3}
$\Delta\rho_{min}$ = −0.81 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···N1^{i}	0.97	2.55	3.480 (12)	160
C25—H25B···O1^{ii}	0.96	2.49	3.403 (8)	159
C32—H32···O5^{iii}	0.93	2.55	3.153 (10)	123

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

The crystals were small and weakly diffracting, which explains the low fraction of significant data collected and the poor residuals. Repeated attempts to grow larger crystals have been unsuccessful. All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). All other H atoms were placed in geometrically idealized positions, with C—H = 0.93–0.97 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). Atoms C21 and C25 are disordered over two sites; the site-occupancy factors for the two orientations refined to 0.62 (3) and 0.73 (3), respectively, for the major components.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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References

Bruker (2001). *SMART* (Version 5.628), *SAINT* (Version 6.45) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.

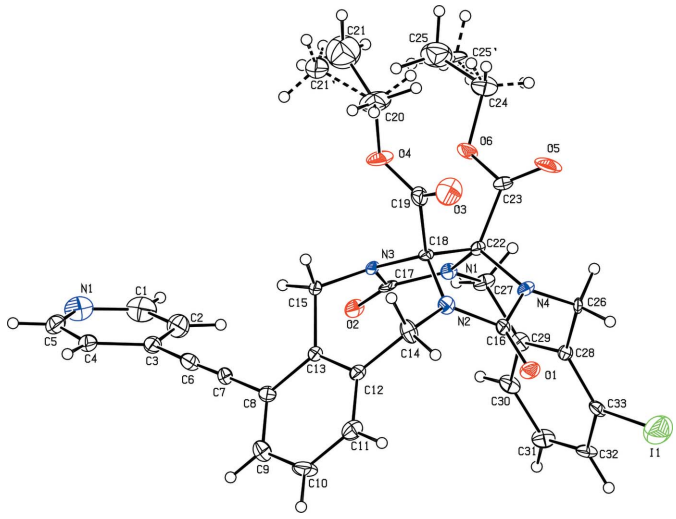

Figure 1
The molecular structure of (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level. Bonds to atoms of the minor disorder components are drawn as dashed lines.

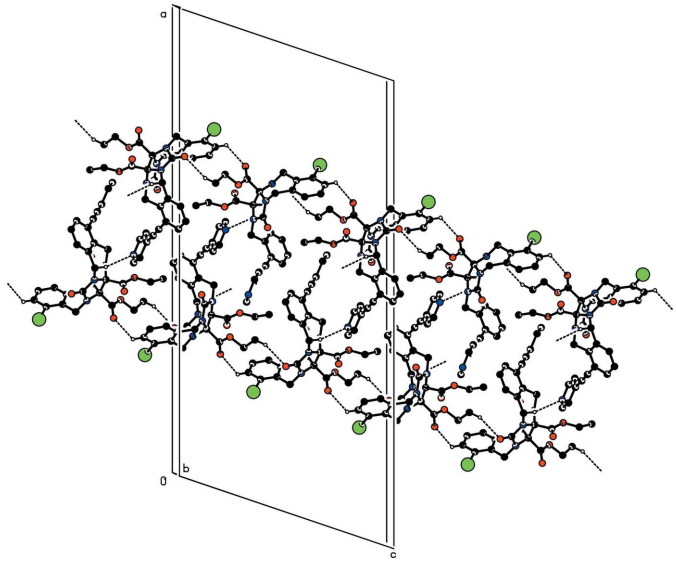

Figure 2
The packing of (I), with hydrogen bonds shown as dashed lines. For clarity, H atoms not involved in hydrogen bonding and the atoms of the minor disorder components have been omitted.

Chakraborty, A., Wu, A., Witt, D., Lagona, J., Fettinger, J. C. & Isaacs, L. (2002). *J. Am. Chem. Soc.* **124**, 8297–8306.
Chen, Y.-F., Xu, M. & Wu, A.-X. (2006). *Acta Cryst.* E**62**, o2254–o2255.
Reek, J. N. H., Priem, A. H., Engelkamp, H., Rowan, A. E., Elemans, J. A. A. W. & Nolte, R. J. M. (1997). *J. Am. Chem. Soc.* **119**, 9956–9964.
Rowan, A. E., Elemans, J. A. A. W. & Nolte, R. J. M. (1999). *Acc. Chem. Res.* **32**, 995–1006.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
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.