Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-(Biphenyl-4-yl)-8-hydroxypentacyclo-[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-11-one ethylene ketal

Grant A. Boyle,[a] Thavendran Govender,[b] Hendrik G. Kruger[a]* and Glenn E. M. Maguire[a]

[a]School of Chemistry, University of KwaZulu-Natal, Durban 4000, South Africa, and [b]School of Pharmacy and Pharmacology, University of KwaZulu-Natal, Durban 4000, South Africa
Correspondence e-mail: kruger@ukzn.ac.za

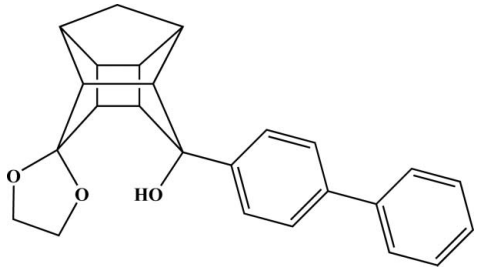
Received 19 November 2007; accepted 6 December 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.109; data-to-parameter ratio = 15.2.

The title compound, $C_{25}H_{24}O_3$, synthesized as a potential chiral catalyst, exhibits a range of C—C bond lengths in the pentacycloundecane cage between 1.5144 (18) and 1.5856 (16) Å. The two benzene rings are not planar with respect to each other, but rather are twisted at a torsion angle of 34.67 (17)°. The molecule has an intramolecular O—H···O interaction and participates in two C—H···O intermolecular interactions to form a one-dimensional chain.

Related literature

For related literature, see: Flippen-Anderson *et al.* (1991); Linden *et al.* (2005); Kruger *et al.* (2005, 2006); Boyle *et al.* (2007).



Experimental

Crystal data

$C_{25}H_{24}O_3$
M_r = 372.44
Monoclinic, $P2_1/c$
a = 10.2527 (2) Å
b = 16.9832 (3) Å
c = 10.3650 (2) Å
β = 90.5760 (10)°
V = 1804.70 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.47 × 0.45 × 0.37 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
27563 measured reflections
3904 independent reflections
3358 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 1.04
3904 reflections
257 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*H*···O2	0.877 (18)	1.769 (19)	2.6153 (12)	161.6 (18)
C12—H12*B*···O1^{i}	0.99	2.54	3.2455 (18)	128
C24—H24···O3^{ii}	0.95	2.60	3.4955 (16)	158

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *WinGX* (Farrugia, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank Dr Manuel Fernandes of the Jan Boeyens Structural Chemistry Laboratory at the University of the Witwatersrand for his assistance with the crystallographic data collection. This work was supported by grants from the National Research Foundation (South Africa) (grant No. GUN 2046819), Aspen Pharmacare and the University of KwaZulu-Natal.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2055).

References

Boyle, G. A., Govender, T., Karpoormath, R. & Kruger, H. G. (2007). *Acta Cryst.* E**63**, o3977.
Bruker (1999). *SAINT-Plus* (Version 6.02) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Version 2.0-1. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flippen-Anderson, J. L., George, C., Gilardi, R., Zajac, W. W., Walters, T. R., Marchand, A., Dave, P. R. & Arney, B. E. (1991). *Acta Cryst.* C**47**, 813–817.
Kruger, H. G., Rademeyer, M., Govender, T. & Gokul, V. (2006). *Acta Cryst.* E**62**, o42–o44.
Kruger, H. G., Rademeyer, M. & Ramdhani, R. (2005). *Acta Cryst.* E**61**, o3968–o3970.
Linden, A., Romański, J., Mlostoń, G. & Heimgartner, H. (2005). *Acta Cryst.* C**61**, o221–o226.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 doi:10.1107/S1600536807065920 © 2008 International Union of Crystallography

supplementary materials

Acta Cryst. (2008). E**64**, o283 [**doi:10.1107/S1600536807065920**]

8-(Biphenyl-4-yl)-8-hydroxypentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-11-one ethylene ketal

G. A. Boyle, T. Govender, H. G. Kruger and G. E. M. Maguire

Comment

The molecule was synthesized as part of an ongoing study into the synthesis of chiral cage ligands for applications in asymmetric catalysis. The title molecule, which exists as a racemic mixture, has the potential to be a very unique ligand once it is resolved into an enantiopure compound.

A number of publications have focused on the molecular geometries of PCU cage derivatives as well as the bond lengths which deviate from the normal value of 1.54 Å (Flippen-Anderson *et al.*, 1991; Linden *et al.*,2005; Kruger *et al.*, 2005, 2006, Boyle *et al.*, 2007). Certain bonds in the cage skeleton are longer (*e.g.* C9—C10, 1.5922 Å) while others are significantly shorter (*e.g.* C1—C11, 1.5106 Å). The molecule **(I)** consists of a large hydrophobic hydrocarbon skeleton as well as a hydrophilic ketal group and hydroxyl moiety. The two aromatic rings attached to C8 are not planar with respect to each other, but rather twisted at a torsion angle of 34.67 (17)° as expected due to steric factors. Fig. 1 shows the molecular structure and the numbering scheme employed.

The molecule exhibits intramolecular hydrogen bonding (Fig. 2) between the hydroxyl group and the ketal group (O3—H3H···O2). There is no intermolecular hydrogen bonding present in the structure, however a complex network of weak Van der Waals interactions between neighbouring molecules (Fig. 3) results in a layered packing effect with alternating hydrophilic and hydrophobic layers made up of the hydrophobic cage molecules and aromatic moeties, and the hydrophilic hydroxyl and ketal groups, respectively (Fig. 4).

Experimental

A solution of 4-bromobiphenyl in dry THF (3 mol eq) was cooled to −78°C using a dry-ice-acetone bath. Butyllithium solution (15% in hexane, 1.2 mole equivalents relative to bromobiphenyl) was added and the solution stirred for 10 minutes. A solution of pentacyclo-[5.4.0.0^{2,6}.0^{3,10}.0$^{5.,9}$]-undecane-8,11-dione-mono-ethylene ketal (1 mol eq-up to 1 g scale) in dry THF was added and the solution stirred at −78°C for 1 h then at room temperature overnight. The reaction was quenched by adding water dropwise. The solvent was removed *in vacuo*. The product was isolated using column chromatography (EtOAc/Hexane, 10:90). The oily product crystallized on standing at room temperature overnight.

Refinement

Non-hydrogen atoms were first refined isotropically followed by anisotropic refinement by full matrix least-squares calculations based on F^2 using *SHELXTL*. Hydrogen atoms were first located in the difference map then positioned geometrically and allowed to ride on their respective parent atoms.

Figures

Fig. 1. The asymmetric unit showing ellipsoids at the 50% probability level and the numbering scheme employed.

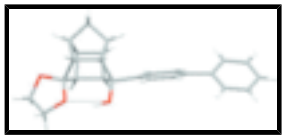


Fig. 2. Diagram of the intermolecular hydrogen bonding.

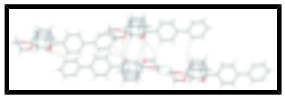


Fig. 3. Diagram of the short intermolecular contacts.

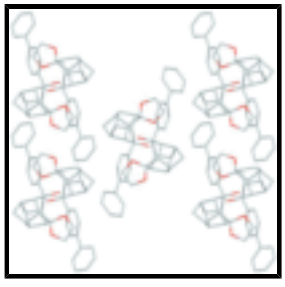


Fig. 4. Depiction of the molecular packing. Hydrogen atoms have been ommited for clarity

8-(Biphenyl-4-yl)-8-hydroxypentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-11-one ethylene ketal

Crystal data

$C_{25}H_{24}O_3$	F_{000} = 792
M_r = 372.44	D_x = 1.371 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6796 reflections
a = 10.2527 (2) Å	θ = 2.3–28.5º
b = 16.9832 (3) Å	μ = 0.09 mm^{-1}
c = 10.3650 (2) Å	T = 173 (2) K
β = 90.5760 (10)º	Block, colourless
V = 1804.70 (6) Å^3	0.47 × 0.45 × 0.37 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	3358 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.037
Monochromator: graphite	θ_{max} = 27.0º
T = 173(2) K	θ_{min} = 2.0º
φ and ω scans	h = −13→13

Absorption correction: none
27563 measured reflections
3904 independent reflections

$k = -21 \rightarrow 21$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.109$
$S = 1.04$
3904 reflections
257 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.5851P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
H3H	0.2134 (18)	−0.0511 (11)	0.4718 (18)	0.045 (5)*
C1	0.27294 (12)	0.04517 (7)	0.25422 (12)	0.0243 (3)
H1	0.3241	0.0168	0.1870	0.029*
C2	0.25040 (13)	0.13435 (8)	0.22877 (13)	0.0286 (3)
H2	0.2840	0.1566	0.1462	0.034*
C3	0.10706 (13)	0.15304 (8)	0.26439 (13)	0.0316 (3)
H3	0.0462	0.1613	0.1896	0.038*
C4	0.12017 (15)	0.22221 (8)	0.35620 (15)	0.0365 (3)
H4B	0.1570	0.2695	0.3144	0.044*
H4A	0.0371	0.2355	0.3989	0.044*
C5	0.21666 (13)	0.18202 (7)	0.44640 (13)	0.0288 (3)
H5	0.2460	0.2142	0.5221	0.035*
C6	0.32659 (12)	0.15486 (7)	0.35544 (12)	0.0267 (3)
H6	0.4050	0.1895	0.3480	0.032*

C7	0.35006 (11)	0.06574 (7)	0.38116 (11)	0.0224 (2)
H7	0.4436	0.0491	0.3825	0.027*
C8	0.27605 (11)	0.05050 (7)	0.50638 (11)	0.0208 (2)
C9	0.15361 (11)	0.10184 (7)	0.48020 (12)	0.0242 (3)
H9	0.0956	0.1053	0.5570	0.029*
C10	0.07520 (12)	0.08160 (8)	0.35180 (12)	0.0268 (3)
H10	−0.0204	0.0747	0.3661	0.032*
C11	0.13428 (12)	0.01565 (7)	0.27329 (12)	0.0252 (3)
C12	0.08855 (16)	−0.06659 (9)	0.10563 (14)	0.0370 (3)
H12A	0.1730	−0.0701	0.0605	0.044*
H12B	0.0177	−0.0832	0.0460	0.044*
C13	0.08947 (14)	−0.11551 (8)	0.22633 (13)	0.0321 (3)
H13A	0.0026	−0.1390	0.2413	0.038*
H13B	0.1547	−0.1583	0.2207	0.038*
C14	0.34966 (11)	0.08247 (7)	0.62415 (11)	0.0215 (2)
C15	0.47065 (12)	0.11841 (8)	0.61578 (12)	0.0274 (3)
H15	0.5085	0.1264	0.5335	0.033*
C16	0.53703 (12)	0.14274 (8)	0.72545 (12)	0.0284 (3)
H16	0.6196	0.1675	0.7168	0.034*
C17	0.48648 (12)	0.13201 (7)	0.84792 (11)	0.0234 (3)
C18	0.36431 (12)	0.09680 (7)	0.85637 (12)	0.0254 (3)
H18	0.3264	0.0891	0.9387	0.031*
C19	0.29713 (12)	0.07281 (7)	0.74651 (12)	0.0245 (3)
H19	0.2136	0.0493	0.7549	0.029*
C20	0.56860 (11)	0.15326 (7)	0.96176 (12)	0.0237 (3)
C21	0.65359 (12)	0.21726 (8)	0.95569 (12)	0.0276 (3)
H21	0.6496	0.2517	0.8835	0.033*
C22	0.74373 (13)	0.23134 (8)	1.05342 (13)	0.0316 (3)
H22	0.8027	0.2742	1.0467	0.038*
C23	0.74797 (13)	0.18301 (8)	1.16083 (13)	0.0319 (3)
H23	0.8111	0.1919	1.2269	0.038*
C24	0.66019 (13)	0.12197 (8)	1.17160 (13)	0.0315 (3)
H24	0.6603	0.0902	1.2470	0.038*
C25	0.57148 (13)	0.10685 (8)	1.07246 (12)	0.0280 (3)
H25	0.5120	0.0643	1.0803	0.034*
O1	0.06660 (10)	0.01105 (6)	0.15323 (9)	0.0329 (2)
O2	0.12307 (9)	−0.06169 (5)	0.32656 (8)	0.0302 (2)
O3	0.25380 (9)	−0.02990 (5)	0.53817 (8)	0.0260 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0280 (6)	0.0256 (6)	0.0193 (6)	0.0020 (5)	0.0011 (5)	0.0009 (5)
C2	0.0352 (7)	0.0264 (6)	0.0242 (6)	0.0020 (5)	0.0004 (5)	0.0051 (5)
C3	0.0322 (7)	0.0296 (7)	0.0328 (7)	0.0073 (5)	−0.0071 (5)	0.0029 (5)
C4	0.0398 (7)	0.0280 (7)	0.0417 (8)	0.0105 (6)	−0.0041 (6)	0.0013 (6)
C5	0.0344 (7)	0.0218 (6)	0.0302 (7)	0.0045 (5)	−0.0013 (5)	−0.0027 (5)
C6	0.0276 (6)	0.0238 (6)	0.0288 (7)	−0.0019 (5)	−0.0007 (5)	0.0048 (5)

C7	0.0221 (5)	0.0246 (6)	0.0205 (6)	0.0005 (4)	0.0006 (4)	0.0010 (4)
C8	0.0219 (5)	0.0207 (5)	0.0198 (6)	−0.0003 (4)	−0.0001 (4)	0.0011 (4)
C9	0.0225 (6)	0.0268 (6)	0.0232 (6)	0.0036 (5)	−0.0002 (5)	−0.0027 (5)
C10	0.0227 (6)	0.0309 (6)	0.0266 (6)	0.0032 (5)	−0.0036 (5)	−0.0017 (5)
C11	0.0292 (6)	0.0264 (6)	0.0200 (6)	0.0001 (5)	−0.0053 (5)	0.0012 (5)
C12	0.0484 (8)	0.0357 (7)	0.0268 (7)	0.0029 (6)	−0.0085 (6)	−0.0061 (6)
C13	0.0376 (7)	0.0304 (7)	0.0281 (7)	−0.0008 (5)	−0.0038 (5)	−0.0082 (5)
C14	0.0232 (5)	0.0208 (5)	0.0207 (6)	0.0020 (4)	−0.0011 (4)	0.0002 (4)
C15	0.0274 (6)	0.0339 (7)	0.0209 (6)	−0.0046 (5)	0.0017 (5)	0.0014 (5)
C16	0.0255 (6)	0.0347 (7)	0.0248 (6)	−0.0071 (5)	−0.0006 (5)	0.0010 (5)
C17	0.0262 (6)	0.0219 (6)	0.0221 (6)	0.0020 (4)	−0.0015 (5)	−0.0004 (4)
C18	0.0272 (6)	0.0288 (6)	0.0204 (6)	0.0007 (5)	0.0037 (5)	−0.0001 (5)
C19	0.0216 (5)	0.0270 (6)	0.0250 (6)	−0.0011 (4)	0.0013 (5)	−0.0003 (5)
C20	0.0240 (6)	0.0252 (6)	0.0220 (6)	0.0030 (4)	0.0003 (5)	−0.0030 (5)
C21	0.0315 (6)	0.0279 (6)	0.0234 (6)	−0.0002 (5)	0.0013 (5)	−0.0018 (5)
C22	0.0300 (6)	0.0331 (7)	0.0317 (7)	−0.0038 (5)	0.0017 (5)	−0.0093 (5)
C23	0.0294 (6)	0.0396 (7)	0.0266 (7)	0.0066 (5)	−0.0054 (5)	−0.0118 (5)
C24	0.0380 (7)	0.0339 (7)	0.0224 (6)	0.0077 (6)	−0.0024 (5)	−0.0012 (5)
C25	0.0311 (6)	0.0277 (6)	0.0252 (6)	0.0002 (5)	0.0006 (5)	−0.0002 (5)
O1	0.0400 (5)	0.0333 (5)	0.0252 (5)	0.0037 (4)	−0.0122 (4)	−0.0026 (4)
O2	0.0414 (5)	0.0256 (5)	0.0233 (5)	−0.0076 (4)	−0.0066 (4)	−0.0006 (4)
O3	0.0343 (5)	0.0222 (4)	0.0214 (4)	−0.0040 (3)	−0.0041 (4)	0.0024 (3)

Geometric parameters (Å, °)

C1—C11	1.5222 (17)	C12—C13	1.502 (2)
C1—C2	1.5542 (17)	C12—H12A	0.9900
C1—C7	1.5677 (16)	C12—H12B	0.9900
C1—H1	1.0000	C13—O2	1.4234 (15)
C2—C3	1.5517 (18)	C13—H13A	0.9900
C2—C6	1.5603 (17)	C13—H13B	0.9900
C2—H2	1.0000	C14—C15	1.3860 (17)
C3—C4	1.5169 (19)	C14—C19	1.3925 (17)
C3—C10	1.5510 (18)	C15—C16	1.3823 (17)
C3—H3	1.0000	C15—H15	0.9500
C4—C5	1.5166 (18)	C16—C17	1.3879 (18)
C4—H4B	0.9900	C16—H16	0.9500
C4—H4A	0.9900	C17—C18	1.3917 (17)
C5—C6	1.5470 (18)	C17—C20	1.4871 (16)
C5—C9	1.5490 (18)	C18—C19	1.3861 (17)
C5—H5	1.0000	C18—H18	0.9500
C6—C7	1.5551 (16)	C19—H19	0.9500
C6—H6	1.0000	C20—C25	1.3921 (18)
C7—C8	1.5321 (16)	C20—C21	1.3949 (18)
C7—H7	1.0000	C21—C22	1.3851 (18)
C8—O3	1.4236 (14)	C21—H21	0.9500
C8—C14	1.5284 (15)	C22—C23	1.383 (2)
C8—C9	1.5502 (16)	C22—H22	0.9500
C9—C10	1.5856 (16)	C23—C24	1.378 (2)

C9—H9	1.0000	C23—H23	0.9500
C10—C11	1.5144 (17)	C24—C25	1.3893 (18)
C10—H10	1.0000	C24—H24	0.9500
C11—O1	1.4209 (14)	C25—H25	0.9500
C11—O2	1.4299 (15)	O3—H3H	0.876 (19)
C12—O1	1.4265 (17)		
C11—C1—C2	101.86 (10)	C9—C10—H10	113.0
C11—C1—C7	115.36 (10)	O1—C11—O2	104.31 (9)
C2—C1—C7	89.89 (9)	O1—C11—C10	108.47 (10)
C11—C1—H1	115.4	O2—C11—C10	115.96 (10)
C2—C1—H1	115.4	O1—C11—C1	110.66 (10)
C7—C1—H1	115.4	O2—C11—C1	115.56 (10)
C3—C2—C1	107.37 (10)	C10—C11—C1	101.85 (10)
C3—C2—C6	102.79 (10)	O1—C12—C13	102.88 (11)
C1—C2—C6	90.14 (9)	O1—C12—H12A	111.2
C3—C2—H2	117.5	C13—C12—H12A	111.2
C1—C2—H2	117.5	O1—C12—H12B	111.2
C6—C2—H2	117.5	C13—C12—H12B	111.2
C4—C3—C10	104.90 (11)	H12A—C12—H12B	109.1
C4—C3—C2	103.27 (11)	O2—C13—C12	104.61 (11)
C10—C3—C2	100.63 (10)	O2—C13—H13A	110.8
C4—C3—H3	115.4	C12—C13—H13A	110.8
C10—C3—H3	115.4	O2—C13—H13B	110.8
C2—C3—H3	115.4	C12—C13—H13B	110.8
C5—C4—C3	95.24 (10)	H13A—C13—H13B	108.9
C5—C4—H4B	112.7	C15—C14—C19	117.61 (11)
C3—C4—H4B	112.7	C15—C14—C8	122.78 (11)
C5—C4—H4A	112.7	C19—C14—C8	119.55 (10)
C3—C4—H4A	112.7	C16—C15—C14	120.91 (12)
H4B—C4—H4A	110.2	C16—C15—H15	119.5
C4—C5—C6	103.49 (11)	C14—C15—H15	119.5
C4—C5—C9	105.31 (11)	C15—C16—C17	121.85 (12)
C6—C5—C9	100.60 (9)	C15—C16—H16	119.1
C4—C5—H5	115.2	C17—C16—H16	119.1
C6—C5—H5	115.2	C16—C17—C18	117.30 (11)
C9—C5—H5	115.2	C16—C17—C20	118.70 (11)
C5—C6—C7	107.35 (10)	C18—C17—C20	123.85 (11)
C5—C6—C2	102.58 (10)	C19—C18—C17	120.98 (11)
C7—C6—C2	90.13 (9)	C19—C18—H18	119.5
C5—C6—H6	117.6	C17—C18—H18	119.5
C7—C6—H6	117.6	C18—C19—C14	121.34 (11)
C2—C6—H6	117.6	C18—C19—H19	119.3
C8—C7—C6	103.45 (9)	C14—C19—H19	119.3
C8—C7—C1	115.05 (9)	C25—C20—C21	118.05 (11)
C6—C7—C1	89.83 (9)	C25—C20—C17	121.59 (11)
C8—C7—H7	115.1	C21—C20—C17	120.14 (11)
C6—C7—H7	115.1	C22—C21—C20	120.93 (12)
C1—C7—H7	115.1	C22—C21—H21	119.5
O3—C8—C14	103.59 (9)	C20—C21—H21	119.5

O3—C8—C7	116.11 (10)	C23—C22—C21	120.09 (12)
C14—C8—C7	111.83 (9)	C23—C22—H22	120.0
O3—C8—C9	116.65 (9)	C21—C22—H22	120.0
C14—C8—C9	109.42 (9)	C24—C23—C22	119.75 (12)
C7—C8—C9	99.43 (9)	C24—C23—H23	120.1
C5—C9—C8	101.24 (9)	C22—C23—H23	120.1
C5—C9—C10	102.12 (10)	C23—C24—C25	120.16 (12)
C8—C9—C10	115.33 (10)	C23—C24—H24	119.9
C5—C9—H9	112.4	C25—C24—H24	119.9
C8—C9—H9	112.4	C24—C25—C20	120.88 (12)
C10—C9—H9	112.4	C24—C25—H25	119.6
C11—C10—C3	100.19 (10)	C20—C25—H25	119.6
C11—C10—C9	114.16 (10)	C11—O1—C12	106.04 (9)
C3—C10—C9	102.28 (10)	C13—O2—C11	109.14 (9)
C11—C10—H10	113.0	C8—O3—H3H	106.7 (12)
C3—C10—H10	113.0		
C11—C1—C2—C3	12.21 (12)	C8—C9—C10—C3	−108.23 (11)
C7—C1—C2—C3	−103.71 (10)	C3—C10—C11—O1	−63.21 (12)
C11—C1—C2—C6	115.67 (10)	C9—C10—C11—O1	−171.72 (10)
C7—C1—C2—C6	−0.25 (9)	C3—C10—C11—O2	179.88 (10)
C1—C2—C3—C4	127.66 (11)	C9—C10—C11—O2	71.37 (14)
C6—C2—C3—C4	33.44 (12)	C3—C10—C11—C1	53.55 (11)
C1—C2—C3—C10	19.42 (12)	C9—C10—C11—C1	−54.96 (13)
C6—C2—C3—C10	−74.81 (11)	C2—C1—C11—O1	74.93 (12)
C10—C3—C4—C5	51.75 (12)	C7—C1—C11—O1	170.52 (10)
C2—C3—C4—C5	−53.26 (12)	C2—C1—C11—O2	−166.82 (10)
C3—C4—C5—C6	53.65 (12)	C7—C1—C11—O2	−71.23 (13)
C3—C4—C5—C9	−51.53 (13)	C2—C1—C11—C10	−40.23 (11)
C4—C5—C6—C7	−128.13 (11)	C7—C1—C11—C10	55.37 (12)
C9—C5—C6—C7	−19.40 (12)	O1—C12—C13—O2	−23.62 (14)
C4—C5—C6—C2	−33.99 (12)	O3—C8—C14—C15	123.86 (12)
C9—C5—C6—C2	74.74 (11)	C7—C8—C14—C15	−1.91 (16)
C3—C2—C6—C5	0.29 (12)	C9—C8—C14—C15	−111.07 (13)
C1—C2—C6—C5	−107.56 (10)	O3—C8—C14—C19	−53.41 (13)
C3—C2—C6—C7	108.11 (10)	C7—C8—C14—C19	−179.18 (10)
C1—C2—C6—C7	0.25 (9)	C9—C8—C14—C19	71.66 (13)
C5—C6—C7—C8	−12.78 (12)	C19—C14—C15—C16	0.82 (19)
C2—C6—C7—C8	−116.01 (9)	C8—C14—C15—C16	−176.51 (12)
C5—C6—C7—C1	102.98 (10)	C14—C15—C16—C17	0.4 (2)
C2—C6—C7—C1	−0.25 (9)	C15—C16—C17—C18	−1.12 (19)
C11—C1—C7—C8	1.95 (15)	C15—C16—C17—C20	174.66 (12)
C2—C1—C7—C8	105.05 (11)	C16—C17—C18—C19	0.68 (18)
C11—C1—C7—C6	−102.85 (11)	C20—C17—C18—C19	−174.86 (11)
C2—C1—C7—C6	0.25 (9)	C17—C18—C19—C14	0.51 (19)
C6—C7—C8—O3	165.61 (9)	C15—C14—C19—C18	−1.25 (18)
C1—C7—C8—O3	69.38 (13)	C8—C14—C19—C18	176.16 (11)
C6—C7—C8—C14	−75.83 (11)	C16—C17—C20—C25	−139.90 (13)
C1—C7—C8—C14	−172.06 (9)	C18—C17—C20—C25	35.57 (18)
C6—C7—C8—C9	39.61 (11)	C16—C17—C20—C21	34.67 (17)

C1—C7—C8—C9	−56.62 (12)	C18—C17—C20—C21	−149.86 (12)
C4—C5—C9—C8	151.54 (10)	C25—C20—C21—C22	3.91 (18)
C6—C5—C9—C8	44.25 (11)	C17—C20—C21—C22	−170.85 (11)
C4—C5—C9—C10	32.28 (12)	C20—C21—C22—C23	−1.9 (2)
C6—C5—C9—C10	−75.02 (11)	C21—C22—C23—C24	−1.5 (2)
O3—C8—C9—C5	−178.53 (10)	C22—C23—C24—C25	2.8 (2)
C14—C8—C9—C5	64.36 (11)	C23—C24—C25—C20	−0.7 (2)
C7—C8—C9—C5	−52.91 (11)	C21—C20—C25—C24	−2.62 (18)
O3—C8—C9—C10	−69.21 (14)	C17—C20—C25—C24	172.06 (12)
C14—C8—C9—C10	173.68 (10)	O2—C11—O1—C12	−34.19 (13)
C7—C8—C9—C10	56.41 (12)	C10—C11—O1—C12	−158.35 (11)
C4—C3—C10—C11	−150.89 (10)	C1—C11—O1—C12	90.71 (12)
C2—C3—C10—C11	−43.93 (11)	C13—C12—O1—C11	35.81 (14)
C4—C3—C10—C9	−33.20 (12)	C12—C13—O2—C11	3.28 (14)
C2—C3—C10—C9	73.76 (11)	O1—C11—O2—C13	18.51 (13)
C5—C9—C10—C11	107.79 (12)	C10—C11—O2—C13	137.71 (11)
C8—C9—C10—C11	−1.01 (15)	C1—C11—O2—C13	−103.21 (12)
C5—C9—C10—C3	0.57 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3H···O2	0.877 (18)	1.769 (19)	2.6153 (12)	161.6 (18)
C12—H12B···O1^{i}	0.99	2.54	3.2455 (18)	128
C24—H24···O3^{ii}	0.95	2.60	3.4955 (16)	158

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+1, −*y*, −*z*+2.

Fig. 1

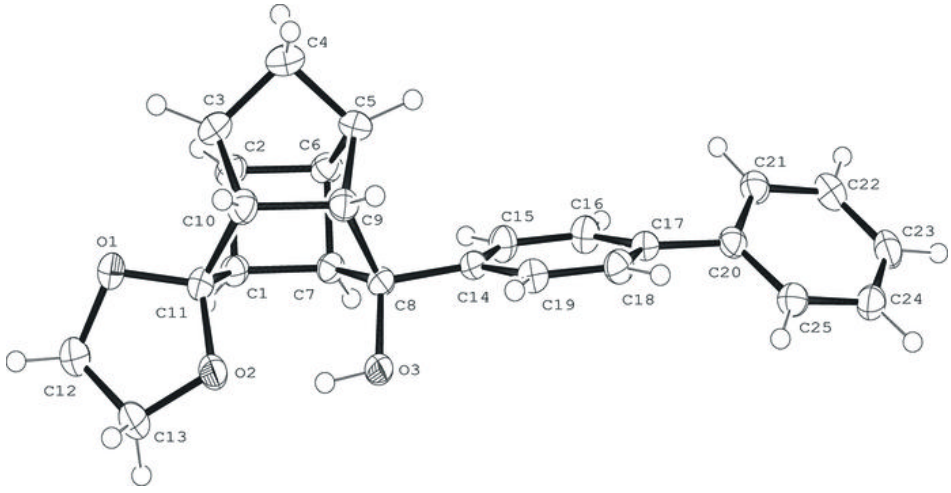

C4
C3
C5
C2
C6
C10
C9
O1
C11
C1
C7
C8
C14
C15
C16
C17
C19
C18
C20
C21
C22
C23
C24
C25
C12
O2
O3
C13

Fig. 2

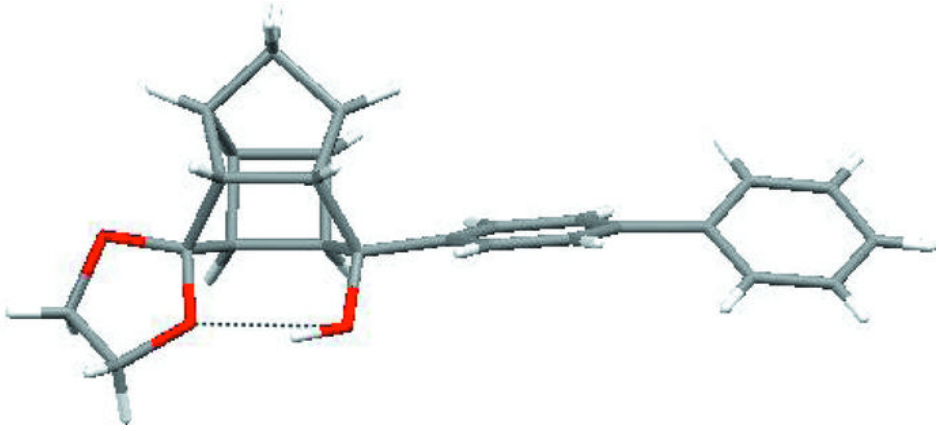


Fig. 3

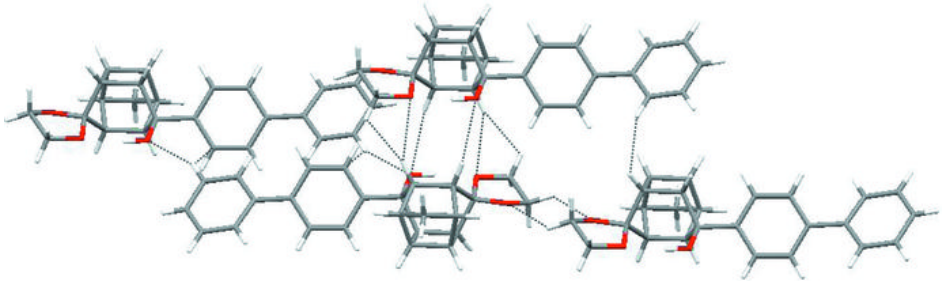


Fig. 4