

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,3-Dihydro-1H-cyclopenta[*b*]naphthalen-1-ol

 İsmail Çelik,^a Mehmet Akkurt,^{b*} Ahmet Tutar,^c Ramazan Erenler^d and Santiago García-Granda^e

^aDepartment of Physics, Faculty of Arts and Sciences, Cumhuriyet University, 58140 Sivas, Turkey, ^bDepartment of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, ^cSakarya University, Faculty of Arts and Sciences, Department of Chemistry, 54187 Adapazarı, Turkey, ^dGaziosmanpaşa University, Faculty of Arts and Sciences, Department of Chemistry, 60240 Tokat, Turkey, and ^eDepartamento Química Física y Analítica, Facultad de Química, Universidad Oviedo, C/ Julián Clavería, 8, 33006 Oviedo (Asturias), Spain
Correspondence e-mail: akkurt@erciyes.edu.tr

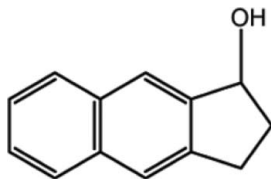
Received 3 February 2012; accepted 6 February 2012

Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 13.7.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{O}$, the cyclopentene ring fused with the naphthalene group adopts an envelope conformation. The cyclopentene torsion angle, with the fusion bond at the centre, has a magnitude of 1.16 (16)°. In the crystal, neighbouring molecules are connected through $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds into an $R_4^4(8)$ ring motif. The crystal packing also features weak $\pi-\pi$ stacking interactions [centroid-centroid distance = 3.8981 (8) Å] and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the synthesis of the title compound, see: Carpino & Lin (1990). For the crystal structure of a similar compound, see: Çelik *et al.* (2009). For puckering parameters, see: Cremer & Pople (1975). For graph-set analysis, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{O}$
 $M_r = 184.23$
 Tetragonal, $I4_1/a$
 $a = 25.3105$ (4) Å
 $c = 6.0995$ (2) Å
 $V = 3907.47$ (18) Å³
 $Z = 16$
 Cu $K\alpha$ radiation
 $\mu = 0.61$ mm⁻¹
 $T = 299$ K
 $0.46 \times 0.17 \times 0.11$ mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
 $T_{\min} = 0.884$, $T_{\max} = 0.936$
 5702 measured reflections
 1791 independent reflections
 1580 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.126$
 $S = 1.04$
 1791 reflections
 131 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.10$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg2 is a centroid of the C1–C6 benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1O}\cdots\text{O1}^i$	0.93 (2)	1.78 (2)	2.7113 (15)	174.4 (18)
$\text{C3}-\text{H3}\cdots\text{Cg2}^{ii}$	0.93	2.71	3.633 (2)	171
$\text{C11}-\text{H11A}\cdots\text{Cg2}^{iii}$	0.97	2.89	3.706 (2)	142

 Symmetry codes: (i) $-y + \frac{1}{4}, x - \frac{1}{4}, z - \frac{1}{4}$; (ii) $-y - \frac{1}{4}, x - \frac{1}{4}, -z - \frac{1}{4}$; (iii) $-x, -y, -z$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QM2052).

References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies UK Ltd, Yarnton, Oxfordshire, England.
 Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Carpino, L. A. & Lin, Y.-Z. (1990). *J. Org. Chem.* **55**, 247–250.
 Çelik, İ., Akkurt, M., Şenocak, A., Çakmak, O., Torre-Fernández, L. & García-Granda, S. (2009). *Acta Cryst.* **E65**, o1376.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2012). E68, o687 [doi:10.1107/S1600536812005181]

2,3-Dihydro-1*H*-cyclopenta[*b*]naphthalen-1-ol

İsmail Çelik, Mehmet Akkurt, Ahmet Tutar, Ramazan Erenler and Santiago García-Granda

S1. Comment

The treatment of benz[*f*]indanone with sodium borohydride in THF/MeOH for 2 h afforded the corresponding benz[*f*]indan-1-ol in an 89% yield (Carpino & Lin, 1990). Two signal groups, aliphatic and aromatic were observed in ¹H-NMR spectra. The H2 and H3 protons exhibited a AA'BB' splitting pattern. H1 appeared at lower magnetic field than the 2,3 H atoms. This is due to the electronegativity of oxygen atom attached to C-1. The signal observed at δ 5.39 as a broad singlet could be attributed to OH group. Moreover a thirteen line ¹³C-NMR spectrum supports the proposed structure. The exact configuration of the molecule was established by X-ray diffraction.

The molecular structure of the title compound (I) is shown in Fig. 1. All bond lengths and angles in the title compound (I) show normal values (Allen *et al.*, 1987; Çelik *et al.*, 2009). The cyclopentene ring (C8–C9/C11–C13) fused with the naphthalene group (C1–C10) adopts an envelope conformation [puckering parameters: $Q(2) = 0.2503(18)$ Å, $\varphi(2) = 110.1(4)^\circ$; (Cremer & Pople, 1975)] with atom C12 deviating from the ring plane. The naphthalene group is essentially planar with a maximum deviation of $-0.013(1)$ Å for C8. The torsion angles C9–C8–C13–O1 and C7–C8–C13–O1 are $-141.52(12)$ and $38.33(19)^\circ$, respectively.

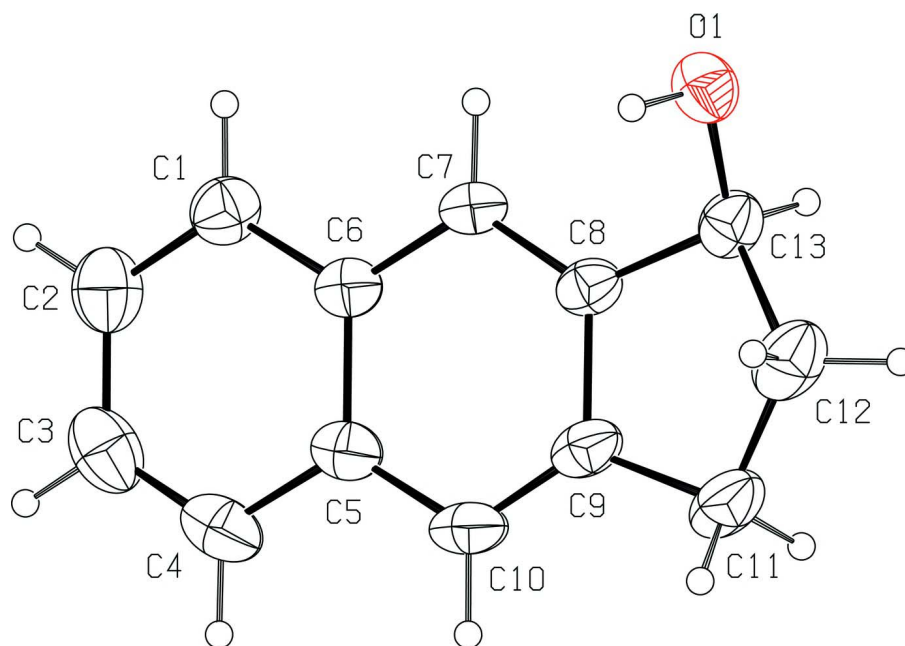
In the crystal, neighbouring molecules are linked with intermolecular O—H \cdots O hydrogen bonds, forming $R_4^4(8)$ ring motifs (Bernstein *et al.*, 1995). A weak π - π stacking interaction [$Cg3\cdots Cg3^{iii} = 3.8981(8)$ Å (symmetry code: (iii) = $-x, -y, -z$)] between the C5–C10 benzene rings and two C—H $\cdots\pi$ interactions contribute to the stabilization of the crystal packing.

S2. Experimental

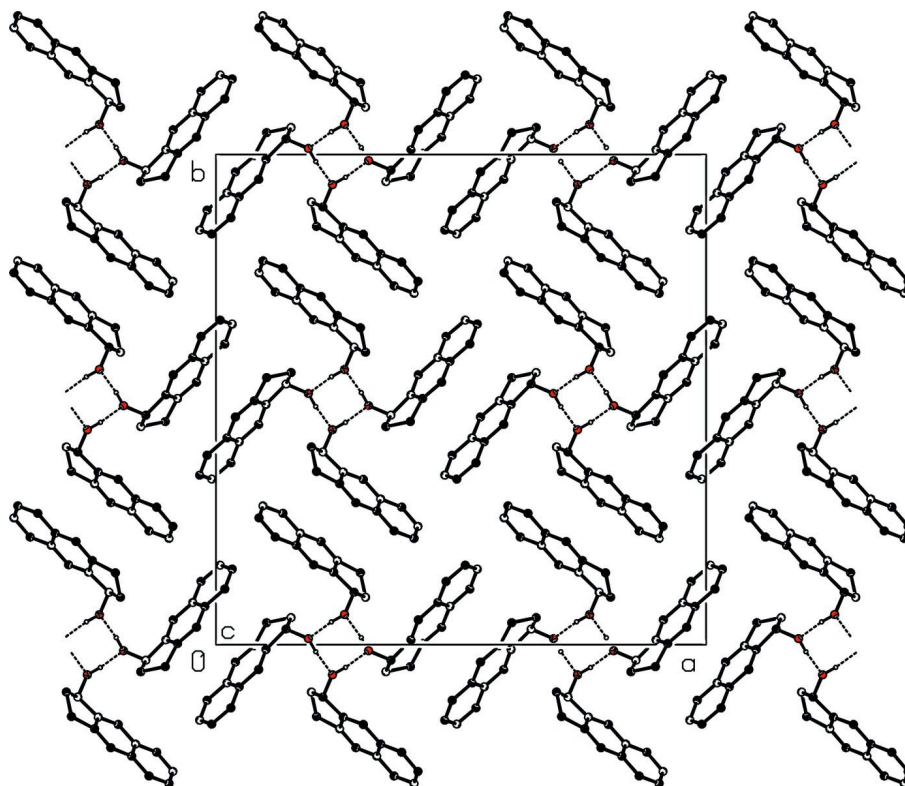
To an ice-cold, stirred solution of benz[*f*]indanone (4.0 g, 22 mmol) in THF/MeOH (30/20 ml) mixture was added NaBH₄ (0.34 g, 8.8 mmol). The reaction mixture was allowed to warm to room temperature. After the completion of the reaction, 2 h, water was added to the reaction mixture which was extracted with diethyl ether (3 \times 50 ml), dried over MgSO₄ and concentrated in *vacuo* affording benz[*f*]indan-1-ol which was crystallized from hexane/chloroform as yellow needles (3.6 g, 89%). ¹H-NMR (400 MHz, CDCl₃): δ 2.0–2.09 (m, 2H, H2), 2.54–2.60 (m, 1H, H3), 2.94–3.02 (m, 1H, H3'), 3.18–3.26 (m, 1H, H1), 5.39 (brs, 1H, OH), 7.43–7.49 (m, 2H, ArH), 7.70 (brs, 1H, ArH), 7.80–7.85 (brs, 2H, ArH), 7.87 (brs, 1H, ArH); ¹³C-NMR (100 MHz, CDCl₃): δ 29.3, 36.6, 75.9, 122.7, 122.9, 125.2, 125.8, 127.6, 128.2, 132.9, 133.9, 141.5, 144.3.

S3. Refinement

The H atom of the hydroxyl group was located in a difference Fourier map and refined freely. Carbon-bound H-atoms were placed in calculated positions and refined with constrained C—H bond lengths of 0.93 Å for aromatic, 0.97 Å for methylene and 0.98 Å for methine H atoms, and $U_{iso}(H) = 1.2 U_{eq}(C)$ allowing them to ride on the parent C atom. The (6 6 0), (-6 12 0) and (-2 6 0) reflections were omitted owing to bad disagreement.

**Figure 1**

The title molecule with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

**Figure 2**

The packing and hydrogen bonding of the title compound, viewing down the *c* axis. H atoms not involved in hydrogen bonding have been omitted.

2,3-Dihydro-1*H*-cyclopenta[*b*]naphthalen-1-ol

Crystal data

C₁₃H₁₂O $M_r = 184.23$ Tetragonal, $I4_1/a$ Hall symbol: $-I\ 4ad$ $a = 25.3105\ (4)\ \text{\AA}$ $c = 6.0995\ (2)\ \text{\AA}$ $V = 3907.47\ (18)\ \text{\AA}^3$ $Z = 16$ $F(000) = 1568$ $D_x = 1.253\ \text{Mg m}^{-3}$ Cu $K\alpha$ radiation, $\lambda = 1.5418\ \text{\AA}$

Cell parameters from 2801 reflections

 $\theta = 3.5\text{--}68.0^\circ$ $\mu = 0.61\ \text{mm}^{-1}$ $T = 299\ \text{K}$

Needle, colourless

 $0.46 \times 0.17 \times 0.11\ \text{mm}$

Data collection

Agilent Xcalibur Ruby Gemini
diffractometer

Radiation source: Enhance (Cu) X-ray Source

Graphite monochromator

Detector resolution: $10.2673\ \text{pixels mm}^{-1}$ ω scansAbsorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2011) $T_{\min} = 0.884$, $T_{\max} = 0.936$

5702 measured reflections

1791 independent reflections

1580 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$ $\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 3.5^\circ$ $h = -30\text{--}18$ $k = -25\text{--}28$ $l = -7\text{--}6$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.044$ $wR(F^2) = 0.126$ $S = 1.04$

1791 reflections

131 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.8441P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.10\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.17\ \text{e \AA}^{-3}$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.18860 (4)	0.01236 (4)	0.19317 (19)	0.0630 (4)
C1	0.03049 (6)	-0.12900 (6)	0.2324 (3)	0.0625 (5)
C2	-0.00864 (7)	-0.16213 (7)	0.1633 (3)	0.0760 (6)
C3	-0.03313 (7)	-0.15412 (8)	-0.0393 (4)	0.0800 (7)

C4	-0.01801 (6)	-0.11348 (7)	-0.1700 (3)	0.0717 (6)
C5	0.02266 (5)	-0.07803 (6)	-0.1061 (2)	0.0550 (4)
C6	0.04727 (5)	-0.08598 (5)	0.1014 (2)	0.0505 (4)
C7	0.08716 (5)	-0.05063 (5)	0.1706 (2)	0.0494 (4)
C8	0.10205 (5)	-0.00984 (5)	0.0392 (2)	0.0487 (4)
C9	0.07840 (6)	-0.00232 (5)	-0.1682 (2)	0.0566 (4)
C10	0.03969 (6)	-0.03549 (6)	-0.2388 (2)	0.0615 (5)
C11	0.10340 (8)	0.04480 (6)	-0.2785 (3)	0.0755 (6)
C12	0.15329 (8)	0.05519 (7)	-0.1448 (3)	0.0794 (6)
C13	0.14311 (6)	0.03180 (5)	0.0832 (2)	0.0570 (4)
H1	0.04640	-0.13480	0.36780	0.0750*
H1O	0.2035 (8)	-0.0143 (8)	0.108 (3)	0.084 (6)*
H2	-0.01910	-0.19020	0.25160	0.0910*
H3	-0.05990	-0.17680	-0.08470	0.0960*
H4	-0.03470	-0.10870	-0.30450	0.0860*
H7	0.10330	-0.05530	0.30630	0.0590*
H10	0.02430	-0.03020	-0.37550	0.0740*
H11A	0.07990	0.07510	-0.27460	0.0910*
H11B	0.11210	0.03690	-0.42990	0.0910*
H12A	0.16010	0.09280	-0.13440	0.0950*
H12B	0.18360	0.03830	-0.21260	0.0950*
H13	0.12710	0.05930	0.17480	0.0680*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0583 (6)	0.0621 (6)	0.0686 (7)	-0.0038 (4)	-0.0021 (5)	-0.0055 (5)
C1	0.0597 (8)	0.0665 (8)	0.0614 (9)	-0.0024 (6)	0.0036 (7)	0.0065 (7)
C2	0.0656 (9)	0.0701 (9)	0.0924 (13)	-0.0087 (7)	0.0122 (9)	-0.0013 (9)
C3	0.0583 (9)	0.0830 (11)	0.0987 (14)	-0.0093 (8)	0.0023 (9)	-0.0230 (10)
C4	0.0569 (8)	0.0917 (11)	0.0666 (10)	0.0124 (7)	-0.0110 (7)	-0.0251 (9)
C5	0.0505 (7)	0.0668 (8)	0.0476 (7)	0.0136 (6)	-0.0029 (5)	-0.0088 (6)
C6	0.0464 (6)	0.0588 (7)	0.0464 (7)	0.0077 (5)	0.0019 (5)	0.0000 (5)
C7	0.0496 (6)	0.0603 (7)	0.0384 (6)	0.0049 (5)	-0.0036 (5)	0.0053 (5)
C8	0.0510 (7)	0.0548 (7)	0.0402 (6)	0.0094 (5)	0.0042 (5)	0.0032 (5)
C9	0.0703 (8)	0.0599 (8)	0.0396 (7)	0.0203 (6)	0.0043 (6)	0.0058 (6)
C10	0.0687 (9)	0.0767 (9)	0.0391 (7)	0.0237 (7)	-0.0097 (6)	-0.0025 (6)
C11	0.1115 (14)	0.0666 (9)	0.0485 (8)	0.0158 (9)	0.0098 (8)	0.0158 (7)
C12	0.0947 (12)	0.0727 (10)	0.0707 (11)	-0.0038 (9)	0.0191 (9)	0.0210 (8)
C13	0.0636 (8)	0.0532 (7)	0.0541 (8)	0.0046 (6)	0.0085 (6)	0.0034 (6)

Geometric parameters (Å, °)

O1—C13	1.4205 (18)	C9—C11	1.508 (2)
O1—H1O	0.93 (2)	C11—C12	1.526 (3)
C1—C6	1.416 (2)	C12—C13	1.533 (2)
C1—C2	1.364 (2)	C1—H1	0.9300
C2—C3	1.397 (3)	C2—H2	0.9300

C3—C4	1.357 (3)	C3—H3	0.9300
C4—C5	1.420 (2)	C4—H4	0.9300
C5—C10	1.414 (2)	C7—H7	0.9300
C5—C6	1.4249 (17)	C10—H10	0.9300
C6—C7	1.4135 (18)	C11—H11A	0.9700
C7—C8	1.3603 (18)	C11—H11B	0.9700
C8—C13	1.5043 (19)	C12—H12A	0.9700
C8—C9	1.4124 (18)	C12—H12B	0.9700
C9—C10	1.360 (2)	C13—H13	0.9800
C13—O1—H10	108.4 (12)	C6—C1—H1	120.00
C2—C1—C6	121.07 (16)	C1—C2—H2	120.00
C1—C2—C3	120.40 (17)	C3—C2—H2	120.00
C2—C3—C4	120.31 (17)	C2—C3—H3	120.00
C3—C4—C5	121.49 (16)	C4—C3—H3	120.00
C4—C5—C6	118.13 (13)	C3—C4—H4	119.00
C6—C5—C10	118.86 (12)	C5—C4—H4	119.00
C4—C5—C10	123.02 (13)	C6—C7—H7	120.00
C1—C6—C5	118.61 (12)	C8—C7—H7	120.00
C5—C6—C7	119.22 (12)	C5—C10—H10	120.00
C1—C6—C7	122.18 (12)	C9—C10—H10	120.00
C6—C7—C8	120.16 (11)	C9—C11—H11A	111.00
C7—C8—C9	120.84 (12)	C9—C11—H11B	111.00
C7—C8—C13	128.17 (11)	C12—C11—H11A	111.00
C9—C8—C13	110.99 (11)	C12—C11—H11B	111.00
C8—C9—C10	120.37 (12)	H11A—C11—H11B	109.00
C8—C9—C11	109.16 (13)	C11—C12—H12A	111.00
C10—C9—C11	130.46 (13)	C11—C12—H12B	111.00
C5—C10—C9	120.55 (12)	C13—C12—H12A	110.00
C9—C11—C12	104.18 (14)	C13—C12—H12B	110.00
C11—C12—C13	106.21 (15)	H12A—C12—H12B	109.00
O1—C13—C12	115.20 (13)	O1—C13—H13	108.00
C8—C13—C12	102.99 (11)	C8—C13—H13	108.00
O1—C13—C8	113.68 (10)	C12—C13—H13	108.00
C2—C1—H1	119.00		
C6—C1—C2—C3	-0.1 (3)	C6—C7—C8—C13	179.66 (12)
C2—C1—C6—C5	-0.4 (2)	C7—C8—C9—C10	0.9 (2)
C2—C1—C6—C7	179.04 (14)	C7—C8—C9—C11	-178.70 (13)
C1—C2—C3—C4	0.4 (3)	C13—C8—C9—C10	-179.25 (13)
C2—C3—C4—C5	-0.1 (3)	C13—C8—C9—C11	1.16 (16)
C3—C4—C5—C6	-0.4 (2)	C7—C8—C13—O1	38.33 (19)
C3—C4—C5—C10	179.53 (16)	C7—C8—C13—C12	163.67 (14)
C4—C5—C6—C1	0.7 (2)	C9—C8—C13—O1	-141.52 (12)
C4—C5—C6—C7	-178.80 (13)	C9—C8—C13—C12	-16.18 (15)
C10—C5—C6—C1	-179.26 (13)	C8—C9—C10—C5	-0.2 (2)
C10—C5—C6—C7	1.25 (19)	C11—C9—C10—C5	179.32 (15)
C4—C5—C10—C9	179.17 (14)	C8—C9—C11—C12	14.52 (17)

C6—C5—C10—C9	-0.9 (2)	C10—C9—C11—C12	-165.02 (16)
C1—C6—C7—C8	179.97 (13)	C9—C11—C12—C13	-24.27 (17)
C5—C6—C7—C8	-0.57 (19)	C11—C12—C13—O1	148.97 (12)
C6—C7—C8—C9	-0.50 (19)	C11—C12—C13—C8	24.64 (16)

Hydrogen-bond geometry (Å, °)

Cg2 is a centroid of the C1—C6 benzene ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1O...O1 ⁱ	0.93 (2)	1.78 (2)	2.7113 (15)	174.4 (18)
C3—H3...Cg2 ⁱⁱ	0.93	2.71	3.633 (2)	171
C11—H11A...Cg2 ⁱⁱⁱ	0.97	2.89	3.706 (2)	142

Symmetry codes: (i) $-y+1/4, x-1/4, z-1/4$; (ii) $-y-1/4, x-1/4, -z-1/4$; (iii) $-x, -y, -z$.