

1,4-Bis[(oxan-2-yl)oxy]benzene

Radhakrishnan Mohan Raj,^a Logu Lekha,^b Vijayan Viswanathan,^c Devadasan Velmurugan^c and Deivanayagam Easwaramoorthy^{a*}

^aDepartment of Chemistry, B.S. Abdur Rahman University, Chennai, Tamil Nadu, India, ^bDepartment of Chemistry, Saveetha School of Engineering, Thandalam, Chennai 602 105, Tamil Nadu, India, and ^cCentre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India. *Correspondence e-mail: easwar@bsauniv.ac.in

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Keywords: crystal structure; oxane; 4*H*-pyran nucleus; intramolecular C—H···O contact.

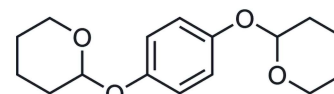
Structural data: full structural data are available from iucrdata.iucr.org

The complete molecule of the title compound, C₁₆H₂₂O₄, is generated by crystallographic inversion symmetry. The oxane ring system adopts a chair conformation with the exocyclic C—C bond in an axial orientation; the dihedral angle between the oxane ring (all non-H atoms) and the benzene ring is 73.45 (1)°. A short intramolecular C—H···O contact occurs. In the crystal, a very weak C—H···π interaction generates [010] chains.

3D view



Chemical scheme



Structure description

The 4*H*-pyran nucleus occurs in molecules possessing a wide spectrum of biological and pharmacological activities (Shehab & Ghoneim, 2016). As part of our studies in this area, we have undertaken the X-ray crystal structure analysis of the title compound (Fig. 1).

The title compound contains a half of the molecule in an asymmetric unit; the complete molecule is generated by crystallographic inversion symmetry with the inversion point at (1, 0, 1) for the asymmetric molecule. The oxane ring (C1–C5/O1) adopts a chair conformation: the mean plane of the oxane ring makes a dihedral angle of 73.45 (1)° with the benzene ring. A short intramolecular C—H···O contact occurs. In the crystal, a very weak C—H···π interaction (Table 1) generates [010] chains (Fig. 2).

Synthesis and crystallization

A solution of *p*-quinone (2.0 mmol), 3,4-dihydro-2*H*-pyran (5.0 mmol) and *p*TsOH (1.0 mmol) in 1,2-dichloroethane (5 ml) was stirred at 83°C under an oxygen atmosphere. The progress of the reaction was monitored by TLC (20% ethyl acetate/hexane). After completion, it was concentrated and the residue was subjected for separation of the

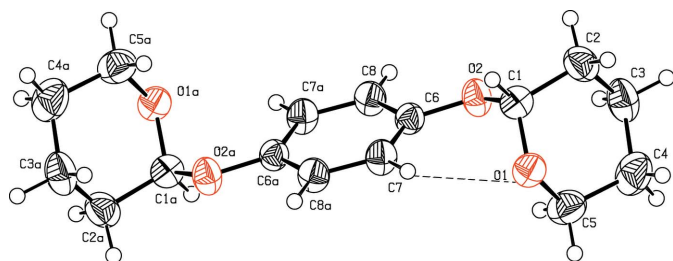


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. The intramolecular C—H···O contact is shown as a dashed line.

respective products by column chromatography using silica gel (eluant: hexane and ethyl acetate): yield: 28%. Colourless blocks were recrystallized from acetonitrile solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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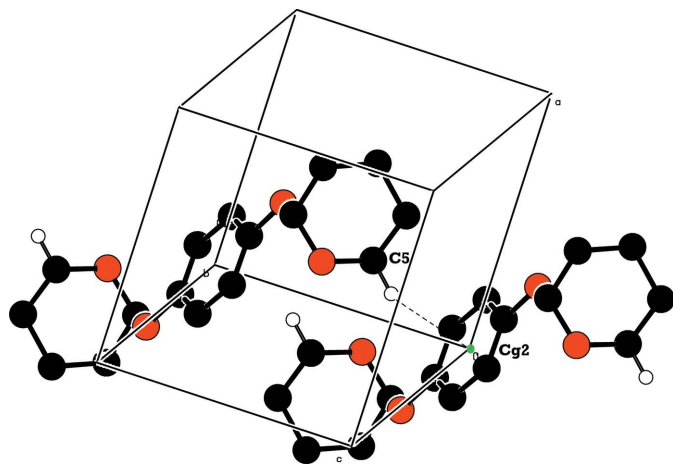


Figure 2
The partial packing of the title compound viewed down the *c* axis showing the C—H··· π interaction as a dashed line. H atoms not involved in this interaction have been excluded for clarity.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

Cg1 is the centroid of the 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>
C7—H7···O1	0.93	2.41	2.966 (2)	119
C5—H5A···Cg1 ⁱ	0.93	2.94	3.621 (3)	129

Symmetry code: (i) *x*, *y* − 1, *z*.

Table 2
Experimental details.

Crystal data	
Chemical formula	$\text{C}_{16}\text{H}_{22}\text{O}_4$
<i>M</i> _r	278.33
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (\AA)	6.767 (5), 6.817 (5), 15.864 (5)
β ($^\circ$)	99.391 (5)
<i>V</i> (\AA^3)	722.0 (8)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm^{-1})	0.09
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2008)
<i>T</i> _{min} , <i>T</i> _{max}	0.756, 0.853
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5817, 1175, 976
<i>R</i> _{int}	0.024
(<i>sin</i> θ / λ) _{max} (\AA^{-1})	0.580
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.033, 0.084, 1.09
No. of reflections	1175
No. of parameters	91
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e \AA^{-3})	0.14, −0.14

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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full crystallographic data

IUCrData (2017). **2**, x170717 [https://doi.org/10.1107/S2414314617007179]

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Crystal data

$C_{16}H_{22}O_4$

$M_r = 278.33$

Monoclinic, $P2_1/n$

$a = 6.767$ (5) Å

$b = 6.817$ (5) Å

$c = 15.864$ (5) Å

$\beta = 99.391$ (5)°

$V = 722.0$ (8) Å³

$Z = 2$

$F(000) = 300$

$D_x = 1.280$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1175 reflections

$\theta = 2.6$ – 24.4 °

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Block, colourless

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

ω and ϕ scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2008)

$T_{\min} = 0.756$, $T_{\max} = 0.853$

5817 measured reflections

1175 independent reflections

976 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 24.4$ °, $\theta_{\min} = 2.6$ °

$h = -7 \rightarrow 7$

$k = -7 \rightarrow 7$

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.084$

$S = 1.09$

1175 reflections

91 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 0.1292P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.14$ e Å⁻³

$\Delta\rho_{\min} = -0.14$ e Å⁻³

Special details

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

Refinement. The hydrogen atoms were placed in calculated positions with C—H = 0.93 Å to 0.98 Å, refined in the riding model with fixed isotropic displacement parameters: $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl group and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for other groups.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6512 (2)	0.2718 (2)	0.83666 (9)	0.0426 (4)
H1	0.683390	0.166073	0.799310	0.051*
C2	0.4358 (2)	0.3315 (2)	0.80928 (10)	0.0526 (4)
H2A	0.348919	0.222001	0.816930	0.063*
H2B	0.413944	0.365739	0.749130	0.063*
C3	0.3834 (2)	0.5052 (3)	0.86102 (10)	0.0582 (5)
H3A	0.249437	0.550953	0.838501	0.070*
H3B	0.386157	0.466188	0.920008	0.070*
C4	0.5329 (3)	0.6680 (3)	0.85653 (13)	0.0660 (5)
H4A	0.517002	0.718745	0.798690	0.079*
H4B	0.507999	0.774275	0.894067	0.079*
C5	0.7431 (2)	0.5937 (2)	0.88259 (11)	0.0565 (4)
H5A	0.837039	0.697816	0.876008	0.068*
H5B	0.762908	0.556370	0.942381	0.068*
C6	0.83603 (19)	0.10172 (19)	0.95814 (9)	0.0377 (3)
C7	1.0155 (2)	0.1063 (2)	0.92723 (9)	0.0431 (4)
H7	1.026663	0.177577	0.878240	0.052*
C8	0.8222 (2)	-0.0044 (2)	1.03046 (9)	0.0425 (4)
H8	0.701375	-0.007568	1.051230	0.051*
O1	0.78259 (13)	0.42824 (14)	0.83219 (6)	0.0445 (3)
O2	0.66348 (13)	0.19763 (15)	0.92132 (6)	0.0456 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0471 (8)	0.0427 (8)	0.0385 (8)	-0.0007 (7)	0.0088 (6)	-0.0002 (6)
C2	0.0457 (8)	0.0616 (11)	0.0477 (9)	-0.0040 (8)	-0.0006 (7)	0.0095 (8)
C3	0.0439 (9)	0.0719 (12)	0.0602 (10)	0.0161 (8)	0.0130 (7)	0.0142 (9)
C4	0.0698 (11)	0.0512 (10)	0.0811 (13)	0.0138 (9)	0.0244 (9)	-0.0040 (9)
C5	0.0593 (10)	0.0461 (10)	0.0658 (11)	-0.0048 (8)	0.0153 (8)	-0.0094 (8)
C6	0.0356 (7)	0.0364 (8)	0.0419 (8)	0.0027 (6)	0.0091 (6)	0.0020 (6)
C7	0.0425 (8)	0.0465 (9)	0.0430 (8)	0.0039 (7)	0.0152 (6)	0.0106 (7)
C8	0.0368 (7)	0.0460 (8)	0.0483 (8)	0.0036 (6)	0.0171 (6)	0.0058 (7)
O1	0.0438 (5)	0.0432 (6)	0.0495 (6)	0.0004 (5)	0.0168 (4)	0.0033 (5)
O2	0.0378 (5)	0.0532 (7)	0.0475 (6)	0.0086 (5)	0.0122 (4)	0.0146 (5)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.3977 (18)	C4—H4B	0.9700
C1—O2	1.4247 (17)	C5—O1	1.4324 (19)
C1—C2	1.507 (2)	C5—H5A	0.9700
C1—H1	0.9800	C5—H5B	0.9700
C2—C3	1.515 (2)	C6—C8	1.372 (2)
C2—H2A	0.9700	C6—O2	1.3818 (17)
C2—H2B	0.9700	C6—C7	1.383 (2)

C3—C4	1.511 (3)	C7—C8 ⁱ	1.378 (2)
C3—H3A	0.9700	C7—H7	0.9300
C3—H3B	0.9700	C8—C7 ⁱ	1.378 (2)
C4—C5	1.503 (2)	C8—H8	0.9300
C4—H4A	0.9700		
O1—C1—O2	112.26 (11)	C5—C4—H4B	109.5
O1—C1—C2	111.88 (13)	C3—C4—H4B	109.5
O2—C1—C2	105.81 (11)	H4A—C4—H4B	108.1
O1—C1—H1	108.9	O1—C5—C4	111.49 (13)
O2—C1—H1	108.9	O1—C5—H5A	109.3
C2—C1—H1	108.9	C4—C5—H5A	109.3
C1—C2—C3	110.83 (12)	O1—C5—H5B	109.3
C1—C2—H2A	109.5	C4—C5—H5B	109.3
C3—C2—H2A	109.5	H5A—C5—H5B	108.0
C1—C2—H2B	109.5	C8—C6—O2	115.75 (12)
C3—C2—H2B	109.5	C8—C6—C7	119.49 (12)
H2A—C2—H2B	108.1	O2—C6—C7	124.76 (13)
C4—C3—C2	109.30 (13)	C8 ⁱ —C7—C6	119.50 (13)
C4—C3—H3A	109.8	C8 ⁱ —C7—H7	120.2
C2—C3—H3A	109.8	C6—C7—H7	120.2
C4—C3—H3B	109.8	C6—C8—C7 ⁱ	121.01 (13)
C2—C3—H3B	109.8	C6—C8—H8	119.5
H3A—C3—H3B	108.3	C7 ⁱ —C8—H8	119.5
C5—C4—C3	110.50 (15)	C1—O1—C5	113.21 (11)
C5—C4—H4A	109.5	C6—O2—C1	118.80 (10)
C3—C4—H4A	109.5		
O1—C1—C2—C3	-55.28 (16)	C7—C6—C8—C7 ⁱ	0.1 (2)
O2—C1—C2—C3	67.28 (16)	O2—C1—O1—C5	-61.30 (15)
C1—C2—C3—C4	53.15 (18)	C2—C1—O1—C5	57.50 (16)
C2—C3—C4—C5	-53.58 (19)	C4—C5—O1—C1	-57.90 (17)
C3—C4—C5—O1	55.58 (19)	C8—C6—O2—C1	-165.57 (12)
C8—C6—C7—C8 ⁱ	-0.1 (2)	C7—C6—O2—C1	14.3 (2)
O2—C6—C7—C8 ⁱ	-179.97 (13)	O1—C1—O2—C6	-68.23 (16)
O2—C6—C8—C7 ⁱ	179.98 (13)	C2—C1—O2—C6	169.46 (12)

Symmetry code: (i) $-x+2, -y, -z+2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

Cg1 is the centroid of the benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 \cdots O1	0.93	2.41	2.966 (2)	119
C5—H5A \cdots Cg1 ⁱⁱ	0.93	2.94	3.621 (3)	129

Symmetry code: (ii) $x, y-1, z$.