

2,3-Bis(phenoxymethyl)buta-1,3-diene

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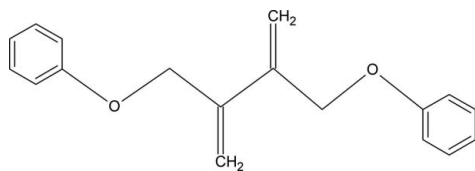
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.126; data-to-parameter ratio = 16.3.

The molecule of the title compound, $\text{C}_{18}\text{H}_{18}\text{O}_2$, a symmetrically phenol-substituted divinyl analog, exhibits crystallographically imposed C_2 symmetry. The phenolic and divinyl planar groups intersect each other orthogonally, with a dihedral angle of $82.7(1)^\circ$. The structure is stabilized by a short intramolecular $\text{C}-\text{H}\cdots\text{O}$ contact. The molecules are held together by $\text{C}-\text{H}\cdots\pi$ interactions, forming a sheet structure parallel to the (201) plane.

Related literature

The crystal structures of three analogous compounds have been published thus far (Alcock *et al.*, 2006; Sathiyarayanan *et al.*, 2007, 2008). For molecular and crystal symmetry, see Yao *et al.* (2002); Pidcock *et al.* (2003); Narasegowda *et al.* (2005); Schmidt *et al.* (2006).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{18}\text{O}_2$
 $M_r = 266.32$
Monoclinic, $P2_1/c$
 $a = 10.7575(5)$ Å
 $b = 7.0750(3)$ Å
 $c = 9.7939(5)$ Å
 $\beta = 109.180(2)^\circ$

$V = 704.03(6)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 295(2)$ K
 $0.30 \times 0.20 \times 0.16$ mm

Data collection

Bruker Kappa APEXII
diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2004)
 $T_{\min} = 0.958$, $T_{\max} = 0.981$

9151 measured reflections
2065 independent reflections
1558 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.126$
 $S = 1.02$
2065 reflections

127 parameters
All H-atom parameters refined
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C9}-\text{H9B}\cdots\text{O1}$	0.98 (2)	2.41 (2)	2.770 (2)	101 (1)
$\text{C4}-\text{H4A}\cdots\text{Cg1}^i$	0.97 (2)	2.92 (2)	3.727 (2)	142 (1)
$\text{C9}-\text{H9A}\cdots\text{Cg1}^{ii}$	0.99 (2)	2.73 (2)	3.591 (1)	146 (1)

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z - \frac{1}{2}$; (ii) $x, y + 1, z$. Cg1 is the centroid of the C1–C6 ring.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2124).

References

- Alcock, N. W., Leigh, D. A. & Lacy, S. M. (2006). Private communication (refcode XAVTIY). CCDC, Union Road, Cambridge, England.
- Bruker (2004). *APEX2* (Version 1.08), *SAINT* (Version 7.23A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Narasegowda, R. S., Malathy Sony, S. M., Mondal, S., Nagaraj, B., Yathirajan, H. S., Narasimhamurthy, T., Charles, P., Ponnuswamy, M. N., Nethaji, M. & Rathore, R. S. (2005). *Acta Cryst.* **E61**, o843–o845.
- Pidcock, E., Motherwell, W. D. S. & Cole, J. C. (2003). *Acta Cryst.* **B59**, 634–640.
- Sathiyarayanan, K., George Fernand, A., Dhanasekaran, V. & Rathore, R. S. (2007). *Acta Cryst.* **E63**, o2504–o2505.
- Sathiyarayanan, K., George Fernand, A., Dhanasekaran, V. & Rathore, R. S. (2008). *Acta Cryst.* **E64**, o123.
- Schmidt, M. U., Schmiermund, T. & Bolte, M. (2006). *Acta Cryst.* **C62**, m37–m40.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Yao, J. W., Cole, J. C., Pidcock, E., Allen, F. H., Howard, J. A. K. & Motherwell, W. D. S. (2002). *Acta Cryst.* **B58**, 640–646.

supplementary materials

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2,3-Bis(phenoxyethyl)buta-1,3-diene

K. Sathiyarayanan, A. George Fernand, V. Dhanasekaran and R. S. Rathore

Comment

Previously, we had reported a benzenethiol-substituted divinyl analog, ({2-methylidene-3-[(phenylsulfanyl)methyl]but-3-en-1-yl}sulfanyl)benzene, (I), possessing a C_2 point-group symmetry at the center of divinyl group. In this series, the title compound {[2-methylidene-3-(phenoxyethyl)but-3-en-1-yl]oxy}benzene, (III), is a symmetrically phenol-substituted divinyl analog and discussed in the present report. The molecular structure with atom numbering scheme is shown in Fig 1. Three similar compounds have been reported so far. They are: (a) (I) in space group $Pbca$ (Sathiyarayanan *et al.*, 2007); (b) 2-methylphenol-substituted divinyl analog *i.e.*, 1-methyl-2-({2-methylidene-3-[(2-methylphenoxy)methyl]but-3-en-1-yl}oxy)benzene, (II), in space group $P2_1/n$ (Sathiyarayanan *et al.*, 2008); and (c) 4-(3-hydroxy-3-methoxypropyl)phenol-substituted analog, namely, 2,3-bis(4-(2-(methoxycarbonyl)ethyl)phenoxyethyl)buta-1,3-diene, (IV), in space group $P 2_1/c$ (Alcock *et al.* 2006; CCDC 277599, private communication).

The molecular symmetry (C_2) is retained in the crystal of (III) and the asymmetric unit is composed of one-half of the molecule ($Z' = 1/2$) as observed in (I), (II) and (IV). The database analysis has revealed that among organic molecules, there is a persistent tendency for molecular symmetry to be retained in the crystal (Yao *et al.*, 2002), although exceptions to this general trend have also been reported even in the case of inversion center that is mostly conserved (Narasegowda *et al.*, 2005; Schmidt *et al.*, 2006). Recent work (Pidcock *et al.*, 2003) has led to the conclusion that the C_2 point group symmetry is conserved in about 60% of their reported cases.

Selected bond distances and angles are provided in Table 1. The least square planes in (III) are defined by phenol (O1/C1—C6) and divinyl [$C7/C8/C9/C7^i/C8^i/C9^i$; symmetry code (i): $1 - x, 1 - y, -z$] groups. These planar groups intersect each other orthogonally [inclination angle is $82.7 (1)^\circ$], as also observed in (I). In contrast, (II) is essentially planar and in (IV), the corresponding adjacent groups are coplanar. The torsion angles describing molecular conformation namely, $C2-C1-O1-C7$, $C8-C7-O1-C1$ and $O1-C7-C8-C8^i$ are *trans*, *gauche*⁻ and *trans*, respectively (Table 1).

Hydrogen bond parameters are provided in Table 1. The structure is stabilized by a short intermolecular contact $C9-H9B \cdots O1$. (III) lacks any conventional hydrogen-bonding donors. As a result of that, the crystal packing is determined purely by weak intermolecular forces. The molecules form a sheet structure in (2 0 1) plane that are held together by $C4-H4A \cdots Cg1^{ii}$ [symmetry code (ii): $-x, -1/2 + y, -1/2 - z$] and $C9-H9A \cdots Cg1^{iii}$ [symmetry code (iii): $x, 1 + y, z$]. $Cg1$ is the centroid of (C1—C6) ring. The crystal packing view is shown in Fig. 2.

Experimental

One mole of 2,3-bis(iodomethyl)buta-1,3-diene in DMF was added to two moles of sodium phenoxide in DMF dropwise with cooling. The reaction mixture was stirred overnight at room temperature and poured into crushed ice. The solids were filtered and dissolved in ether. The ether extract was washed with sodium thiosulfate and 10% sodium hydroxide and finally

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with water. The solid product was obtained by removal of ether after drying, which was recrystallized from hexane at room temperature (m.p. 364° K).

Refinement

The positions of all the H atoms were freely refined. The distances with H-atoms are in ranges:- C_{aromatic}—H = 0.94 (1)–1.00 (2); C_{sp²}—H = 0.98 (2)–0.99 (2); and C_{methylene}—H = 0.96 (2)–1.00 (2) Å.

Figures

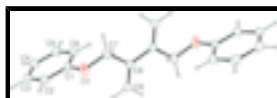


Fig. 1. A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level.

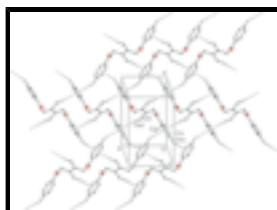


Fig. 2. C—H...π mediated molecular association into a sheet structure in (2 0 1) plane shown in a crystal packing view. Cg1 is the centroid of (C1—C6) ring. Dashed lines represent hydrogen bonds. For clarity, only the selected H atoms, involved in the interactions, are shown.

2,3-Bis(phenoxyethyl)buta-1,3-diene

Crystal data

C₁₈H₁₈O₂

M_r = 266.32

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 10.7575 (5) Å

b = 7.0750 (3) Å

c = 9.7939 (5) Å

β = 109.180 (2)°

V = 704.03 (6) Å³

Z = 2

*F*₀₀₀ = 284

D_x = 1.256 Mg m⁻³

Melting point: 364 K

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 3326 reflections

θ = 3.5–29.4°

μ = 0.08 mm⁻¹

T = 295 (2) K

Prism, colourless

0.30 × 0.20 × 0.16 mm

Data collection

Bruker Kappa
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 295(2) K

ω and φ-scan

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

2065 independent reflections

1558 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.026

θ_{max} = 30.2°

θ_{min} = 2.0°

h = -15→14

$T_{\min} = 0.958$, $T_{\max} = 0.981$
9151 measured reflections

$k = -9 \rightarrow 9$
 $l = -7 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.126$

$S = 1.02$

2065 reflections

127 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.0874P]$$

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

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

$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

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.

Weighted least-squares planes through the starred atoms (Nardelli, Musatti, Domiano & Andreotti Ric.Sci.(1965),15(II—A),807).
Equation of the plane: $m_1 * X + m_2 * Y + m_3 * Z = d$

Plane 1 $m_1 = -0.70263(0.00094)$ $m_2 = -0.43813(0.00092)$ $m_3 = 0.56068(0.00161)$ $D = -5.31928(0.00495)$ Atom d s d/s (d/s)**2
C7 * 0.0000 0.0013 0.000 0.000 C8 * 0.0000 0.0010 0.000 0.000 C9 * 0.0000 0.0013 0.000 0.000 O1 0.0945 0.0009 108.132
11692.442 ===== Sum((d/s)**2) for starred atoms 0.000

Plane 2 $m_1 = -0.52237(0.00027)$ $m_2 = 0.81703(0.00021)$ $m_3 = -0.24412(0.00046)$ $D = -0.87330(0.00135)$ Atom d s d/s (d/s)**2
O1 * -0.0015 0.0009 - 1.703 2.900 C1 * -0.0008 0.0010 - 0.726 0.527 C2 * 0.0022 0.0012 1.889 3.567 C3 * 0.0011 0.0012 0.877
0.769 C4 * -0.0031 0.0013 - 2.359 5.563 C5 * -0.0023 0.0013 - 1.742 3.035 C6 * 0.0048 0.0012 4.058 16.471 C7 - 0.2091 0.0013 -
164.276 26986.654 ===== Sum((d/s)**2) for starred atoms 32.831 Chi-squared at 95% for 4 degrees of freedom: 9.49 The
group of atoms deviates significantly from planarity

Dihedral angles formed by LSQ-planes Plane - plane angle (s.u.) angle (s.u.) 1 2 82.66 (0.06) 97.34 (0.06)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.27879 (10)	0.09878 (14)	-0.21059 (11)	0.0352 (2)
C2	0.19476 (11)	0.00566 (16)	-0.33013 (11)	0.0406 (3)
H2A	0.2117 (13)	0.011 (2)	-0.4223 (16)	0.053 (4)*

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C3	0.08982 (12)	-0.09532 (17)	-0.31788 (13)	0.0453 (3)
H3A	0.0324 (14)	-0.158 (2)	-0.4013 (17)	0.056 (4)*
C4	0.06662 (13)	-0.10558 (18)	-0.18746 (14)	0.0486 (3)
H4A	-0.0079 (17)	-0.176 (2)	-0.1799 (18)	0.068 (5)*
C5	0.15010 (13)	-0.01250 (18)	-0.06949 (13)	0.0475 (3)
H5A	0.1334 (14)	-0.017 (2)	0.0254 (17)	0.060 (4)*
C6	0.25622 (12)	0.09092 (16)	-0.07924 (11)	0.0398 (3)
H6A	0.3116 (13)	0.158 (2)	0.0011 (15)	0.048 (3)*
C7	0.48149 (11)	0.26949 (18)	-0.11341 (14)	0.0445 (3)
H7A	0.5603 (15)	0.277 (2)	-0.1465 (17)	0.064 (4)*
H7B	0.5038 (14)	0.180 (2)	-0.0351 (15)	0.046 (3)*
C8	0.45057 (9)	0.46076 (14)	-0.06518 (10)	0.0344 (2)
O1	0.38019 (8)	0.19483 (12)	-0.23461 (9)	0.0474 (2)
C9	0.34154 (12)	0.55102 (19)	-0.13827 (14)	0.0472 (3)
H9B	0.2789 (14)	0.496 (2)	-0.2264 (16)	0.053 (4)*
H9A	0.3180 (16)	0.677 (2)	-0.1096 (18)	0.068 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0346 (5)	0.0316 (5)	0.0387 (5)	0.0011 (4)	0.0112 (4)	-0.0036 (4)
C2	0.0431 (6)	0.0404 (6)	0.0355 (5)	0.0012 (5)	0.0089 (4)	-0.0049 (4)
C3	0.0402 (6)	0.0406 (6)	0.0470 (6)	-0.0019 (5)	0.0033 (5)	-0.0059 (5)
C4	0.0407 (6)	0.0438 (7)	0.0612 (7)	-0.0051 (5)	0.0166 (5)	0.0018 (5)
C5	0.0531 (7)	0.0475 (7)	0.0459 (6)	0.0000 (5)	0.0215 (5)	0.0025 (5)
C6	0.0441 (6)	0.0384 (6)	0.0350 (5)	-0.0006 (5)	0.0103 (4)	-0.0043 (4)
C7	0.0338 (6)	0.0431 (6)	0.0546 (7)	-0.0031 (5)	0.0119 (5)	-0.0096 (5)
C8	0.0316 (5)	0.0335 (5)	0.0377 (5)	-0.0036 (4)	0.0110 (4)	0.0026 (4)
O1	0.0453 (5)	0.0537 (5)	0.0464 (4)	-0.0140 (4)	0.0194 (4)	-0.0128 (4)
C9	0.0408 (6)	0.0432 (6)	0.0481 (6)	0.0011 (5)	0.0019 (5)	0.0026 (5)

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

C1—O1	1.3695 (13)	C5—H5A	1.003 (16)
C1—C6	1.3865 (14)	C6—H6A	0.944 (14)
C1—C2	1.3880 (14)	C7—O1	1.4232 (14)
C2—C3	1.3741 (17)	C7—C8	1.5060 (15)
C2—H2A	0.979 (15)	C7—H7A	1.003 (16)
C3—C4	1.3816 (18)	C7—H7B	0.961 (15)
C3—H3A	0.958 (16)	C8—C9	1.3206 (16)
C4—C5	1.3759 (18)	C8—C8 ⁱ	1.476 (2)
C4—H4A	0.967 (17)	C9—H9B	0.983 (15)
C5—C6	1.3854 (17)	C9—H9A	0.991 (17)
O1—C1—C6	124.85 (9)	C5—C6—H6A	121.1 (8)
O1—C1—C2	115.18 (9)	C1—C6—H6A	119.8 (8)
C6—C1—C2	119.97 (10)	O1—C7—C8	114.05 (10)
C3—C2—C1	119.95 (10)	O1—C7—H7A	104.4 (9)
C3—C2—H2A	120.5 (8)	C8—C7—H7A	110.4 (9)

C1—C2—H2A	119.6 (8)	O1—C7—H7B	110.1 (8)
C2—C3—C4	120.75 (11)	C8—C7—H7B	111.3 (8)
C2—C3—H3A	119.1 (9)	H7A—C7—H7B	106.1 (13)
C4—C3—H3A	120.2 (9)	C9—C8—C8 ⁱ	123.11 (13)
C5—C4—C3	119.00 (11)	C9—C8—C7	120.85 (10)
C5—C4—H4A	120.8 (10)	C8 ⁱ —C8—C7	116.03 (11)
C3—C4—H4A	120.2 (10)	C1—O1—C7	118.41 (9)
C4—C5—C6	121.34 (11)	C8—C9—H9B	121.1 (8)
C4—C5—H5A	119.8 (9)	C8—C9—H9A	123.0 (10)
C6—C5—H5A	118.9 (9)	H9B—C9—H9A	115.9 (13)
C5—C6—C1	118.99 (10)		
O1—C1—C2—C3	179.90 (10)	C2—C1—C6—C5	0.74 (17)
C6—C1—C2—C3	-0.46 (16)	O1—C7—C8—C9	-4.17 (16)
C1—C2—C3—C4	0.00 (17)	C6—C1—O1—C7	9.79 (16)
C2—C3—C4—C5	0.18 (19)	C2—C1—O1—C7	-170.59 (10)
C3—C4—C5—C6	0.11 (19)	C8—C7—O1—C1	-84.83 (13)
C4—C5—C6—C1	-0.57 (18)	O1—C7—C8—C8 ⁱ	176.68 (10)
O1—C1—C6—C5	-179.66 (10)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9B \cdots O1	0.98 (2)	2.41 (2)	2.770 (2)	101 (1)
C4—H4A \cdots Cg1 ⁱⁱ	0.97 (2)	2.92 (2)	3.727 (2)	142 (1)
C9—H9A \cdots Cg1 ⁱⁱⁱ	0.99 (2)	2.73 (2)	3.591 (1)	146 (1)

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

Fig. 1

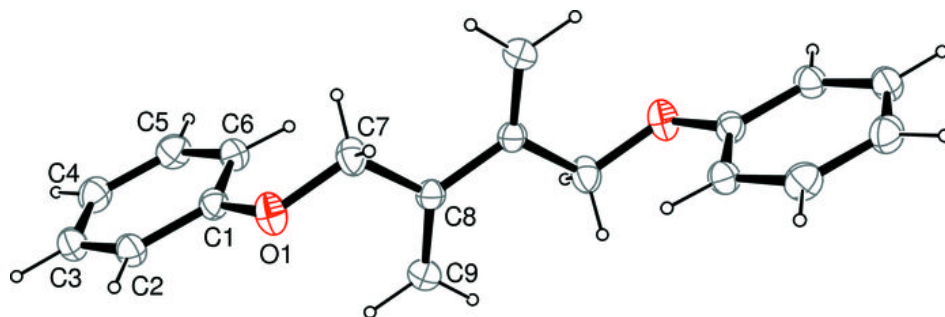


Fig. 2

