Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,3-Bis[(2-methylphenoxy)methyl]-buta-1,3-diene

K. Sathiyanarayanan,[a] A. George Fernand,[a] V. Dhanasekaran[a] and R. S. Rathore[b]*

[a]Chemistry Division, School of Science and Humanities, VIT University, Vellore 632 014, India, and [b]School of Biotechnology, Devi Ahilya University, Indore 452 001, India
Correspondence e-mail: ravindranath_rathore@yahoo.com

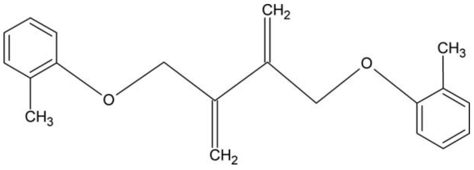
Received 14 November 2007; accepted 26 November 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.060; wR factor = 0.141; data-to-parameter ratio = 11.4.

The molecule of the title compound, $C_{20}H_{22}O_2$, a symmetrically 2-methylphenol-substituted divinyl analog, exhibits crystallographically imposed C_2 symmetry. The molecular structure is essentially planar. The structure is stabilized by a short intermolecular C—H···O contact. Cooperative C—H···π interactions generate an infinite one-dimensional chain of molecules along the a axis.

Related literature

The crystal structures of three analogous compounds have been published thus far (Alcock *et al.*, 2006; Sathiyanarayanan *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

$C_{20}H_{22}O_2$
M_r = 294.38
Monoclinic, $P2_1/n$
a = 5.2241 (5) Å
b = 21.6274 (19) Å
c = 7.5101 (7) Å
β = 102.044 (4)°
V = 829.84 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 295 (2) K
0.22 × 0.18 × 0.16 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.973, T_{max} = 0.980
7281 measured reflections
1632 independent reflections
1352 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.141
S = 1.15
1632 reflections
143 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O1	0.95 (3)	2.29 (3)	2.655 (4)	102 (2)
C8—H8B···$Cg1^{i}$	1.00 (2)	2.70 (3)	3.534 (3)	141 (2)

Symmetry code: (i) $x + 1, y, z$. $Cg1$ 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*.

We thank the DRDO, India, for financial support (grant No. ERIP/ER/0403461/M/01). RSR acknowledges the Science & Engineering Research Council (DST), Government of India, for providing a Fast-Track grant and the Bioinformatics Center of the School of Biotechnology, Devi Ahilya University, Indore, India, for the use of the computational facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2123).

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.* E**61**, o843–o845.
Pidcock, E., Motherwell, W. D. S. & Cole, J. C. (2003). *Acta Cryst.* B**59**, 634–640.
Sathiyanarayanan, K., George Fernand, A., Dhanasekaran, V. & Rathore, R. S. (2007). *Acta Cryst.* E**63**, o2504–o2505.
Sathiyanarayanan, K., George Fernand, A., Dhanasekaran, V. & Rathore, R. S. (2008). *Acta Cryst.* E**64**, o124.
Schmidt, M. U., Schmiermund, T. & Bolte, M. (2006). *Acta Cryst.* C**62**, 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.* B**58**, 640–646.

supporting information

Acta Cryst. (2008). E**64**, o123   [https://doi.org/10.1107/S1600536807063398]

2,3-Bis[(2-methylphenoxy)methyl]buta-1,3-diene

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

S1. Comment

We had previously 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 1-methyl-2-({2-methylidene-3-[(2-methylphenoxy)methyl] but-3-en-1-yl}oxy)benzene, (II), is a symmetrically 2-methylphenol-substituted divinyl analog. The present discussion is concerned with the structure of (II). 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 (Sathiyanarayanan *et al.*, 2007); (*b*) phenol-substituted divinyl analog *i.e.*, {[2-methylidene-3-(phenoxymethyl)but-3-en-1-yl]oxy}benzene, (III), in space group $P2_1/c$ (Sathiyanarayanan *et al.*, 2008); and (*c*) 4-(3-hydroxy-3-methoxypropyl)phenol-substituted analog, namely, 2,3-bis(4-(2-(methoxycarbonyl)ethyl)phenoxymethyl)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 (II) and the asymmetric unit is composed of one-half of the molecule ($Z' = 1/2$) as observed in (I), (III) 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 trend have also been reported even in case of inversion center that is mostly conserved in the crystal (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 the reported cases.

Selected bond distances, angles and conformational parameters are provided in Table. 1. 2-methylphenol (O1/C1—C7) and divinyl [C8/C9/C10/C8^i/C9^i/C10^i; symmetry code (i): 2 - *x*, -*y*, -*z*] units are coplanar, giving rise to essentially a planar structure of (II). All the three torsion angles describing molecular conformation *i.e.*, C1—C6—O1—C8, C9—C8—O1—C6 and O1—C8—C9—C9^i are *trans*. The corresponding angles in (IV) are also *trans*, while in (I) and (III) these angles are [*trans*, *trans* and (*gauche*$^-$ or *gauche*$^+$)] and [*trans*, *gauche*$^-$ and *trans*], respectively. Among other differences, the analog (I) exhibits a flip-flop disorder of divinyl group (Sathiyanarayanan *et al.*, 2007)

An intra-molecular C10—H10B···O1 hydrogen bond stabilize the structure of (I). Hydrogen bond parameters are provided in Table 2. Crystal packing is purely governed by weak intermolecular forces. The molecular association *via* cooperative C8—H8B ··· *Cg*1$^{(ii)}$ interactions [symmetry code (ii): 1 + *x*, *y*, *z*] give rise to one-dimensional chain of molecules along *a* axis. *Cg*1 is the centroid of (C1—C6) ring. Crystal packing view is diplayed in Fig. 2.

S2. 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 with water. The solid product was obtained by removal of ether after drying, which was recrystallized from hexane at room temperature (m.p. 346° K).

S3. Refinement

The positions of all H atoms were freely refined. The distances with H-atoms are in ranges:- $C_{aromatic}$—H = 0.97 (2)–1.02 (3); C_{sp^2}—H = 0.95 (3)–1.02 (3); C_{methyl}—H = 0.99 (4)–1.02 (3) and $C_{methylene}$—H = 1.00 (2)–1.01 (2) Å.

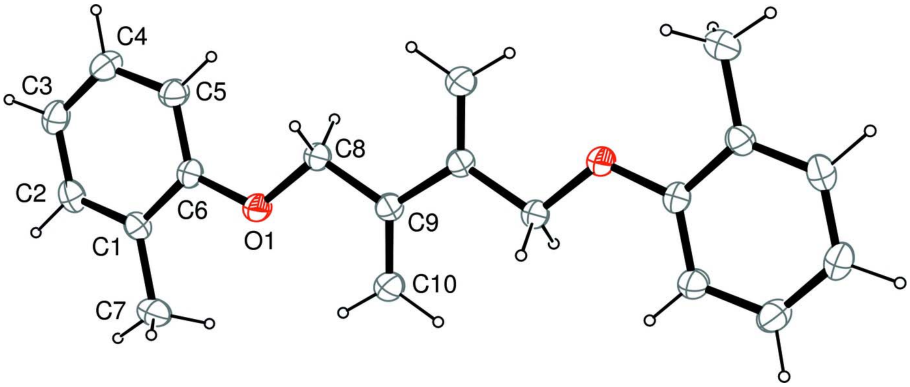


Figure 1

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

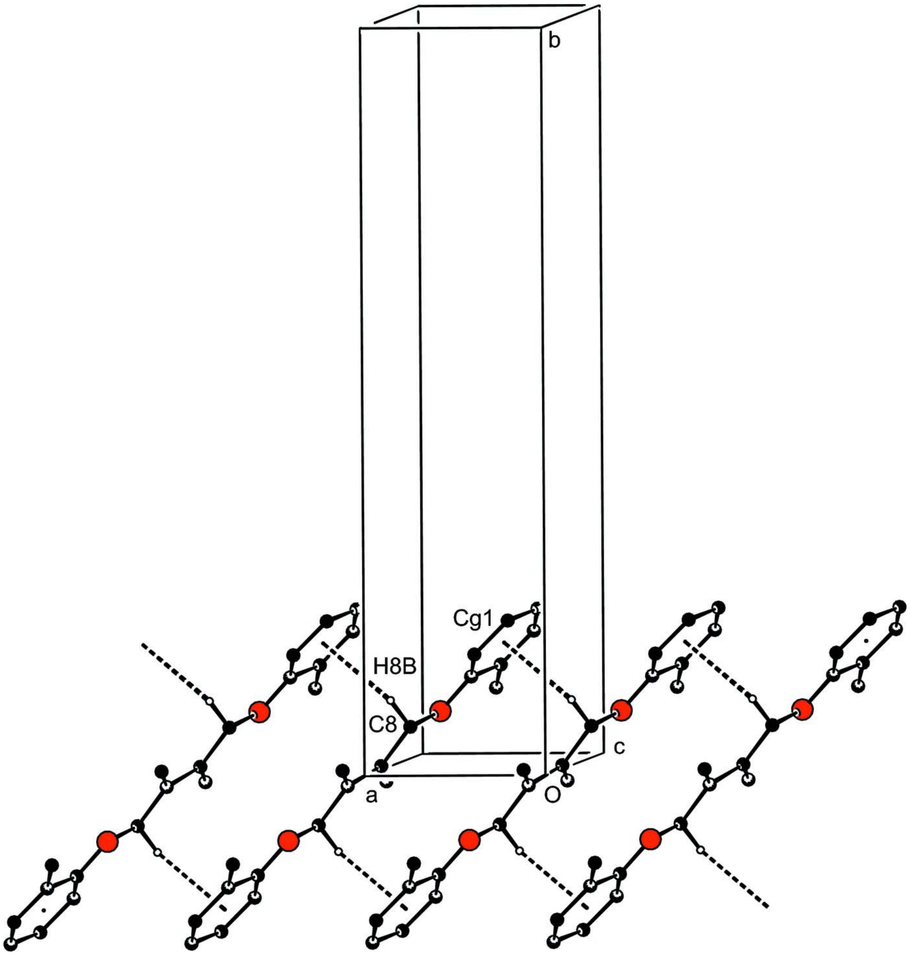


Figure 2

Projection of the crystal packing down the *c* axis showing cooperative C—H···*π* interactions forming one-dimensional chain of molecular along *a* axis. *Cg*1 is the centroid of (C1—C6) ring. Only selected hydrogen atoms are shown.

2,3-Bis[(2-methylphenoxy)methyl]buta-1,3-diene

Crystal data

$C_{20}H_{22}O_2$
$M_r = 294.38$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.2241$ (5) Å
$b = 21.6274$ (19) Å
$c = 7.5101$ (7) Å
$\beta = 102.044$ (4)°
$V = 829.84$ (13) Å^3
$Z = 2$

$F(000) = 316$
$D_x = 1.178$ Mg m^{-3}
Melting point: 346 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4455 reflections
$\theta = 2.8$–31.0°
$\mu = 0.07$ mm^{-1}
$T = 295$ K
Prism, colourless
0.22 × 0.18 × 0.16 mm

Data collection

BrukerKkappa diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ–scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min} = 0.973$, $T_{\max} = 0.980$

7281 measured reflections
1632 independent reflections
1352 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 26.0°$, $\theta_{\min} = 2.9°$
$h = -6 \rightarrow 6$
$k = -25 \rightarrow 26$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.141$
$S = 1.15$
1632 reflections
143 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 0.7904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

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 & Andreetti Ric.Sci.(1965),15(II—A),807). Equation of the plane: m1**X*+m2*Y+m3**Z*=d
Plane 1 m1 = -0.65734(0.00051) m2 = -0.66742(0.00044) m3 = -0.34994(0.00085) D = -3.59801(0.00113) Atom d s d/s (d/s)**2 O1 * 0.0138 0.0018 7.856 61.715 C1 * -0.0049 0.0023 - 2.109 4.447 C2 * 0.0165 0.0026 6.321 39.959 C3 * 0.0134 0.0027 4.955 24.548 C4 * -0.0055 0.0027 - 2.046 4.184 C5 * -0.0199 0.0026 - 7.572 57.329 C6 * -0.0077 0.0022 - 3.472 12.052 C7 * -0.0354 0.0037 - 9.660 93.324 C8 0.0645 0.0024 26.886 722.869 C9 0.1395 0.0021 64.912 4213.542 C10 0.1687 0.0030 56.080 3144.964 ============ Sum((d/s)**2) for starred atoms 297.558 Chi-squared at 95% for 5 degrees of freedom: 11.10 The group of atoms deviates significantly from planarity
Plane 2 m1 = -0.68850(0.00264) m2 = -0.62945(0.00157) m3 = -0.36022(0.00316) D = -3.60224(0.00936) Atom d s d/s (d/s)**2 C8 * 0.0000 0.0024 0.000 0.000 C9 * 0.0000 0.0021 0.000 0.000 C10 * 0.0000 0.0030 0.000 0.000 O1 - 0.0218 0.0018 - 12.401 153.784 ============ Sum((d/s)**2) for starred atoms 0.000
Plane 3 m1 = -0.67385(0.00031) m2 = -0.65235(0.00031) m3 = -0.34694(0.00075) D = -3.56623(0.00074) Atom d s d/s (d/s)**2 O1 * -0.0381 0.0018 - 21.735 472.397 C1 * -0.0064 0.0023 - 2.735 7.482 C2 * 0.0453 0.0026 17.389 302.387 C3 * 0.0520 0.0027 19.220 369.400 C4 * 0.0123 0.0027 4.532 20.540 C5 * -0.0327 0.0026 - 12.442 154.803 C6 * -0.0301 0.0022 - 13.569 184.128 C7 * -0.0475 0.0037 - 12.943 167.509 C8 * -0.0100 0.0024 - 4.185 17.517 C9 * 0.0345 0.0021 16.077 258.455 C10 * 0.0611 0.0030 20.305 412.299 ============ Sum((d/s)**2) for starred atoms 2366.918 Chi-squared at 95% for 8 degrees of freedom: 15.50 The group of atoms deviates significantly from planarity
Dihedral angles formed by LSQ-planes Plane - plane angle (s.u.) angle (s.u.) 1 2 2.88 (0.12) 177.12 (0.12) 1 3 1.29 (0.03) 178.71 (0.03) 2 3 1.73 (0.13) 178.27 (0.13)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.3342 (4)	0.14009 (11)	0.2814 (3)	0.0386 (5)
C2	0.1449 (5)	0.18556 (12)	0.2633 (4)	0.0464 (6)
H2	0.055 (5)	0.1937 (12)	0.369 (4)	0.056*
C3	0.0729 (5)	0.21981 (12)	0.1049 (4)	0.0501 (7)
H3	−0.070 (5)	0.2516 (13)	0.098 (4)	0.059 (8)*
C4	0.1911 (5)	0.20830 (12)	−0.0385 (4)	0.0504 (7)
H4	0.151 (6)	0.2322 (14)	−0.150 (4)	0.071 (9)*
C5	0.3809 (5)	0.16273 (12)	−0.0253 (3)	0.0459 (6)
H5	0.466 (5)	0.1542 (11)	−0.125 (3)	0.045 (7)*
C6	0.4503 (4)	0.12846 (10)	0.1331 (3)	0.0368 (5)
C7	0.4171 (7)	0.10395 (17)	0.4548 (4)	0.0592 (8)
H7A	0.416 (6)	0.0576 (17)	0.432 (4)	0.081 (10)*
H7B	0.297 (7)	0.1125 (16)	0.537 (5)	0.095 (12)*
H7C	0.601 (8)	0.1140 (18)	0.512 (5)	0.108 (14)*
C8	0.7492 (5)	0.06522 (11)	0.0126 (3)	0.0378 (5)
C9	0.9288 (4)	0.01133 (10)	0.0684 (3)	0.0354 (5)
C10	0.9538 (6)	−0.01399 (14)	0.2310 (4)	0.0532 (7)
O1	0.6296 (3)	0.08156 (8)	0.1591 (2)	0.0478 (5)
H8A	0.608 (5)	0.0526 (10)	−0.094 (3)	0.037 (6)*

H8B	0.848 (5)	0.1015 (12)	−0.019 (3)	0.044 (7)*
H10A	1.075 (5)	−0.0505 (13)	0.270 (4)	0.058 (8)*
H10B	0.863 (5)	0.0022 (12)	0.318 (4)	0.053 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0386 (12)	0.0410 (13)	0.0358 (12)	−0.0014 (10)	0.0069 (9)	−0.0044 (10)
C2	0.0435 (14)	0.0495 (15)	0.0480 (14)	0.0027 (11)	0.0135 (11)	−0.0113 (11)
C3	0.0456 (14)	0.0426 (15)	0.0612 (17)	0.0109 (12)	0.0089 (12)	−0.0008 (12)
C4	0.0504 (15)	0.0459 (15)	0.0548 (16)	0.0070 (12)	0.0106 (12)	0.0122 (12)
C5	0.0493 (14)	0.0476 (15)	0.0439 (14)	0.0086 (11)	0.0166 (11)	0.0087 (11)
C6	0.0341 (11)	0.0344 (12)	0.0422 (12)	0.0028 (9)	0.0085 (9)	−0.0006 (10)
C7	0.072 (2)	0.069 (2)	0.0381 (14)	0.0117 (17)	0.0150 (14)	0.0041 (13)
C8	0.0373 (12)	0.0397 (13)	0.0365 (12)	0.0060 (10)	0.0085 (10)	0.0012 (10)
C9	0.0299 (11)	0.0389 (12)	0.0362 (12)	0.0012 (9)	0.0042 (9)	−0.0009 (9)
C10	0.0617 (17)	0.0565 (17)	0.0434 (14)	0.0210 (14)	0.0156 (12)	0.0080 (12)
O1	0.0539 (11)	0.0516 (11)	0.0405 (9)	0.0211 (8)	0.0158 (8)	0.0076 (8)

Geometric parameters (Å, º)

C1—C2	1.381 (3)	C7—H7A	1.02 (3)
C1—C6	1.397 (3)	C7—H7B	0.99 (4)
C1—C7	1.504 (4)	C7—H7C	0.99 (4)
C2—C3	1.385 (4)	C8—O1	1.418 (3)
C2—H2	1.02 (3)	C8—C9	1.500 (3)
C3—C4	1.372 (4)	C8—H8A	1.01 (2)
C3—H3	1.01 (3)	C8—H8B	1.00 (2)
C4—C5	1.387 (3)	C9—C10	1.320 (3)
C4—H4	0.97 (3)	C9—C9^{i}	1.472 (4)
C5—C6	1.384 (3)	C10—H10A	1.02 (3)
C5—H5	0.97 (2)	C10—H10B	0.95 (3)
C6—O1	1.367 (3)		
C2—C1—C6	118.1 (2)	C1—C7—H7B	110 (2)
C2—C1—C7	121.8 (2)	H7A—C7—H7B	108 (3)
C6—C1—C7	120.1 (2)	C1—C7—H7C	110 (2)
C1—C2—C3	121.7 (2)	H7A—C7—H7C	105 (3)
C1—C2—H2	119.0 (15)	H7B—C7—H7C	112 (3)
C3—C2—H2	119.3 (15)	O1—C8—C9	109.15 (18)
C4—C3—C2	119.4 (2)	O1—C8—H8A	108.4 (13)
C4—C3—H3	122.2 (16)	C9—C8—H8A	108.9 (13)
C2—C3—H3	118.3 (16)	O1—C8—H8B	109.0 (14)
C3—C4—C5	120.3 (2)	C9—C8—H8B	110.8 (14)
C3—C4—H4	122.0 (18)	H8A—C8—H8B	110.6 (19)
C5—C4—H4	117.7 (18)	C10—C9—C9^{i}	122.7 (3)
C6—C5—C4	119.8 (2)	C10—C9—C8	121.1 (2)
C6—C5—H5	119.1 (15)	C9^{i}—C9—C8	116.2 (2)

C4—C5—H5	121.1 (15)	C9—C10—H10A	121.5 (15)
O1—C6—C5	124.7 (2)	C9—C10—H10B	120.9 (16)
O1—C6—C1	114.7 (2)	H10A—C10—H10B	118 (2)
C5—C6—C1	120.6 (2)	C6—O1—C8	118.28 (17)
C1—C7—H7A	112.0 (18)		

C6—C1—C2—C3	−1.0 (4)	C7—C1—C6—O1	2.4 (3)
C7—C1—C2—C3	178.5 (3)	C2—C1—C6—C5	1.5 (3)
C1—C2—C3—C4	0.3 (4)	C7—C1—C6—C5	−178.1 (3)
C2—C3—C4—C5	0.1 (4)	O1—C8—C9—C10	0.9 (3)
C3—C4—C5—C6	0.4 (4)	C5—C6—O1—C8	−1.8 (3)
C4—C5—C6—O1	178.3 (2)	C1—C6—O1—C8	177.6 (2)
C4—C5—C6—C1	−1.1 (4)	C9—C8—O1—C6	−177.60 (19)
C2—C1—C6—O1	−178.0 (2)	O1—C8—C9—C9^{i}	−179.5 (2)

Symmetry code: (i) −*x*+2, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O1	0.95 (3)	2.29 (3)	2.655 (4)	102 (2)
C8—H8*B*···*Cg*1^{ii}	1.00 (2)	2.70 (3)	3.534 (3)	141 (2)

Symmetry code: (ii) *x*+1, *y*, *z*.