



Crystal structure of (*E*)-4,4'-(but-2-ene-1,4-diyl)bis(2-methoxyphenol)

Kyle S. Knight* and Patrick J. Carey

Department of Chemistry, The University of Tennessee at Chattanooga, Chattanooga, TN 37403, USA. *Correspondence e-mail: kyle-knight@utc.edu

Received 9 June 2015; accepted 15 June 2015

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

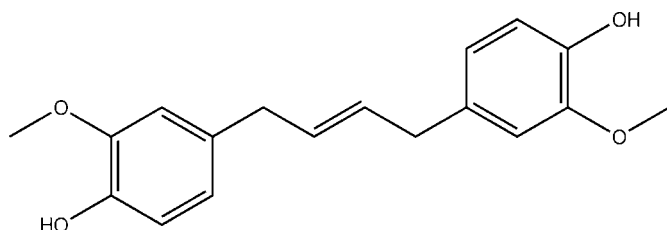
The title compound, C₁₈H₂₀O₄, was synthesized *via* the ruthenium-catalyzed alkene metathesis dimerization of eugenol. The whole molecule is generated by inversion symmetry; the center of inversion being located at the midpoint of the *trans* C=C bond. The phenol ring is inclined to the mean plane of the central C=C=C unit (r.m.s. deviation = 0.014 Å) by 68.83 (16)°. In the crystal, molecules are linked *via* O—H...O hydrogen bonds, involving the hydroxy and methoxy groups, forming undulating sheets parallel to (010).

Keywords: crystal structure; metathesis; dimerization of eugenol; hydrogen bonding.

CCDC reference: 1406832

1. Related literature

For a general review of alkene metathesis catalyzed by ruthenium carbenes, see: Grubbs (2004). For the second generation Grubbs ruthenium carbene catalyst, see: Scholl *et al.* (1999). For the synthesis of the title compound, see: Taber & Frankowski (2006).



2. Experimental

2.1. Crystal data

C₁₈H₂₀O₄

M_r = 300.34

Orthorhombic, *Pbca*
a = 4.8846 (2) Å
b = 10.7002 (4) Å
c = 29.5666 (11) Å
V = 1545.33 (10) Å³

Z = 4
Mo *K*α radiation
μ = 0.09 mm⁻¹
T = 198 K
0.6 × 0.55 × 0.2 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.927, *T_{max}* = 1.000

25610 measured reflections
1352 independent reflections
1199 reflections with *I* > 2σ(*I*)
R_{int} = 0.036

2.3. Refinement

R[*F*² > 2σ(*F*²)] = 0.033
wR(*F*²) = 0.096
S = 1.05
1352 reflections

105 parameters
H-atom parameters constrained
Δρ_{max} = 0.16 e Å⁻³
Δρ_{min} = -0.13 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<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>
O8—H8...O1 ⁱ	0.78 (2)	2.57 (2)	3.1784 (13)	136 (1)

Symmetry code: (i) *x* + ½, *y*, -*z* + ½.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2*.

Acknowledgements

We are grateful to the National Science Foundation MRI Program (CHE-0951711), the Grote Chemistry Fund at the University of Tennessee at Chattanooga, and to Materia Inc. of Pasadena, CA, USA, for their generous support of our work.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5153).

References

- Bruker (2009). *APEX2*, *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Grubbs, R. H. (2004). *Tetrahedron*, **60**, 7117–7140.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Scholl, M., Ding, S., Lee, C. W. & Grubbs, R. H. (1999). *Org. Lett.* **1**, 953–956.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Taber, D. F. & Frankowski, K. J. (2006). *J. Chem. Educ.* **83**, 283–284.

supporting information

Acta Cryst. (2015). E71, o500 [doi:10.1107/S2056989015011585]

Crystal structure of (*E*)-4,4'-(but-2-ene-1,4-diyl)bis(2-methoxyphenol)

Kyle S. Knight and Patrick J. Carey

S1. Synthesis and crystallization

The title compound was prepared from eugenol by alkene metathesis dimerization using the second generation Grubbs ruthenium carbene catalyst (Scholl *et al.*, 1999) as described previously (Taber & Frankowski, 2006).

S2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl H atom was located in a difference Fourier map and refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The C-bound H atoms were positioned geometrically and constrained to ride on their parent atoms: C—H = 0.95 - 1.0 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for other H atoms.

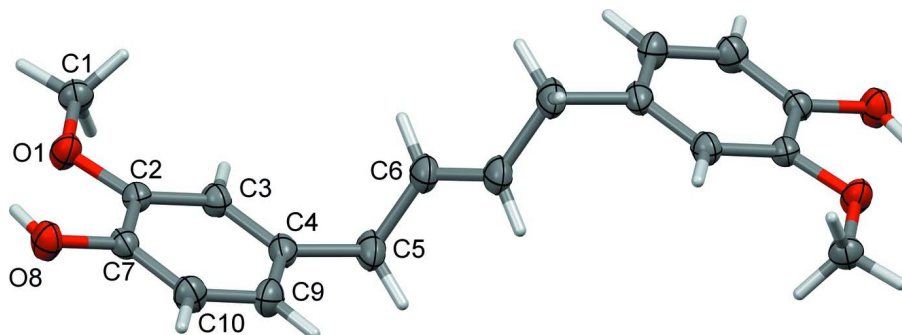
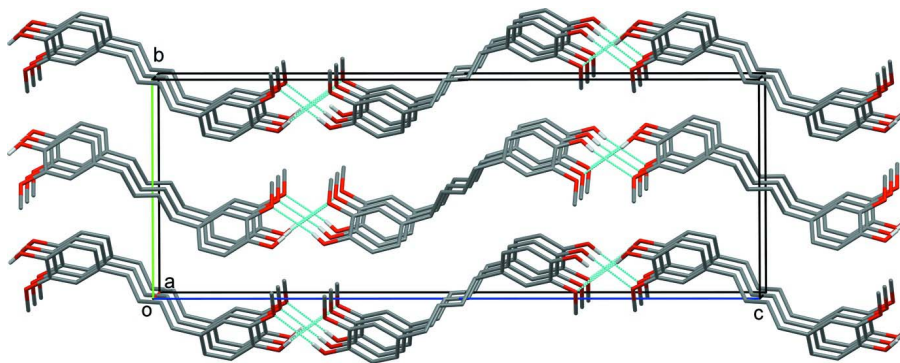


Figure 1

A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to the labelled atoms by inversion symmetry (symmetry code: $-x + 1, -y + 2, -z$).

**Figure 2**

A view along the *a* axis of the crystal packing of the title compound, with hydrogen bonds shown as dashed lines (see Table 1 for details). C-bound H atoms have been omitted for clarity.

(E)-4,4'-(But-2-ene-1,4-diyl)bis(2-methoxyphenol)*Crystal data* $C_{18}H_{20}O_4$ $M_r = 300.34$ Orthorhombic, *Pbca* $a = 4.8846 (2) \text{ \AA}$ $b = 10.7002 (4) \text{ \AA}$ $c = 29.5666 (11) \text{ \AA}$ $V = 1545.33 (10) \text{ \AA}^3$ $Z = 4$ $F(000) = 640$ $D_x = 1.291 \text{ Mg m}^{-3}$ Mo *K* α radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9944 reflections

 $\theta = 2.8\text{--}24.8^\circ$ $\mu = 0.09 \text{ mm}^{-1}$ $T = 198 \text{ K}$

Plate, colorless

 $0.6 \times 0.55 \times 0.2 \text{ mm}$ *Data collection*Bruker APEXII CCD
diffractometer

Graphite monochromator

 φ and ω scansAbsorption correction: multi-scan
(*SADABS*; Bruker, 2009) $T_{\min} = 0.927$, $T_{\max} = 1.000$

25610 measured reflections

1352 independent reflections

1199 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.036$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.8^\circ$ $h = -5 \rightarrow 5$ $k = -12 \rightarrow 12$ $l = -35 \rightarrow 35$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.033$ $wR(F^2) = 0.096$ $S = 1.05$

1352 reflections

105 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.352P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.13 \text{ e \AA}^{-3}$ Extinction correction: *SHELXL2014* (Sheldrick,
2015), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.009 (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.

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	0.3753 (2)	0.94521 (9)	0.20535 (3)	0.0531 (3)
C1	0.1882 (3)	1.04703 (13)	0.20379 (5)	0.0505 (4)
H1A	0.2599	1.1120	0.1837	0.076*
H1B	0.1646	1.0815	0.2343	0.076*
H1C	0.0111	1.0178	0.1924	0.076*
C2	0.4437 (3)	0.88991 (11)	0.16499 (4)	0.0395 (3)
C3	0.3285 (3)	0.91684 (12)	0.12337 (4)	0.0454 (3)
H3	0.1922	0.9798	0.1211	0.054*
C4	0.4108 (3)	0.85237 (13)	0.08473 (4)	0.0475 (4)
C5	0.2924 (3)	0.88644 (16)	0.03888 (4)	0.0605 (4)
H5A	0.3136	0.8146	0.0181	0.073*
H5B	0.0941	0.9035	0.0422	0.073*
C6	0.4294 (3)	0.99880 (15)	0.01871 (4)	0.0554 (4)
H6	0.4116	1.0754	0.0347	0.066*
C7	0.6459 (3)	0.79875 (11)	0.16851 (4)	0.0419 (3)
O8	0.7636 (2)	0.77137 (10)	0.20946 (3)	0.0556 (3)
H8	0.699 (3)	0.8143 (16)	0.2278 (6)	0.067*
C9	0.6067 (3)	0.76024 (14)	0.08901 (4)	0.0552 (4)
H9	0.6619	0.7143	0.0631	0.066*
C10	0.7244 (3)	0.73365 (14)	0.13057 (5)	0.0536 (4)
H10	0.8598	0.6702	0.1329	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0688 (7)	0.0527 (6)	0.0378 (5)	0.0162 (5)	-0.0078 (4)	-0.0042 (4)
C1	0.0538 (8)	0.0470 (8)	0.0505 (8)	0.0073 (6)	0.0032 (6)	-0.0004 (6)
C2	0.0455 (7)	0.0381 (6)	0.0349 (6)	-0.0030 (5)	-0.0011 (5)	0.0016 (5)
C3	0.0491 (8)	0.0459 (7)	0.0413 (7)	0.0003 (6)	-0.0055 (5)	0.0057 (6)
C4	0.0534 (8)	0.0541 (8)	0.0349 (7)	-0.0139 (7)	-0.0006 (5)	0.0057 (5)
C5	0.0676 (10)	0.0772 (10)	0.0368 (7)	-0.0170 (8)	-0.0082 (6)	0.0073 (6)
C6	0.0660 (10)	0.0653 (9)	0.0348 (6)	-0.0031 (8)	-0.0081 (6)	0.0054 (6)
C7	0.0456 (7)	0.0415 (7)	0.0386 (6)	-0.0018 (5)	-0.0004 (5)	0.0071 (5)
O8	0.0644 (7)	0.0597 (6)	0.0426 (6)	0.0163 (5)	-0.0077 (5)	0.0057 (4)
C9	0.0643 (9)	0.0621 (9)	0.0393 (7)	-0.0016 (7)	0.0112 (6)	-0.0037 (6)
C10	0.0564 (8)	0.0544 (8)	0.0500 (8)	0.0109 (7)	0.0084 (6)	0.0038 (6)

Geometric parameters (Å, °)

O1—C1	1.4228 (16)	C5—H5A	0.9900
O1—C2	1.3732 (15)	C5—H5B	0.9900
C1—H1A	0.9800	C5—C6	1.500 (2)
C1—H1B	0.9800	C6—C6 ⁱ	1.304 (3)
C1—H1C	0.9800	C6—H6	0.9500
C2—C3	1.3834 (17)	C7—O8	1.3720 (15)
C2—C7	1.3921 (18)	C7—C10	1.3751 (19)
C3—H3	0.9500	O8—H8	0.777 (17)
C3—C4	1.3938 (18)	C9—H9	0.9500
C4—C5	1.5183 (17)	C9—C10	1.386 (2)
C4—C9	1.380 (2)	C10—H10	0.9500
C2—O1—C1	117.26 (10)	H5A—C5—H5B	107.9
O1—C1—H1A	109.5	C6—C5—C4	112.18 (12)
O1—C1—H1B	109.5	C6—C5—H5A	109.2
O1—C1—H1C	109.5	C6—C5—H5B	109.2
H1A—C1—H1B	109.5	C5—C6—H6	116.9
H1A—C1—H1C	109.5	C6 ⁱ —C6—C5	126.11 (19)
H1B—C1—H1C	109.5	C6 ⁱ —C6—H6	116.9
O1—C2—C3	125.76 (12)	O8—C7—C2	120.87 (11)
O1—C2—C7	114.17 (10)	O8—C7—C10	119.65 (12)
C3—C2—C7	120.07 (11)	C10—C7—C2	119.46 (11)
C2—C3—H3	119.7	C7—O8—H8	108.7 (13)
C2—C3—C4	120.59 (13)	C4—C9—H9	119.5
C4—C3—H3	119.7	C4—C9—C10	121.05 (12)
C3—C4—C5	120.21 (13)	C10—C9—H9	119.5
C9—C4—C3	118.58 (12)	C7—C10—C9	120.23 (13)
C9—C4—C5	121.19 (13)	C7—C10—H10	119.9
C4—C5—H5A	109.2	C9—C10—H10	119.9
C4—C5—H5B	109.2		
O1—C2—C3—C4	-178.58 (12)	C3—C2—C7—C10	-1.74 (19)
O1—C2—C7—O8	-1.02 (18)	C3—C4—C5—C6	80.50 (17)
O1—C2—C7—C10	177.67 (12)	C3—C4—C9—C10	-1.4 (2)
C1—O1—C2—C3	-6.20 (19)	C4—C5—C6—C6 ⁱ	116.9 (2)
C1—O1—C2—C7	174.44 (11)	C4—C9—C10—C7	0.4 (2)
C2—C3—C4—C5	-177.32 (12)	C5—C4—C9—C10	176.73 (13)
C2—C3—C4—C9	0.8 (2)	C7—C2—C3—C4	0.75 (19)
C2—C7—C10—C9	1.2 (2)	O8—C7—C10—C9	179.89 (13)
C3—C2—C7—O8	179.57 (12)	C9—C4—C5—C6	-97.55 (17)

Symmetry code: (i) $-x+1, -y+2, -z$.

Hydrogen-bond geometry (Å, °)

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
----------------	-----	--------------	--------------	----------------

O8—H8 \cdots O1 ⁱⁱ	0.78 (2)	2.57 (2)	3.1784 (13)	136 (1)
---------------------------------	----------	----------	-------------	---------

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