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4-[(*E*)-4-Hydroxybut-2-en-1-yl]-2-methoxyphenol

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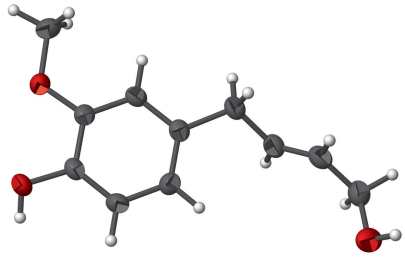
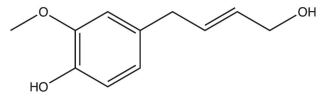
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{11}H_{14}O_3$, was synthesized by a cross-metathesis reaction. The dihedral angle between the aromatic ring and the butenol side chain is 30.2 (2)°. In the crystal, inversion dimers are formed through O—H···O hydrogen bonds and these are linked into chains by additional O—H···O contacts. These chains are linked into sheets in the *bc* plane by C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) was synthesized by the cross-metathesis (Scholl, *et al.* 1999) of eugenol and *cis*-2-butene-1,4-diol, as previously described (Taber & Frankowski, 2006). This compound is a natural product that can also be isolated from the rhizomes of *Zingiber cassumunar* (Masuda & Jitoe, 1995).

The dihedral angle between the aromatic ring and the butenol side chain is 30.2 (2)°. The methyl group of the methoxy-substituent is twisted out of the plane of the aromatic ring by 6.8 (2)°. In the crystal, the unit cell contains inversion dimers connected by hydrogen bonding. Each phenol hydroxyl group acts as a hydrogen-bond donor to the allylic hydroxyl in its dimeric counterpart through O1—H1···O2 hydrogen bonds (Table 1 and Fig. 2). The allylic hydroxyl group is a bifurcated donor, forming O2—H2···O1 and O2—H2···O3 hydrogen bonds that link the dimers into supramolecular chains propagated along the *c*-axis direction. Chains of dimers are linked by C7–H7···O3 hydrogen bonds forming sheets of molecules in the *bc* plane

Synthesis and crystallization

The Grubbs second-generation catalyst, tricyclohexylphosphine[1,3-bis-(2,4,6-trimethylphenyl)-4,5-dihydroimidazol-2-ylidene][benzylidene]ruthenium(IV) dichloride (Grubbs, 2004), was used to facilitate the cross metathesis of eugenol with *cis*-1,4-butenediol, to

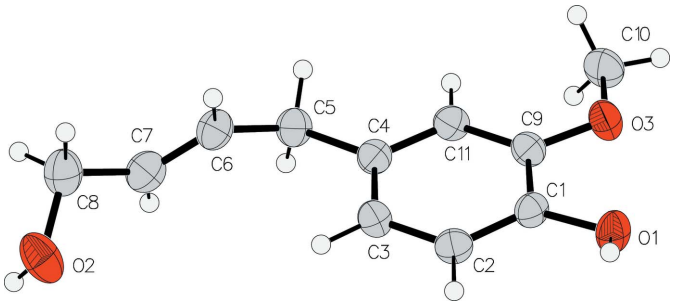


Figure 1
A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

form the title compound. The product was mpurified by column chromatography, and allowed to crystallize from dichloromethane at room temperature over the course of 14 days.

Refinement

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

Acknowledgements

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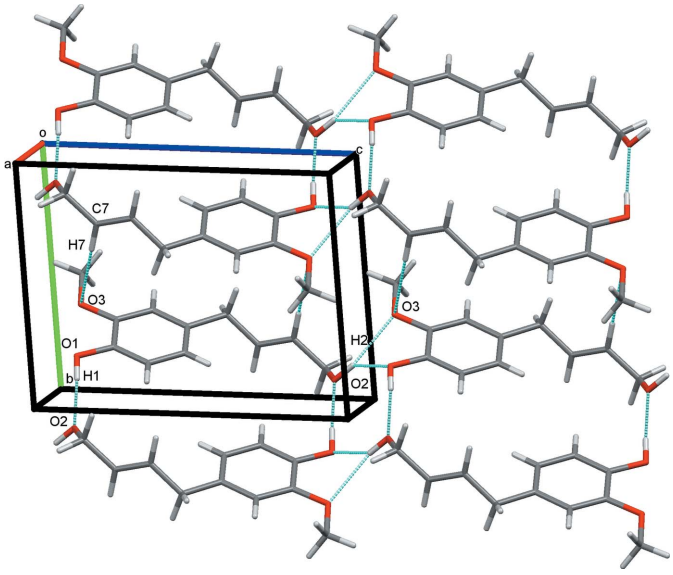

Figure 2
Crystal packing of the title compound, viewed along the *a* axis with hydrogen bonds drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.84	1.80 (1)	2.635 (2)	174 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{14}O_3$
M_r	194.22
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	200
a, b, c (Å)	5.7659 (11), 8.5396 (17), 10.804 (2)
α, β, γ (°)	81.579 (6), 88.020 (6), 71.167 (6)
V (Å^3)	498.03 (17)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.6 × 0.4 × 0.05
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9408, 1736, 1487
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.114, 1.07
No. of reflections	1736
No. of parameters	130
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.18

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

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full crystallographic data

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Crystal data

$C_{11}H_{14}O_3$
M_r = 194.22
Triclinic, $P\overline{1}$
a = 5.7659 (11) Å
b = 8.5396 (17) Å
c = 10.804 (2) Å
α = 81.579 (6)°
β = 88.020 (6)°
γ = 71.167 (6)°
V = 498.03 (17) Å^3
Z = 2
$F(000)$ = 208
D_x = 1.295 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3014 reflections
θ = 2.5–24.8°
μ = 0.09 mm^{-1}
T = 200 K
Plate, colorless
0.6 × 0.4 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
φ and ω scans
9408 measured reflections
1736 independent reflections
1487 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.0°, θ_{min} = 3.0°
h = −6→6
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.114
S = 1.07
1736 reflections
130 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.2128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.6301 (2)	0.80940 (16)	0.11536 (11)	0.0390 (4)

H1	0.6644	0.8981	0.1139	0.058*
O2	0.2842 (3)	0.90321 (18)	0.90202 (13)	0.0513 (4)
H2	0.3365	0.8367	0.9679	0.077*
O3	0.3545 (2)	0.62145 (15)	0.11605 (11)	0.0369 (3)
C1	0.5208 (3)	0.7770 (2)	0.22611 (16)	0.0306 (4)
C2	0.5516 (3)	0.8357 (2)	0.33455 (16)	0.0335 (4)
H2A	0.6563	0.9017	0.3353	0.040*
C3	0.4301 (3)	0.7991 (2)	0.44320 (16)	0.0336 (4)
H3	0.4542	0.8394	0.5176	0.040*
C4	0.2748 (3)	0.7049 (2)	0.44395 (16)	0.0304 (4)
C5	0.1392 (3)	0.6612 (2)	0.56028 (16)	0.0356 (4)
H5A	−0.0142	0.6465	0.5337	0.043*
H5B	0.2414	0.5527	0.6055	0.043*
C6	0.0766 (3)	0.7860 (2)	0.64830 (17)	0.0377 (5)
H6	−0.0171	0.8977	0.6168	0.045*
C7	0.1425 (4)	0.7521 (2)	0.76809 (18)	0.0402 (5)
H7	0.2404	0.6412	0.7990	0.048*
C8	0.0739 (4)	0.8755 (3)	0.85676 (19)	0.0467 (5)
H8A	−0.0388	0.9824	0.8144	0.056*
H8B	−0.0137	0.8345	0.9281	0.056*
C9	0.3699 (3)	0.6775 (2)	0.22712 (15)	0.0299 (4)
C10	0.2208 (4)	0.5064 (2)	0.11609 (19)	0.0418 (5)
H10A	0.2849	0.4120	0.1827	0.063*
H10B	0.2388	0.4653	0.0351	0.063*
H10C	0.0470	0.5630	0.1305	0.063*
C11	0.2463 (3)	0.6443 (2)	0.33454 (16)	0.0315 (4)
H11	0.1405	0.5792	0.3337	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0481 (8)	0.0432 (8)	0.0330 (7)	−0.0256 (6)	0.0103 (6)	−0.0055 (6)
O2	0.0763 (11)	0.0483 (9)	0.0362 (8)	−0.0328 (8)	−0.0110 (7)	0.0048 (6)
O3	0.0461 (8)	0.0439 (7)	0.0273 (7)	−0.0233 (6)	0.0004 (5)	−0.0059 (5)
C1	0.0304 (9)	0.0308 (9)	0.0289 (9)	−0.0100 (7)	0.0027 (7)	0.0003 (7)
C2	0.0335 (10)	0.0345 (9)	0.0370 (10)	−0.0173 (8)	0.0014 (8)	−0.0049 (8)
C3	0.0365 (10)	0.0357 (10)	0.0299 (9)	−0.0124 (8)	−0.0010 (7)	−0.0062 (7)
C4	0.0307 (9)	0.0286 (9)	0.0292 (9)	−0.0074 (7)	0.0002 (7)	−0.0005 (7)
C5	0.0383 (10)	0.0371 (10)	0.0311 (10)	−0.0144 (8)	0.0024 (8)	0.0009 (7)
C6	0.0399 (11)	0.0382 (10)	0.0352 (10)	−0.0145 (8)	0.0069 (8)	−0.0029 (8)
C7	0.0421 (11)	0.0381 (10)	0.0419 (11)	−0.0172 (9)	0.0061 (9)	−0.0017 (8)
C8	0.0560 (13)	0.0495 (12)	0.0371 (11)	−0.0218 (10)	0.0066 (9)	−0.0049 (9)
C9	0.0321 (9)	0.0283 (9)	0.0280 (9)	−0.0092 (7)	−0.0023 (7)	−0.0012 (7)
C10	0.0447 (11)	0.0472 (11)	0.0427 (11)	−0.0249 (9)	−0.0008 (9)	−0.0123 (9)
C11	0.0326 (10)	0.0319 (9)	0.0318 (9)	−0.0144 (8)	−0.0004 (7)	−0.0003 (7)

Geometric parameters (Å, º)

O1—H1	0.8400	C5—H5A	0.9900
O1—C1	1.367 (2)	C5—H5B	0.9900
O2—H2	0.8400	C5—C6	1.481 (3)
O2—C8	1.424 (3)	C6—H6	0.9500
O3—C9	1.371 (2)	C6—C7	1.325 (3)
O3—C10	1.431 (2)	C7—H7	0.9500
C1—C2	1.378 (3)	C7—C8	1.478 (3)
C1—C9	1.397 (2)	C8—H8A	0.9900
C2—H2A	0.9500	C8—H8B	0.9900
C2—C3	1.394 (2)	C9—C11	1.383 (2)
C3—H3	0.9500	C10—H10A	0.9800
C3—C4	1.383 (3)	C10—H10B	0.9800
C4—C5	1.521 (2)	C10—H10C	0.9800
C4—C11	1.392 (2)	C11—H11	0.9500
C1—O1—H1	109.5	C7—C6—H6	117.8
C8—O2—H2	109.5	C6—C7—H7	117.8
C9—O3—C10	116.99 (14)	C6—C7—C8	124.33 (19)
O1—C1—C2	124.05 (16)	C8—C7—H7	117.8
O1—C1—C9	116.82 (15)	O2—C8—C7	111.23 (18)
C2—C1—C9	119.13 (16)	O2—C8—H8A	109.4
C1—C2—H2A	119.8	O2—C8—H8B	109.4
C1—C2—C3	120.46 (16)	C7—C8—H8A	109.4
C3—C2—H2A	119.8	C7—C8—H8B	109.4
C2—C3—H3	119.6	H8A—C8—H8B	108.0
C4—C3—C2	120.74 (16)	O3—C9—C1	115.16 (15)
C4—C3—H3	119.6	O3—C9—C11	124.74 (16)
C3—C4—C5	122.59 (16)	C11—C9—C1	120.09 (16)
C3—C4—C11	118.57 (16)	O3—C10—H10A	109.5
C11—C4—C5	118.82 (15)	O3—C10—H10B	109.5
C4—C5—H5A	108.5	O3—C10—H10C	109.5
C4—C5—H5B	108.5	H10A—C10—H10B	109.5
H5A—C5—H5B	107.5	H10A—C10—H10C	109.5
C6—C5—C4	115.18 (15)	H10B—C10—H10C	109.5
C6—C5—H5A	108.5	C4—C11—H11	119.5
C6—C5—H5B	108.5	C9—C11—C4	120.96 (16)
C5—C6—H6	117.8	C9—C11—H11	119.5
C7—C6—C5	124.35 (18)		
O1—C1—C2—C3	178.96 (16)	C3—C4—C5—C6	30.2 (2)
O1—C1—C9—O3	1.3 (2)	C3—C4—C11—C9	−0.1 (3)
O1—C1—C9—C11	−177.74 (15)	C4—C5—C6—C7	−123.8 (2)
O3—C9—C11—C4	179.28 (15)	C5—C4—C11—C9	−178.60 (15)
C1—C2—C3—C4	−0.7 (3)	C5—C6—C7—C8	−178.11 (18)
C1—C9—C11—C4	−1.8 (3)	C6—C7—C8—O2	−116.2 (2)
C2—C1—C9—O3	−178.53 (15)	C9—C1—C2—C3	−1.2 (3)

C2—C1—C9—C11	2.4 (3)	C10—O3—C9—C1	174.23 (15)
C2—C3—C4—C5	179.76 (16)	C10—O3—C9—C11	−6.8 (2)
C2—C3—C4—C11	1.3 (3)	C11—C4—C5—C6	−151.41 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	1.80 (1)	2.635 (2)	174 (2)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.