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(*E*)-3-Hydroxy-5,5-dimethyl-2-(3-phenylprop-2-en-1-yl)cyclohex-2-en-1-one

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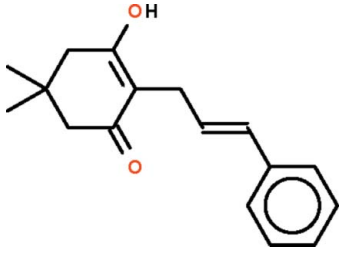
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.120; data-to-parameter ratio = 17.9.

Five of the atoms of the six-membered cyclohexene ring of the title compound, $C_{17}H_{20}O_2$, are essentially coplanar (r.m.s. deviation = 0.006 Å), with the sixth (the dimethylmethyl C atom) deviating from the mean plane of the five atoms by 0.610 (2) Å. This plane is nearly perpendicular to the cinnamyl portion, the two planes being aligned at 85.1 (1)°. Two molecules are linked by an O—H···O hydrogen bond about a center of inversion. The cyclohexene ring is disordered over two directly overlapping positions. As a result, the hydroxy group and the keto O atom cannot be distinguished from one another.

Related literature

For the synthesis, see: Gan *et al.* (2008).



Experimental

Crystal data

$C_{17}H_{20}O_2$
M_r = 256.33
Triclinic, $P\overline{1}$
a = 5.6480 (2) Å
b = 10.9077 (5) Å
c = 12.4762 (8) Å
α = 70.999 (5)°
β = 89.533 (4)°
γ = 75.783 (4)°
V = 702.31 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.977, T_{max} = 0.985
6734 measured reflections
3119 independent reflections
2554 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.120
S = 1.02
3119 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1^{i}	0.84	1.76	2.582 (2)	166
O2—H2···O2^{ii}	0.84	1.74	2.569 (2)	167

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the Iran National Science Foundation and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5605).

References

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Gan, K.-H., Jhong, C.-J. & Yang, S.-C. (2008). *Tetrahedron*, **64**, 1204–1212.
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Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2011). E**67**, o2337 [doi:10.1107/S160053681103220X]

(*E*)-3-Hydroxy-5,5-dimethyl-2-(3-phenylprop-2-en-1-yl)cyclohex-2-en-1-one

Afsaneh Zonouzi, Zakieh Izakiana and Seik Weng Ng

S1. Comment

Dimedone condenses with aromatic aldehydes. Cinnamaldehyde is the aldehyde in the present study. The compound $C_{17}H_{20}O_2$ (Scheme I, Fig. 1) possess a hydroxy as well as a ketonic unit; these interact by an O–H···O hydrogen bond to generate a hydrogen-bonded dimer (Table 1). The synthesis illustrates the direct activation of a C–O bond of a cyclic 1,3-dione to yield the *C*-alkylated product; an earlier report detailed the synthesis that is catalyzed by palladium compounds (Gan *et al.*, 2008). Five of the atoms of the six-membered cyclohexene ring of $C_{17}H_{20}O_2$ are coplanar, with the sixth (the dimethylmethyl carbon) deviating from the mean-plane of the five. This plane is perpendicular to the cinnamyl portion. Two molecules are linked by an O–H···O hydrogen bond about a center-of-inversion (Table 1).

S2. Experimental

To a stirred solution of dimedone (0.68 g, 5 mmol) and cinnamaldehyde (0.66 g, 5 mmol) in ethanol (10 ml) was added zinc chloride (1 mmol) along with a small amount of a primary amine as catalyst. The mixture was heated for 12 h. The product was purified by column chromatography on silica gel by using an ethyl acetate/*n*-hexane (1:3) solvent system. The compound was recrystallized from ethyl acetate to give colorless crystals (yield 70%).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The hydroxy H-atom is disordered over two positions, and the occupancy was assumed to be 0.5. The two half-occupancy atoms were already treated as riding [O—H 0.84 Å, U_{iso}(H) 1.5U_{eq}(O)].

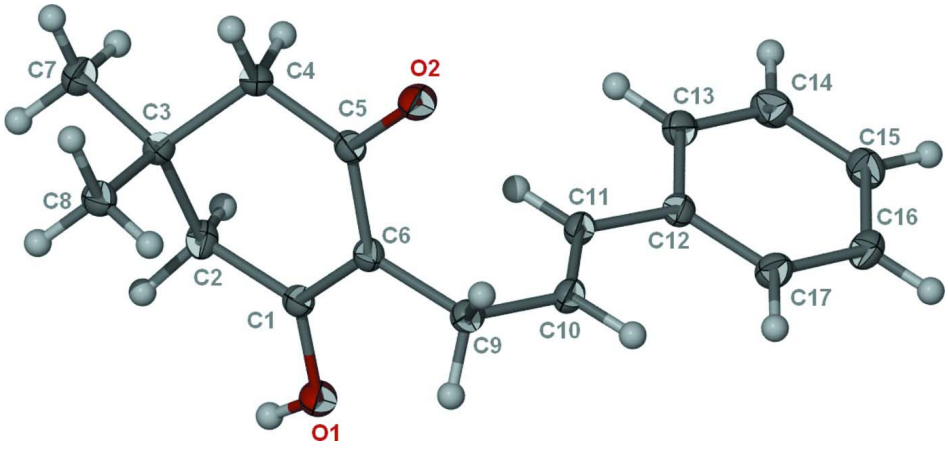

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{17}H_{20}O_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-3-Hydroxy-5,5-dimethyl-2-(3-phenylprop-2-en-1-yl)cyclohex-2-en-1-one

Crystal data

$C_{17}H_{20}O_2$
$M_r = 256.33$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.6480\ (2)$ Å
$b = 10.9077\ (5)$ Å
$c = 12.4762\ (8)$ Å
$\alpha = 70.999\ (5)°$
$\beta = 89.533\ (4)°$
$\gamma = 75.783\ (4)°$
$V = 702.31\ (6)$ Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.212$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3442 reflections
$\theta = 2.3–29.3°$
$\mu = 0.08$ mm^{-1}
$T = 100$ K
Block, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.977$, $T_{max} = 0.985$
6734 measured reflections
3119 independent reflections
2554 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -7\rightarrow7$
$k = -14\rightarrow14$
$l = -11\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.120$
$S = 1.02$
3119 reflections
174 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.1884P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.86917 (17)	0.62115 (9)	0.45112 (9)	0.0271 (3)	
H1	0.9361	0.5411	0.4900	0.041*	0.50
O2	0.19642 (18)	0.96431 (9)	0.45314 (9)	0.0290 (3)	
H2	0.0794	0.9797	0.4929	0.044*	0.50
C1	0.6812 (2)	0.66673 (13)	0.50012 (11)	0.0197 (3)	
C2	0.6155 (3)	0.57371 (13)	0.60766 (12)	0.0233 (3)	
H2A	0.7686	0.5134	0.6523	0.028*	
H2B	0.5231	0.5172	0.5873	0.028*	
C3	0.4630 (2)	0.64378 (13)	0.68296 (12)	0.0205 (3)	
C4	0.2525 (2)	0.75600 (14)	0.60738 (12)	0.0232 (3)	
H4A	0.1305	0.7150	0.5857	0.028*	
H4B	0.1708	0.8122	0.6521	0.028*	
C5	0.3312 (2)	0.84458 (13)	0.50111 (11)	0.0205 (3)	
C6	0.5413 (2)	0.79848 (13)	0.45053 (11)	0.0204 (3)	
C7	0.3597 (3)	0.54314 (14)	0.77432 (13)	0.0279 (3)	
H7A	0.2623	0.5885	0.8223	0.042*	
H7B	0.4949	0.4707	0.8213	0.042*	
H7C	0.2558	0.5056	0.7380	0.042*	
C8	0.6225 (2)	0.70218 (14)	0.74076 (12)	0.0246 (3)	
H8A	0.5235	0.7468	0.7888	0.037*	
H8B	0.6887	0.7674	0.6828	0.037*	
H8C	0.7578	0.6298	0.7878	0.037*	
C9	0.6047 (2)	0.88745 (13)	0.34042 (12)	0.0218 (3)	
H9A	0.7842	0.8612	0.3361	0.026*	
H9B	0.5595	0.9811	0.3404	0.026*	
C10	0.4806 (2)	0.88266 (13)	0.23604 (12)	0.0232 (3)	
H10	0.5208	0.9356	0.1648	0.028*	
C11	0.3217 (2)	0.81262 (13)	0.23315 (12)	0.0223 (3)	
H11	0.2803	0.7602	0.3043	0.027*	
C12	0.2022 (2)	0.80775 (13)	0.13049 (12)	0.0218 (3)	
C13	0.0192 (2)	0.74014 (14)	0.14118 (13)	0.0257 (3)	
H13	−0.0262	0.6972	0.2147	0.031*	
C14	−0.0978 (3)	0.73432 (15)	0.04654 (14)	0.0303 (4)	
H14	−0.2210	0.6869	0.0557	0.036*	
C15	−0.0356 (3)	0.79759 (15)	−0.06144 (14)	0.0294 (3)	
H15	−0.1168	0.7944	−0.1265	0.035*	
C16	0.1453 (3)	0.86525 (14)	−0.07378 (13)	0.0275 (3)	
H16	0.1881	0.9093	−0.1476	0.033*	
C17	0.2645 (3)	0.86914 (14)	0.02109 (13)	0.0256 (3)	
H17	0.3910	0.9144	0.0116	0.031*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0254 (5)	0.0248 (5)	0.0264 (6)	0.0015 (4)	0.0035 (4)	−0.0083 (4)
O2	0.0268 (5)	0.0248 (5)	0.0275 (6)	0.0032 (4)	0.0059 (4)	−0.0055 (4)
C1	0.0183 (6)	0.0232 (6)	0.0187 (7)	−0.0033 (5)	0.0006 (5)	−0.0100 (6)
C2	0.0290 (7)	0.0201 (6)	0.0191 (7)	−0.0029 (6)	0.0011 (5)	−0.0065 (5)
C3	0.0201 (6)	0.0218 (6)	0.0188 (7)	−0.0050 (5)	0.0027 (5)	−0.0059 (5)
C4	0.0189 (6)	0.0276 (7)	0.0210 (8)	−0.0039 (6)	0.0022 (5)	−0.0067 (6)
C5	0.0190 (6)	0.0216 (6)	0.0197 (7)	−0.0021 (5)	−0.0008 (5)	−0.0076 (5)
C6	0.0208 (6)	0.0225 (6)	0.0179 (7)	−0.0045 (5)	0.0004 (5)	−0.0075 (5)
C7	0.0292 (7)	0.0281 (7)	0.0236 (8)	−0.0078 (6)	0.0044 (6)	−0.0047 (6)
C8	0.0226 (7)	0.0296 (7)	0.0226 (8)	−0.0047 (6)	0.0006 (5)	−0.0116 (6)
C9	0.0215 (6)	0.0202 (6)	0.0220 (8)	−0.0041 (5)	0.0030 (5)	−0.0056 (5)
C10	0.0249 (7)	0.0237 (7)	0.0180 (7)	−0.0047 (6)	0.0038 (5)	−0.0040 (5)
C11	0.0228 (6)	0.0225 (6)	0.0174 (7)	−0.0026 (5)	0.0028 (5)	−0.0036 (5)
C12	0.0214 (6)	0.0204 (6)	0.0204 (7)	−0.0008 (5)	0.0016 (5)	−0.0061 (5)
C13	0.0242 (7)	0.0239 (7)	0.0247 (8)	−0.0046 (6)	0.0017 (6)	−0.0035 (6)
C14	0.0257 (7)	0.0306 (7)	0.0354 (9)	−0.0109 (6)	0.0003 (6)	−0.0093 (7)
C15	0.0286 (7)	0.0323 (8)	0.0288 (9)	−0.0058 (6)	−0.0025 (6)	−0.0134 (7)
C16	0.0319 (7)	0.0296 (7)	0.0216 (8)	−0.0076 (6)	0.0054 (6)	−0.0098 (6)
C17	0.0253 (7)	0.0289 (7)	0.0250 (8)	−0.0106 (6)	0.0047 (6)	−0.0095 (6)

Geometric parameters (Å, º)

O1—C1	1.2946 (16)	C8—H8B	0.9800
O1—H1	0.8400	C8—H8C	0.9800
O2—C5	1.2858 (16)	C9—C10	1.506 (2)
O2—H2	0.8400	C9—H9A	0.9900
C1—C6	1.3941 (18)	C9—H9B	0.9900
C1—C2	1.5026 (19)	C10—C11	1.3212 (19)
C2—C3	1.5296 (19)	C10—H10	0.9500
C2—H2A	0.9900	C11—C12	1.475 (2)
C2—H2B	0.9900	C11—H11	0.9500
C3—C8	1.5260 (19)	C12—C13	1.3922 (19)
C3—C7	1.5276 (19)	C12—C17	1.396 (2)
C3—C4	1.5339 (17)	C13—C14	1.385 (2)
C4—C5	1.5020 (19)	C13—H13	0.9500
C4—H4A	0.9900	C14—C15	1.386 (2)
C4—H4B	0.9900	C14—H14	0.9500
C5—C6	1.4002 (18)	C15—C16	1.381 (2)
C6—C9	1.5005 (19)	C15—H15	0.9500
C7—H7A	0.9800	C16—C17	1.384 (2)
C7—H7B	0.9800	C16—H16	0.9500
C7—H7C	0.9800	C17—H17	0.9500
C8—H8A	0.9800		
C1—O1—H1	109.5	C3—C8—H8B	109.5

C5—O2—H2	109.5	H8A—C8—H8B	109.5
O1—C1—C6	119.59 (12)	C3—C8—H8C	109.5
O1—C1—C2	118.58 (11)	H8A—C8—H8C	109.5
C6—C1—C2	121.80 (11)	H8B—C8—H8C	109.5
C1—C2—C3	114.71 (11)	C6—C9—C10	114.36 (11)
C1—C2—H2A	108.6	C6—C9—H9A	108.7
C3—C2—H2A	108.6	C10—C9—H9A	108.7
C1—C2—H2B	108.6	C6—C9—H9B	108.7
C3—C2—H2B	108.6	C10—C9—H9B	108.7
H2A—C2—H2B	107.6	H9A—C9—H9B	107.6
C8—C3—C7	108.85 (12)	C11—C10—C9	126.85 (13)
C8—C3—C2	110.02 (11)	C11—C10—H10	116.6
C7—C3—C2	109.82 (11)	C9—C10—H10	116.6
C8—C3—C4	110.21 (11)	C10—C11—C12	126.52 (13)
C7—C3—C4	109.84 (11)	C10—C11—H11	116.7
C2—C3—C4	108.10 (11)	C12—C11—H11	116.7
C5—C4—C3	114.10 (11)	C13—C12—C17	117.74 (13)
C5—C4—H4A	108.7	C13—C12—C11	119.70 (13)
C3—C4—H4A	108.7	C17—C12—C11	122.56 (12)
C5—C4—H4B	108.7	C14—C13—C12	121.20 (14)
C3—C4—H4B	108.7	C14—C13—H13	119.4
H4A—C4—H4B	107.6	C12—C13—H13	119.4
O2—C5—C6	119.25 (12)	C13—C14—C15	120.14 (14)
O2—C5—C4	118.89 (11)	C13—C14—H14	119.9
C6—C5—C4	121.80 (11)	C15—C14—H14	119.9
C1—C6—C5	119.10 (12)	C16—C15—C14	119.51 (14)
C1—C6—C9	120.71 (12)	C16—C15—H15	120.2
C5—C6—C9	120.07 (11)	C14—C15—H15	120.2
C3—C7—H7A	109.5	C15—C16—C17	120.19 (14)
C3—C7—H7B	109.5	C15—C16—H16	119.9
H7A—C7—H7B	109.5	C17—C16—H16	119.9
C3—C7—H7C	109.5	C16—C17—C12	121.20 (13)
H7A—C7—H7C	109.5	C16—C17—H17	119.4
H7B—C7—H7C	109.5	C12—C17—H17	119.4
C3—C8—H8A	109.5		

O1—C1—C2—C3	157.55 (12)	O2—C5—C6—C9	−1.2 (2)
C6—C1—C2—C3	−24.44 (18)	C4—C5—C6—C9	176.16 (12)
C1—C2—C3—C8	−72.89 (14)	C1—C6—C9—C10	90.63 (15)
C1—C2—C3—C7	167.32 (11)	C5—C6—C9—C10	−85.41 (15)
C1—C2—C3—C4	47.50 (15)	C6—C9—C10—C11	2.01 (19)
C8—C3—C4—C5	71.88 (15)	C9—C10—C11—C12	−179.55 (12)
C7—C3—C4—C5	−168.20 (12)	C10—C11—C12—C13	−173.29 (13)
C2—C3—C4—C5	−48.39 (15)	C10—C11—C12—C17	6.5 (2)
C3—C4—C5—O2	−156.07 (12)	C17—C12—C13—C14	−0.2 (2)
C3—C4—C5—C6	26.60 (18)	C11—C12—C13—C14	179.59 (12)
O1—C1—C6—C5	176.74 (12)	C12—C13—C14—C15	−0.7 (2)
C2—C1—C6—C5	−1.3 (2)	C13—C14—C15—C16	0.6 (2)

O1—C1—C6—C9	0.7 (2)	C14—C15—C16—C17	0.4 (2)
C2—C1—C6—C9	−177.34 (12)	C15—C16—C17—C12	−1.3 (2)
O2—C5—C6—C1	−177.27 (12)	C13—C12—C17—C16	1.2 (2)
C4—C5—C6—C1	0.1 (2)	C11—C12—C17—C16	−178.59 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1^{i}	0.84	1.76	2.582 (2)	166
O2—H2···O2^{ii}	0.84	1.74	2.569 (2)	167

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1.