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## Structure Reports

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1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one<sup>1</sup>

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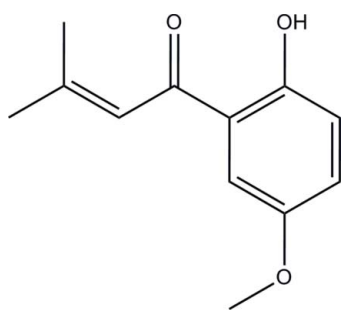
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Key indicators: single-crystal X-ray study;  $T = 100$  K,  $P = 0.0$  kPa; mean  $\sigma(\text{C}-\text{C}) = 0.001$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.127; data-to-parameter ratio = 26.5.

The title compound,  $\text{C}_{12}\text{H}_{14}\text{O}_3$ , is a natural product derived from the medium-sized hawthorn *Crataegus persimilis* ('*pruni-prunifolia*'). The mean plane of the butene moiety is twisted by  $13.27(7)^\circ$  with respect to the that of the dioxobenzaldehyde moiety. There is an intramolecular hydrogen bond between the hydroxyl group and the carbonyl O atom.

## Related literature

For isolation from plant material, see: Castro *et al.* (1989). For the synthesis, see: Camps *et al.* (1985). For photolysis to form 4-chromanones, see: Primo *et al.* (1982). For a related structure, see: Zeller *et al.* (2010).



## Experimental

## Crystal data

$\text{C}_{12}\text{H}_{14}\text{O}_3$   
 $M_r = 206.23$   
 Monoclinic,  $P2_1/c$   
 $a = 14.027(3)$  Å

$b = 5.816(1)$  Å  
 $c = 12.829(3)$  Å  
 $\beta = 91.409(8)^\circ$   
 $V = 1046.3(4)$  Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>

$T = 100$  K  
 $0.45 \times 0.37 \times 0.23$  mm

## Data collection

Nonius KappaCCD diffractometer  
 Absorption correction: multi-scan  
 (HKL SCALEPACK;  
 Otwinowski & Minor 1997)  
 $T_{\min} = 0.959$ ,  $T_{\max} = 0.979$

6425 measured reflections  
 3784 independent reflections  
 3179 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.018$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.127$   
 $S = 1.04$   
 3784 reflections  
 143 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.43$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                         | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|----------|-------------|-------------|---------------|
| $\text{O2}-\text{H2A}\cdots\text{O3}$ | 0.87 (2) | 1.74 (2)    | 2.523 (1)   | 149 (2)       |

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SIR2002 (Burla *et al.*, 2003); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX publication routines (Farrugia, 1999).

The purchase of the diffractometer was made possible by grant No. LEQSF (1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2405).

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<sup>1</sup> CAS Registry 84346–78–1.

## supporting information

*Acta Cryst.* (2012). E68, o3126 [doi:10.1107/S1600536812042158]

**1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one**

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**S1. Comment**

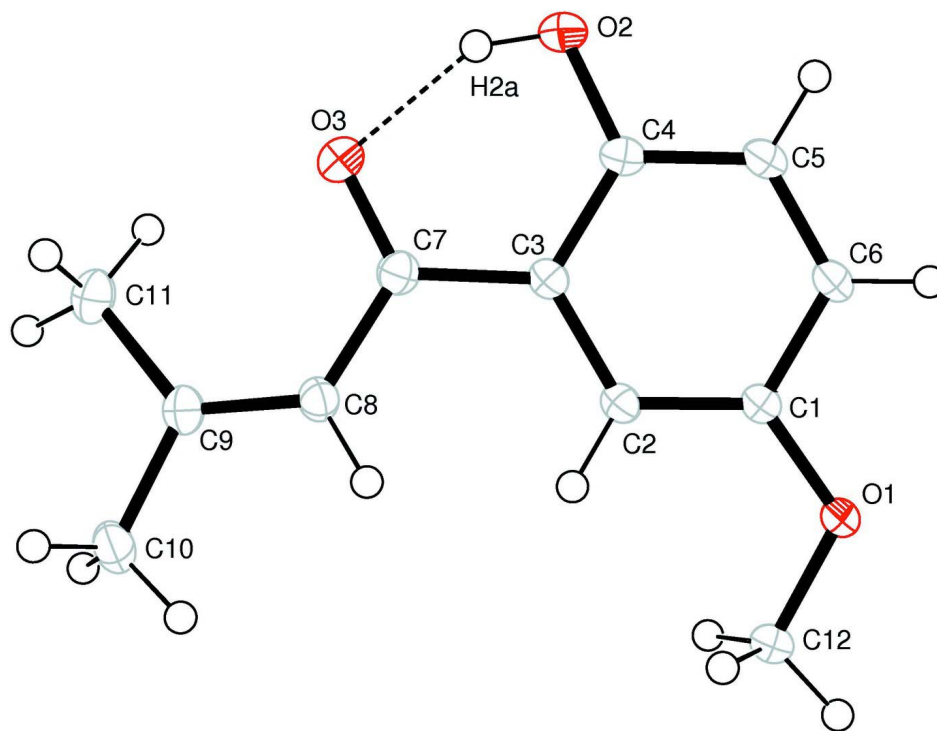
The structure of title compound **I** can be described in terms of four planar moieties as defined by their constituent non-hydrogen atoms. The phenyl ring and three atoms bonded to it define the main molecular plane, with mean deviation of the defining atoms of  $\delta_{\text{r.m.s.}} = 0.0145$  (6) Å. With respect to this molecular plane, the mean plane of the carbonyl group (four atoms,  $\delta_{\text{r.m.s.}} = 0.0044$  (4) Å) and the plane of the methoxy group (three atoms) have dihedral angles of 2.50 (6)° and 4.33 (6)° respectively, while the mean plane of the butene moiety (four atoms,  $\delta_{\text{r.m.s.}} = 0.0018$  (4) Å) has dihedral angle 13.27 (7)°.

**S2. Experimental**

Compound **I** was isolated as a natural product (Castro *et al.*, 1989). It has also been synthesized (Camps *et al.*, 1985). Suitable crystals were formed by very slow evaporation of a hexane solution over a period of three years.

**S3. Refinement**

The positional and isotropic displacement parameters of hydroxyl atom H2A were refined independently. All other H atoms were placed in calculated positions, guided by difference maps, and refined as riding. Torsional parameters for the three methyl groups were refined, with C—H = 0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ , while H atoms attached to *sp*<sup>2</sup> C atoms have C—H = 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

**Figure 1**

Molecular structure of (I) with displacement ellipsoids at the 50% probability level.

### 1-(2-Hydroxy-5-methoxyphenyl)-3-methylbut-2-en-1-one

#### Crystal data

$C_{12}H_{14}O_3$   
 $M_r = 206.23$   
 Monoclinic,  $P2_1/c$   
 Hall symbol: -P 2ybc  
 $a = 14.027$  (3) Å  
 $b = 5.816$  (1) Å  
 $c = 12.829$  (3) Å  
 $\beta = 91.409$  (8)°  
 $V = 1046.3$  (4) Å<sup>3</sup>  
 $Z = 4$

$F(000) = 440$   
 $D_x = 1.309$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
 Cell parameters from 3442 reflections  
 $\theta = 2.5$ – $32.6$ °  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 100$  K  
 Fragment, yellow  
 $0.45 \times 0.37 \times 0.23$  mm

#### Data collection

Nonius KappaCCD  
 diffractometer  
 Radiation source: sealed tube  
 Horizontally mounted graphite crystal  
 monochromator  
 Detector resolution: 9 pixels mm<sup>-1</sup>  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (HKL SCALEPACK; Otwinowski & Minor  
 1997)

$T_{\min} = 0.959$ ,  $T_{\max} = 0.979$   
 6425 measured reflections  
 3784 independent reflections  
 3179 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.018$   
 $\theta_{\max} = 32.6$ °,  $\theta_{\min} = 3.2$ °  
 $h = -21 \rightarrow 21$   
 $k = -8 \rightarrow 7$   
 $l = -19 \rightarrow 19$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.127$   
 $S = 1.04$   
 3784 reflections  
 143 parameters  
 0 restraints  
 0 constraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.2297P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

|      | <i>x</i>    | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|--------------|-------------|----------------------------------|
| C1   | 0.38220 (6) | 1.03873 (14) | 0.88580 (6) | 0.01713 (16)                     |
| C2   | 0.31710 (6) | 0.89827 (14) | 0.93398 (6) | 0.01652 (15)                     |
| H2   | 0.3003      | 0.9281       | 1.0040      | 0.020*                           |
| C3   | 0.27530 (6) | 0.71019 (14) | 0.87973 (6) | 0.01641 (15)                     |
| C4   | 0.30238 (6) | 0.66752 (15) | 0.77617 (6) | 0.01796 (16)                     |
| C5   | 0.36779 (6) | 0.81313 (15) | 0.72851 (6) | 0.01992 (17)                     |
| H5   | 0.3853      | 0.7853       | 0.6586      | 0.024*                           |
| C6   | 0.40689 (6) | 0.99610 (16) | 0.78231 (6) | 0.01999 (17)                     |
| H6   | 0.4509      | 1.0944       | 0.7491      | 0.024*                           |
| C7   | 0.20366 (6) | 0.55840 (14) | 0.92711 (7) | 0.01901 (16)                     |
| C8   | 0.17084 (6) | 0.60621 (15) | 1.03279 (7) | 0.01893 (16)                     |
| H8   | 0.1897      | 0.7485       | 1.0632      | 0.023*                           |
| C9   | 0.11620 (6) | 0.46622 (15) | 1.09083 (7) | 0.01933 (16)                     |
| C10  | 0.08404 (7) | 0.54666 (17) | 1.19537 (7) | 0.02467 (19)                     |
| H10A | 0.1069      | 0.7037       | 1.2079      | 0.037*                           |
| H10B | 0.1100      | 0.4446       | 1.2498      | 0.037*                           |
| H10C | 0.0142      | 0.5442       | 1.1967      | 0.037*                           |
| C11  | 0.08255 (7) | 0.23100 (16) | 1.05970 (8) | 0.02593 (19)                     |
| H11A | 0.0197      | 0.2424       | 1.0251      | 0.039*                           |
| H11B | 0.0781      | 0.1342       | 1.1219      | 0.039*                           |
| H11C | 0.1279      | 0.1625       | 1.0117      | 0.039*                           |
| C12  | 0.40983 (6) | 1.26466 (15) | 1.03865 (7) | 0.02072 (17)                     |
| H12A | 0.3425      | 1.3041       | 1.0465      | 0.031*                           |
| H12B | 0.4497      | 1.3925       | 1.0637      | 0.031*                           |
| H12C | 0.4249      | 1.1263       | 1.0794      | 0.031*                           |
| O1   | 0.42786 (5) | 1.22263 (11) | 0.93148 (5) | 0.02247 (15)                     |
| O2   | 0.26717 (5) | 0.49087 (12) | 0.71825 (5) | 0.02388 (15)                     |
| H2A  | 0.2287 (12) | 0.416 (3)    | 0.7580 (14) | 0.052 (5)*                       |

O3                    0.17042 (5)                    0.39337 (13)                    0.87547 (6)                    0.02755 (16)

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C1  | 0.0188 (3) | 0.0184 (3) | 0.0143 (3) | -0.0008 (3) | 0.0011 (3)  | 0.0000 (3)  |
| C2  | 0.0168 (3) | 0.0186 (3) | 0.0142 (3) | 0.0004 (3)  | 0.0009 (3)  | 0.0004 (3)  |
| C3  | 0.0166 (3) | 0.0175 (3) | 0.0152 (3) | 0.0007 (3)  | 0.0007 (3)  | 0.0006 (3)  |
| C4  | 0.0195 (3) | 0.0188 (3) | 0.0156 (3) | 0.0018 (3)  | -0.0008 (3) | -0.0011 (3) |
| C5  | 0.0230 (4) | 0.0234 (4) | 0.0134 (3) | -0.0002 (3) | 0.0018 (3)  | -0.0006 (3) |
| C6  | 0.0226 (4) | 0.0235 (4) | 0.0140 (3) | -0.0026 (3) | 0.0031 (3)  | 0.0010 (3)  |
| C7  | 0.0181 (3) | 0.0192 (4) | 0.0198 (4) | -0.0002 (3) | 0.0004 (3)  | 0.0002 (3)  |
| C8  | 0.0179 (3) | 0.0191 (3) | 0.0199 (4) | -0.0006 (3) | 0.0027 (3)  | 0.0002 (3)  |
| C9  | 0.0157 (3) | 0.0199 (4) | 0.0224 (4) | 0.0013 (3)  | 0.0007 (3)  | 0.0035 (3)  |
| C10 | 0.0241 (4) | 0.0267 (4) | 0.0236 (4) | -0.0015 (3) | 0.0071 (3)  | 0.0029 (3)  |
| C11 | 0.0265 (4) | 0.0208 (4) | 0.0305 (5) | -0.0040 (3) | 0.0023 (4)  | 0.0039 (3)  |
| C12 | 0.0243 (4) | 0.0223 (4) | 0.0157 (3) | -0.0029 (3) | 0.0026 (3)  | -0.0027 (3) |
| O1  | 0.0286 (3) | 0.0238 (3) | 0.0153 (3) | -0.0094 (2) | 0.0052 (2)  | -0.0030 (2) |
| O2  | 0.0301 (3) | 0.0225 (3) | 0.0190 (3) | -0.0042 (3) | 0.0017 (3)  | -0.0051 (2) |
| O3  | 0.0317 (4) | 0.0260 (3) | 0.0251 (3) | -0.0101 (3) | 0.0034 (3)  | -0.0049 (3) |

*Geometric parameters (Å, °)*

|          |             |               |             |
|----------|-------------|---------------|-------------|
| C1—O1    | 1.3708 (10) | C8—H8         | 0.95        |
| C1—C2    | 1.3824 (11) | C9—C11        | 1.4982 (13) |
| C1—C6    | 1.4023 (12) | C9—C10        | 1.5003 (13) |
| C2—C3    | 1.4159 (11) | C10—H10A      | 0.98        |
| C2—H2    | 0.95        | C10—H10B      | 0.98        |
| C3—C4    | 1.4127 (12) | C10—H10C      | 0.98        |
| C3—C7    | 1.4795 (12) | C11—H11A      | 0.98        |
| C4—O2    | 1.3540 (10) | C11—H11B      | 0.98        |
| C4—C5    | 1.4002 (12) | C11—H11C      | 0.98        |
| C5—C6    | 1.3751 (12) | C12—O1        | 1.4251 (11) |
| C5—H5    | 0.95        | C12—H12A      | 0.98        |
| C6—H6    | 0.95        | C12—H12B      | 0.98        |
| C7—O3    | 1.2499 (11) | C12—H12C      | 0.98        |
| C7—C8    | 1.4690 (12) | O2—H2A        | 0.870 (18)  |
| C8—C9    | 1.3538 (12) |               |             |
| O1—C1—C2 | 125.22 (7)  | C8—C9—C11     | 125.55 (8)  |
| O1—C1—C6 | 114.77 (7)  | C8—C9—C10     | 119.40 (8)  |
| C2—C1—C6 | 120.00 (8)  | C11—C9—C10    | 115.05 (8)  |
| C1—C2—C3 | 120.46 (7)  | C9—C10—H10A   | 109.5       |
| C1—C2—H2 | 119.8       | C9—C10—H10B   | 109.5       |
| C3—C2—H2 | 119.8       | H10A—C10—H10B | 109.5       |
| C4—C3—C2 | 118.71 (7)  | C9—C10—H10C   | 109.5       |
| C4—C3—C7 | 118.86 (7)  | H10A—C10—H10C | 109.5       |
| C2—C3—C7 | 122.43 (7)  | H10B—C10—H10C | 109.5       |

|             |             |               |             |
|-------------|-------------|---------------|-------------|
| O2—C4—C5    | 116.96 (8)  | C9—C11—H11A   | 109.5       |
| O2—C4—C3    | 123.13 (8)  | C9—C11—H11B   | 109.5       |
| C5—C4—C3    | 119.91 (8)  | H11A—C11—H11B | 109.5       |
| C6—C5—C4    | 120.43 (8)  | C9—C11—H11C   | 109.5       |
| C6—C5—H5    | 119.8       | H11A—C11—H11C | 109.5       |
| C4—C5—H5    | 119.8       | H11B—C11—H11C | 109.5       |
| C5—C6—C1    | 120.48 (8)  | O1—C12—H12A   | 109.5       |
| C5—C6—H6    | 119.8       | O1—C12—H12B   | 109.5       |
| C1—C6—H6    | 119.8       | H12A—C12—H12B | 109.5       |
| O3—C7—C8    | 120.88 (8)  | O1—C12—H12C   | 109.5       |
| O3—C7—C3    | 119.26 (8)  | H12A—C12—H12C | 109.5       |
| C8—C7—C3    | 119.85 (7)  | H12B—C12—H12C | 109.5       |
| C9—C8—C7    | 126.04 (8)  | C1—O1—C12     | 117.01 (7)  |
| C9—C8—H8    | 117         | C4—O2—H2A     | 106.4 (12)  |
| C7—C8—H8    | 117         |               |             |
| O1—C1—C2—C3 | -178.95 (8) | C2—C1—C6—C5   | -0.92 (13)  |
| C6—C1—C2—C3 | 0.33 (13)   | C4—C3—C7—O3   | 1.59 (12)   |
| C1—C2—C3—C4 | 0.82 (12)   | C2—C3—C7—O3   | -179.06 (8) |
| C1—C2—C3—C7 | -178.54 (8) | C4—C3—C7—C8   | -176.96 (7) |
| C2—C3—C4—O2 | 179.35 (7)  | C2—C3—C7—C8   | 2.39 (12)   |
| C7—C3—C4—O2 | -1.28 (12)  | O3—C7—C8—C9   | 11.11 (14)  |
| C2—C3—C4—C5 | -1.39 (12)  | C3—C7—C8—C9   | -170.37 (8) |
| C7—C3—C4—C5 | 177.99 (8)  | C7—C8—C9—C11  | 2.89 (14)   |
| O2—C4—C5—C6 | -179.87 (8) | C7—C8—C9—C10  | -176.50 (8) |
| C3—C4—C5—C6 | 0.82 (13)   | C2—C1—O1—C12  | 3.14 (12)   |
| C4—C5—C6—C1 | 0.34 (13)   | C6—C1—O1—C12  | -176.18 (8) |
| O1—C1—C6—C5 | 178.43 (8)  |               |             |

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> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| O2—H2A...O3             | 0.87 (2)    | 1.74 (2)      | 2.523 (1)             | 149 (2)                 |