

(E)-1-(2,4-Dihydroxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one monohydrate

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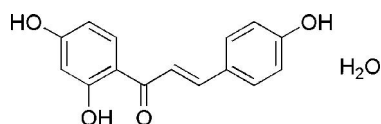
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Key indicators: single-crystal X-ray study; *T* = 298 K; mean σ (C–C) = 0.003 Å; *R* factor = 0.057; *wR* factor = 0.144; data-to-parameter ratio = 13.4.

In the title compound, C₁₅H₁₂O₄·H₂O, the two benzene rings are not coplanar, making a dihedral angle of 7.24 (16)°. An intramolecular hydroxy–carbonyl O–H···O hydrogen bond occurs. In the crystal, four intermolecular O–H···O hydrogen bonds involving the hydroxy residues, the carbonyl group and the water molecule lead to the formation of a three-dimensional network. The supramolecular structure is further stabilized by weak C–H···O interactions.

Related literature

For the biological activity of the title compound, see: Jang *et al.* (2008); Liu *et al.* (2008). For a related structure, see: Ma *et al.* (2005).



Experimental

Crystal data

C₁₅H₁₂O₄·H₂O
M_r = 274.26
Monoclinic, *P*2₁/*c*
a = 11.489 (2) Å
b = 9.5903 (17) Å
c = 12.498 (2) Å
 β = 103.649 (3)°

V = 1338.2 (4) Å³
Z = 4
Mo *K*α radiation
 μ = 0.10 mm⁻¹
T = 298 K
0.12 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
*T*_{min} = 0.988, *T*_{max} = 0.990
8297 measured reflections
2625 independent reflections
2115 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.031

Refinement

R [*F*² > 2σ(*F*²)] = 0.057
wR (*F*²) = 0.144
S = 1.09
2625 reflections
196 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}}$ = 0.33 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.19 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>
O1–H1A···O3	0.87 (2)	1.74 (2)	2.530 (2)	150 (3)
O2–H2A···O5 ⁱ	0.82 (2)	1.83 (2)	2.644 (3)	175 (4)
O4–H4A···O3 ⁱⁱ	0.83 (2)	1.95 (2)	2.776 (2)	175 (4)
O5–H5A···O1 ⁱⁱⁱ	0.84 (2)	1.99 (2)	2.802 (2)	164 (3)
O5–H5B···O4 ^{iv}	0.84 (2)	1.97 (2)	2.785 (3)	165 (3)
C9–H9···O4 ^v	0.93	2.56	3.402 (3)	151

Symmetry codes: (i) *x*, -*y* + ½, *z* + ½; (ii) -*x* + 1, *y* - ½, -*z* - ½; (iii) -*x*, -*y* + 2, -*z*; (iv) -*x* + 1, -*y* + 1, -*z*; (v) -*x* + 1, *y* + ½, -*z* - ½.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2004).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc. Madison, Wisconsin, USA.
Jang, E. Y., Choe, E. S., Hwang, M., Kim, S. C., Lee, J. R., Kim, S. G., Jeon, J. P., Buono, R. J. & Yang, C. H. (2008). *Eur. J. Pharmacol.* **587**, 124–128.
Liu, B., Yang, J., Wen, Q. S. & Li, Y. (2008). *Eur. J. Pharmacol.* **587**, 257–266
Ma, C.-J., Li, G.-S., Zhang, D.-L., Liu, K. & Fan, X. (2005). *J. Chromatogr. A*, **1078**, 188–192.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

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(*E*)-1-(2,4-Dihydroxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one monohydrate

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Comment

The title compound exhibits many biological activities such as tracheal relaxation effects (Liu *et al.*, 2008) and suppressing cocaine-induced extracellular dopamine release (Jang *et al.*, 2008).

One (*E*)-1-(2,4-Dihydroxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one molecule bears one crystalline water molecule (Fig.1). In the molecule, the two benzene rings are not coplanar, the dihedral angle being $7.24(16)^\circ$. The structure displays O—H \cdots O and C—H \cdots O hydrogen bonding (Table 1 and Fig. 2).

Experimental

2, 4-dihydroxyacetophenone (7.6 g, 0.05 mol) and 4-hydroxybenzaldehyde (8.54 g, 0.07 mol) were dissolved in diglycol (25 ml). Then 40% aq. KOH (50 ml) was added, and the reaction mixture was vigorously stirred under nitrogen atmosphere at 333 K for 2 h. The progress of the reaction was monitored by thin- layer chromatography (Si gel, developing solvent $V(\text{ethyl acetate})/V(\text{benzene}) = 1:2$). The mixture was cooled to room temperature and 1:1 (*v/v*) hydrochloric acid was added to acidize the mixture to pH=3 and a solid was obtained. After crystallized by ethanol-water, crystalline yellow needles were obtained, m.p. 472.5–474.2 K.

Refinement

All the carbon-bounded hydrogen atoms were located at their ideal positions with the C—H=0.93 Å and $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$. All the hydrogen atoms bonded to the oxygen atoms were located from the difference maps and refined with the restraints of O—H=0.82 (1) Å and $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$.

Figures

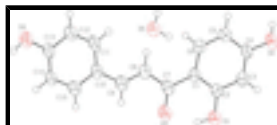


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

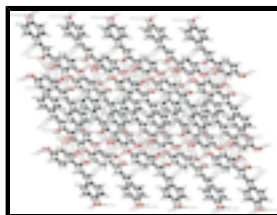


Fig. 2. The crystal packing for (I), with O—H \cdots O and C—H \cdots O interactions shown as dashed lines.

(E)-1-(2,4-Dihydroxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one monohydrate

Crystal data

$C_{15}H_{12}O_4 \cdot H_2O$	$F(000) = 576$
$M_r = 274.26$	$D_x = 1.361 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 2288 reflections
$a = 11.489 (2) \text{ \AA}$	$\theta = 2.7\text{--}25.3^\circ$
$b = 9.5903 (17) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 12.498 (2) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 103.649 (3)^\circ$	Block, yellow
$V = 1338.2 (4) \text{ \AA}^3$	$0.12 \times 0.10 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2625 independent reflections
Radiation source: fine-focus sealed tube graphite	2115 reflections with $I > 2\sigma(I)$
phi and ω scans	$R_{\text{int}} = 0.031$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 2.7^\circ$
$T_{\text{min}} = 0.988$, $T_{\text{max}} = 0.990$	$h = -14 \rightarrow 14$
8297 measured reflections	$k = -11 \rightarrow 9$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.057$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.144$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.499P]$
2625 reflections	where $P = (F_o^2 + 2F_c^2)/3$
196 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
5 restraints	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.19653 (18)	1.0001 (2)	0.07068 (17)	0.0402 (5)
C2	0.10947 (18)	1.1026 (2)	0.07420 (17)	0.0412 (5)
C3	0.0605 (2)	1.1156 (2)	0.16459 (19)	0.0475 (6)
H3	0.0037	1.1844	0.1655	0.057*
C4	0.0950 (2)	1.0275 (2)	0.25329 (18)	0.0452 (5)
C5	0.1818 (2)	0.9256 (3)	0.25306 (19)	0.0500 (6)
H5	0.2061	0.8663	0.3131	0.060*
C6	0.2309 (2)	0.9138 (2)	0.16359 (19)	0.0473 (6)
H6	0.2892	0.8462	0.1644	0.057*
C7	0.24678 (19)	0.9881 (2)	-0.02538 (18)	0.0437 (5)
C8	0.3403 (2)	0.8856 (3)	-0.02910 (19)	0.0494 (6)
H8	0.3588	0.8183	0.0258	0.059*
C9	0.3990 (2)	0.8858 (3)	-0.10772 (19)	0.0493 (6)
H9	0.3751	0.9540	-0.1613	0.059*
C10	0.49523 (19)	0.7959 (2)	-0.12387 (17)	0.0431 (5)
C11	0.5467 (2)	0.6925 (3)	-0.05077 (19)	0.0566 (7)
H11	0.5169	0.6760	0.0111	0.068*
C12	0.6406 (2)	0.6134 (3)	-0.0672 (2)	0.0635 (7)
H12	0.6750	0.5458	-0.0161	0.076*
C13	0.68407 (19)	0.6350 (3)	-0.16102 (18)	0.0474 (6)
C14	0.6341 (2)	0.7353 (3)	-0.23509 (18)	0.0486 (6)
H14	0.6628	0.7498	-0.2978	0.058*
C15	0.5412 (2)	0.8148 (3)	-0.21662 (19)	0.0518 (6)
H15	0.5080	0.8832	-0.2675	0.062*
O1	0.07099 (15)	1.19360 (19)	-0.01001 (14)	0.0589 (5)
H1A	0.107 (3)	1.171 (3)	-0.061 (2)	0.088*
O2	0.04269 (18)	1.0443 (2)	0.33756 (14)	0.0649 (5)
H2A	0.069 (3)	0.988 (3)	0.387 (2)	0.097*
O3	0.21067 (14)	1.06876 (19)	-0.10709 (13)	0.0565 (5)
O4	0.77775 (17)	0.5545 (2)	-0.17385 (14)	0.0668 (6)
H4A	0.784 (3)	0.563 (4)	-0.2384 (17)	0.100*
O5	0.12061 (17)	0.6281 (2)	0.00416 (14)	0.0587 (5)
H5A	0.072 (2)	0.687 (3)	0.017 (3)	0.088*
H5B	0.149 (3)	0.586 (3)	0.0630 (19)	0.088*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0381 (11)	0.0444 (12)	0.0430 (12)	-0.0015 (9)	0.0192 (9)	-0.0060 (10)
C2	0.0413 (11)	0.0446 (12)	0.0415 (11)	-0.0002 (10)	0.0174 (9)	0.0015 (10)
C3	0.0457 (12)	0.0512 (14)	0.0524 (13)	0.0090 (11)	0.0251 (10)	-0.0011 (11)
C4	0.0502 (12)	0.0514 (13)	0.0409 (12)	0.0005 (10)	0.0247 (10)	-0.0039 (10)
C5	0.0583 (14)	0.0530 (14)	0.0436 (12)	0.0078 (11)	0.0217 (11)	0.0070 (10)
C6	0.0485 (12)	0.0485 (13)	0.0497 (13)	0.0086 (10)	0.0213 (10)	-0.0007 (10)
C7	0.0400 (11)	0.0520 (14)	0.0432 (12)	-0.0049 (10)	0.0183 (9)	-0.0041 (10)
C8	0.0513 (13)	0.0551 (14)	0.0483 (13)	0.0061 (11)	0.0249 (10)	-0.0017 (11)
C9	0.0475 (12)	0.0576 (15)	0.0485 (13)	0.0041 (11)	0.0229 (10)	0.0008 (11)
C10	0.0404 (11)	0.0506 (13)	0.0431 (12)	-0.0005 (10)	0.0193 (10)	-0.0044 (10)
C11	0.0605 (15)	0.0757 (18)	0.0437 (13)	0.0113 (13)	0.0323 (12)	0.0049 (12)
C12	0.0690 (16)	0.0791 (19)	0.0495 (14)	0.0290 (15)	0.0285 (12)	0.0156 (13)
C13	0.0425 (12)	0.0596 (15)	0.0446 (12)	0.0065 (11)	0.0193 (10)	-0.0039 (11)
C14	0.0493 (13)	0.0617 (15)	0.0427 (12)	0.0062 (11)	0.0267 (10)	0.0028 (11)
C15	0.0528 (13)	0.0586 (15)	0.0507 (14)	0.0097 (11)	0.0254 (11)	0.0083 (11)
O1	0.0648 (11)	0.0665 (12)	0.0538 (10)	0.0202 (9)	0.0307 (8)	0.0141 (9)
O2	0.0793 (13)	0.0756 (13)	0.0531 (10)	0.0183 (10)	0.0424 (10)	0.0072 (9)
O3	0.0571 (10)	0.0710 (12)	0.0492 (9)	0.0108 (8)	0.0284 (8)	0.0084 (8)
O4	0.0654 (11)	0.0920 (14)	0.0512 (10)	0.0355 (10)	0.0302 (9)	0.0110 (10)
O5	0.0702 (12)	0.0605 (12)	0.0539 (10)	0.0151 (9)	0.0315 (9)	0.0043 (9)

Geometric parameters (\AA , $^\circ$)

C1—C6	1.405 (3)	C9—H9	0.9300
C1—C2	1.410 (3)	C10—C11	1.383 (3)
C1—C7	1.454 (3)	C10—C15	1.394 (3)
C2—O1	1.359 (3)	C11—C12	1.374 (3)
C2—C3	1.382 (3)	C11—H11	0.9300
C3—C4	1.375 (3)	C12—C13	1.393 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—O2	1.340 (3)	C13—C14	1.364 (3)
C4—C5	1.397 (3)	C13—O4	1.364 (3)
C5—C6	1.371 (3)	C14—C15	1.375 (3)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—H15	0.9300
C7—O3	1.270 (3)	O1—H1A	0.867 (18)
C7—C8	1.465 (3)	O2—H2A	0.819 (18)
C8—C9	1.316 (3)	O4—H4A	0.829 (18)
C8—H8	0.9300	O5—H5A	0.836 (18)
C9—C10	1.453 (3)	O5—H5B	0.836 (18)
C6—C1—C2	116.61 (18)	C8—C9—H9	115.0
C6—C1—C7	123.2 (2)	C10—C9—H9	115.0
C2—C1—C7	120.24 (19)	C11—C10—C15	117.1 (2)
O1—C2—C3	117.03 (19)	C11—C10—C9	123.66 (19)

O1—C2—C1	121.81 (18)	C15—C10—C9	119.2 (2)
C3—C2—C1	121.2 (2)	C12—C11—C10	121.7 (2)
C4—C3—C2	120.5 (2)	C12—C11—H11	119.1
C4—C3—H3	119.7	C10—C11—H11	119.1
C2—C3—H3	119.7	C11—C12—C13	119.6 (2)
O2—C4—C3	117.5 (2)	C11—C12—H12	120.2
O2—C4—C5	122.6 (2)	C13—C12—H12	120.2
C3—C4—C5	119.90 (19)	C14—C13—O4	122.43 (19)
C6—C5—C4	119.4 (2)	C14—C13—C12	119.9 (2)
C6—C5—H5	120.3	O4—C13—C12	117.6 (2)
C4—C5—H5	120.3	C13—C14—C15	119.7 (2)
C5—C6—C1	122.4 (2)	C13—C14—H14	120.1
C5—C6—H6	118.8	C15—C14—H14	120.1
C1—C6—H6	118.8	C14—C15—C10	121.9 (2)
O3—C7—C1	119.85 (19)	C14—C15—H15	119.0
O3—C7—C8	119.07 (19)	C10—C15—H15	119.0
C1—C7—C8	121.1 (2)	C2—O1—H1A	107 (2)
C9—C8—C7	122.1 (2)	C4—O2—H2A	111 (3)
C9—C8—H8	119.0	C13—O4—H4A	108 (2)
C7—C8—H8	119.0	H5A—O5—H5B	107 (3)
C8—C9—C10	130.0 (2)		
C6—C1—C2—O1	178.7 (2)	C2—C1—C7—C8	177.9 (2)
C7—C1—C2—O1	-0.9 (3)	O3—C7—C8—C9	8.8 (4)
C6—C1—C2—C3	-0.5 (3)	C1—C7—C8—C9	-170.2 (2)
C7—C1—C2—C3	179.9 (2)	C7—C8—C9—C10	178.4 (2)
O1—C2—C3—C4	-179.8 (2)	C8—C9—C10—C11	-3.0 (4)
C1—C2—C3—C4	-0.6 (3)	C8—C9—C10—C15	178.3 (3)
C2—C3—C4—O2	-179.0 (2)	C15—C10—C11—C12	1.3 (4)
C2—C3—C4—C5	1.1 (4)	C9—C10—C11—C12	-177.5 (2)
O2—C4—C5—C6	179.5 (2)	C10—C11—C12—C13	-1.5 (4)
C3—C4—C5—C6	-0.6 (4)	C11—C12—C13—C14	0.7 (4)
C4—C5—C6—C1	-0.5 (4)	C11—C12—C13—O4	179.7 (2)
C2—C1—C6—C5	1.1 (3)	O4—C13—C14—C15	-178.9 (2)
C7—C1—C6—C5	-179.4 (2)	C12—C13—C14—C15	0.1 (4)
C6—C1—C7—O3	179.3 (2)	C13—C14—C15—C10	-0.3 (4)
C2—C1—C7—O3	-1.1 (3)	C11—C10—C15—C14	-0.4 (4)
C6—C1—C7—C8	-1.7 (3)	C9—C10—C15—C14	178.4 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1A \cdots O3	0.87 (2)	1.74 (2)	2.530 (2)	150 (3)
O2—H2A \cdots O5 ⁱ	0.82 (2)	1.83 (2)	2.644 (3)	175 (4)
O4—H4A \cdots O3 ⁱⁱ	0.83 (2)	1.95 (2)	2.776 (2)	175 (4)
O5—H5A \cdots O1 ⁱⁱⁱ	0.84 (2)	1.99 (2)	2.802 (2)	164 (3)
O5—H5B \cdots O4 ^{iv}	0.84 (2)	1.97 (2)	2.785 (3)	165 (3)
C9—H9 \cdots O4 ^v	0.93	2.56	3.402 (3)	151

Symmetry codes: (i) *x*, -*y*+3/2, *z*+1/2; (ii) -*x*+1, *y*-1/2, -*z*-1/2; (iii) -*x*, -*y*+2, -*z*; (iv) -*x*+1, -*y*+1, -*z*; (v) -*x*+1, *y*+1/2, -*z*-1/2.

Fig. 1

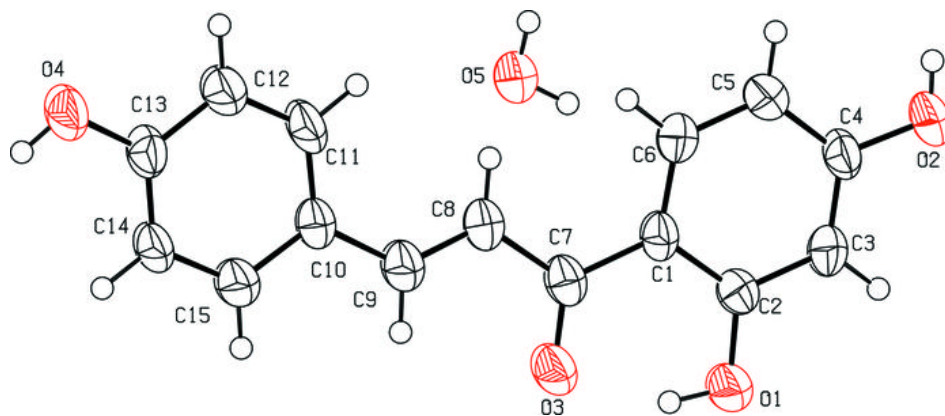


Fig. 2

