

2,2'-Dihydroxychalcone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.048
wR factor = 0.129
Data-to-parameter ratio = 12.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, alternatively called 1-(2-hydroxyphenyl)-3-(2-hydroxyphenyl)-2-propen-1-one, $\text{C}_{15}\text{H}_{12}\text{O}_3$, intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds are formed between molecules related by a center of symmetry, and the dimer is planar as a whole. The dimers are stacked along the shortest axis to form a column. In the column, the $\text{C}=\text{C}$ double bonds related by another center of symmetry approach the shortest $\text{C}\cdots\text{C}$ distance of 3.482 \AA . The intermolecular hydrogen bonds and a chevron structure in the crystal may prohibit topochemical reaction.

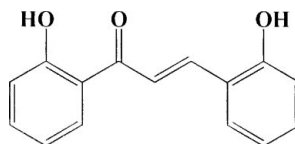
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Comment

Some chalcones are known to be photoreactive in the solid state, e.g. 4-methoxychalcone (Rabinovich & Schmidt, 1970) and one of the six polymorphs of chalcone (Montaudou & Caccamese, 1969). We carried out the present study as part of a topochemical study (Iwamoto & Kashino, 1993; Kanao *et al.*, 1990). After completion of the structure determination for the title compound, (I), we noticed that substantially the same structure has already been reported by Feng *et al.* (1999). However, they could only determine the structure with poor precision because good quality crystals were not available at the time. We report here the crystal structure of (I), determined with significantly higher precision than before.



(I)

An intramolecular $\text{O}2-\text{H}21\cdots\text{O}1$ hydrogen bond is formed in (I) (Fig. 1) and the molecule is roughly planar, as seen from the torsion angles in Table 1. The molecules related by a center of symmetry at Wyckoff position $4a$ form a dimer linked by an $\text{O}3-\text{H}31\cdots\text{O}1^i$ hydrogen bond [symmetry code: (i) $1-x, 1-y, -z$] (Fig. 1 and Table 2). The dimer lies on (014). The dimers are stacked along b to form a column. The columns related by a twofold screw axis form a chevron.

In the column, the $\text{C}=\text{C}$ double bonds of the molecules related by a center of symmetry at Wyckoff position $4b$ approach to give a shortest $\text{C}\cdots\text{C}$ distance of $\text{C}3\cdots\text{C}3^{ii} = 3.482 \text{ \AA}$ [symmetry code: (ii) $1-x, -y, -z$]. However, this is not an optimal contact for photodimerization to occur, because the $\text{C}2^{ii}$ atom does not make a short contact with the $\text{C}3$ atom. The mode of molecular overlapping shows that the

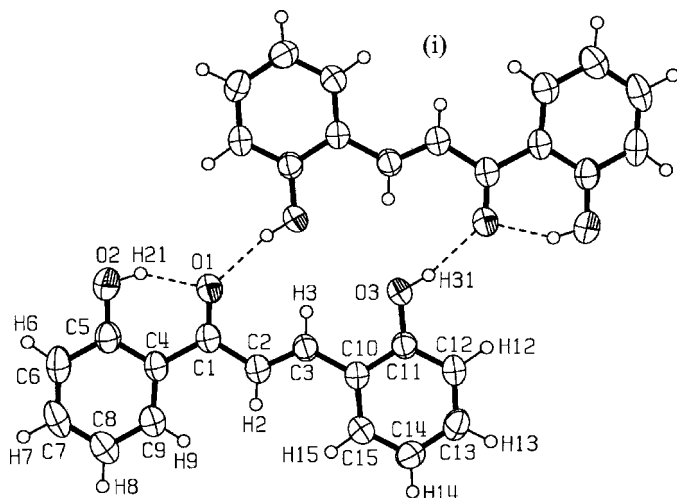


Figure 1
A molecular view of the title compound showing the hydrogen-bonding pattern and the atomic numbering scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines (Farrugia, 1997). [Symmetry code: (i) $1 - x, 1 - y, -z$]

molecule at (ii) should be displaced along the C2—C3 bond by about 1.3 Å relative to the original molecule in order to realise the shortest contact between atoms C3 and C2ⁱⁱ. Such a displacement may be prohibited, because it causes breakdown of the intermolecular hydrogen bonds and the chevron structure formed in the crystal. We could not observe any indication of the photoreaction in the rotation and Weissenberg photographs of the crystals taken after exposing the specimens to sunlight for one month [the method of light exposure used by Cohen *et al.* (1964)].

Experimental

Compound (I) was prepared by the reaction of 2-hydroxyacetophenone and 2-hydroxybenzaldehyde according to the method described by Alcantara *et al.* (1987). Crystals were grown from an ethanol solution by slow evaporation.

Crystal data

$C_{15}H_{12}O_3$
 $M_r = 240.25$
Monoclinic, $C2/c$
 $a = 23.630$ (5) Å
 $b = 5.582$ (2) Å
 $c = 18.004$ (6) Å
 $\beta = 95.04$ (2)°
 $V = 2365.4$ (12) Å³
 $Z = 8$

$D_x = 1.349$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 11.0$ – 11.5°
 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
Plate, yellow
 $0.30 \times 0.25 \times 0.10$ mm

Data collection

Rigaku AFC-5R diffractometer
 ω - 2θ scans
2812 measured reflections
2719 independent reflections
1824 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$
 $\theta_{max} = 27.5^\circ$

$h = -30 \rightarrow 30$
 $k = 0 \rightarrow 7$
 $l = 0 \rightarrow 23$
3 standard reflections every 97 reflections
intensity decay: 0.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.129$
 $S = 1.17$
2718 reflections
212 parameters
All H-atom parameters refined
 $w = 1/[\sigma^2(F_o) + 0.0009|F_o|^2]$

$(\Delta/\sigma)_{max} = 0.01$
 $\Delta\rho_{max} = 0.24$ e Å⁻³
 $\Delta\rho_{min} = -0.19$ e Å⁻³
Extinction correction: Zachariasen (1967)
Extinction coefficient: 4.8 (9) $\times 10^{-7}$

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2467 (18)	C6—C7	1.365 (3)
O2—C5	1.341 (2)	C7—C8	1.381 (3)
O3—C11	1.3500 (18)	C8—C9	1.370 (2)
C1—C2	1.462 (2)	C10—C11	1.401 (2)
C1—C4	1.475 (2)	C10—C15	1.391 (2)
C2—C3	1.318 (2)	C11—C12	1.393 (2)
C3—C10	1.460 (2)	C12—C13	1.373 (2)
C4—C5	1.413 (2)	C13—C14	1.381 (3)
C4—C9	1.399 (2)	C14—C15	1.380 (2)
C5—C6	1.400 (2)		
O1—C1—C2	120.88 (14)	C7—C8—C9	119.19 (19)
O1—C1—C4	119.58 (13)	C4—C9—C8	122.28 (17)
C2—C1—C4	119.52 (14)	C3—C10—C11	118.85 (14)
C1—C2—C3	123.39 (17)	C3—C10—C15	123.14 (13)
C2—C3—C10	127.35 (16)	C11—C10—C15	118.01 (13)
C1—C4—C5	120.13 (14)	O3—C11—C10	117.24 (13)
C1—C4—C9	122.29 (14)	O3—C11—C12	122.20 (14)
C5—C4—C9	117.57 (14)	C10—C11—C12	120.56 (14)
O2—C5—C4	122.68 (14)	C11—C12—C13	119.62 (15)
O2—C5—C6	117.86 (15)	C12—C13—C14	120.94 (15)
C4—C5—C6	119.46 (16)	C13—C14—C15	119.32 (17)
C5—C6—C7	120.63 (17)	C10—C15—C14	121.55 (16)
C6—C7—C8	120.84 (17)		
C1—C2—C3—C10	179.53 (15)	C2—C3—C10—C11	176.16 (17)
C2—C1—C4—C5	173.11 (14)	C3—C2—C1—C4	-170.77 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2—H21 \cdots O1	0.89 (2)	1.76 (2)	2.5630 (17)	150 (2)
O3—H31 \cdots O1 ⁱ	0.83 (2)	1.95 (2)	2.7610 (18)	166.4 (19)
C3—H3 \cdots O3 ⁱ	0.918 (18)	2.388 (18)	3.269 (2)	160.7 (15)

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

All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SAP91* (Fan, 1991); program(s) used to refine structure: *TEXSAN for Windows*; software used to prepare material for publication: *TEXSAN for Windows*.

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