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# 3-(1,3-Benzodioxol-5-yl)-1-(4-methoxyphenyl)prop-2-enone

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**Key indicators**

Single-crystal X-ray study
*T* = 297 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.061
*wR* factor = 0.165
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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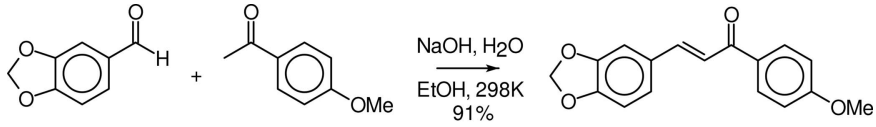
The title compound, $C_{17}H_{14}O_4$, shows satisfactory cytotoxic activity against *Artemia salina*. Two non-classical intermolecular C—H···O hydrogen bonds [C···O = 3.273 (2) and 3.302 (3) Å] join the molecules alternately head-to-head and tail-to-tail across crystallographic inversion centres, resulting in a linear chain along the [1$\bar{4}$1] direction.

## Comment

Chalcones can be easily obtained from the aldol condensation of aromatic aldehydes and aromatic ketones. This class of compounds presents interesting biological properties, such as cytotoxicity (Lawrence *et al.*, 2001), antiherpes activity (Phrutivorapongkul *et al.*, 2003) and antitumour activity (Xia *et al.*, 2000) and may be useful for the chemotherapy of Leishmaniasis (Pandey *et al.*, 2005), among others. The title compound, (I), shows a satisfactory cytotoxic activity against *Artemia salina*. Other tests with *E. coli* and *S. aurus* are being carried out.




(1) (2) (I)

The crystal structure study of (I) was undertaken in order to establish the structure and conformation of the various groups. With this conformational study established, it will be quicker to carry out chemometric methods (multivariate statistics) to verify the results of the pharmacological tests (Camargo *et al.*, 2003).

In the title molecule (Fig. 1 and Table 1), the spatial arrangement of the keto group C7═O1and the olefinic double bond C8═C9 about the linking single bond C7—C8 is *s-cis*, as seen from the O1═C7—C8═C9 torsion angle of 12.9 (3)°. The dihedral angle between the least-squares plane through the benzodioxol ring (O2/O3/C10···C16) and the benzene ring C1···C6 is 41.12 (4)°. The widening of the C5—C6—C7 angle to 122.75 (16)° can be related to the short interatomic contact between atoms H5 and H8 (2.24 Å). In the same way, the strain induced by the short H8···H11 contact (2.20 Å) results in a slight opening of the C8—C9—C10 angle to 127.33 (17)°.

We have found two entries with the chalcone skeleton type of (I) in the Cambridge Structural Database (CSD; Version 5.26; Allen, 2002), *viz*. refcodes CIBCOF ($C_{29}H_{25}NO_7$; Pridgen *et al.*, 1999) and BAGXIR ($C_{30}H_{23}ClO_6$; Kerr *et al.*, 2001). In both cases, the olefinic double bond is trisubstituted.

The former compound shows bond lengths and angles in good agreement with (I). The second compound contains two molecules in the asymmetric unit and the related bond distances and angles of the $\alpha,\beta$–unsaturated ketone show some significant differences: the corresponding average bond distances are C7═O1 = 1.256 Å, C7—C8 = 1.473 Å and C9—C10 = 1.474 Å, with bond angles at C6, C7, C8 and C9 in the range 123.0–127.8°.

Intermolecular C—H···O hydrogen bonds are formed (Table 2). The molecules are linked alternately head-to-head and tail-to-tail across inversion centres, resulting in a linear chain along the [1$\bar{4}$1] direction, as shown in Fig. 2. A contact between O1 and C16$^{i}$ of the neighbouring molecular chain [3.800 (3) Å; symmetry code: (i) $-1+x, 1+y, z$] probably contributes to the relatively large C1···C6—C7═O1 torsion angle of 22.3 (3)°.

## Experimental

Compound (I) was obtained in 91% yield by the aldol condensation of piperonal, (1), and 4-methoxyacetophenone, (2), using a method described previously by Lawrence *et al.* (2001). Prismatic crystals were obtained from an EtOH solution (m.p. 403 K). $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 3.88 (*s*, 3H, H17), 6.02 (*s*, 2H, H16), 6.82–6.85 (*d*, *J* = 7.79 Hz, 2H, H2 and H4), 6.95–7.00 (*m*, 1H, H12), 7.10–7.12 (*dd*, *J* = 1.87 and 8.41 Hz, 1H, H11), 7.16–7.17 (*d*, *J* = 1.87 Hz, 1H, H15), 7.35–7.41 (*d*, *J* = 15.88 Hz, 1H, H8), 7.70–7.75 (*d*, *J* = 15.58 Hz, 1H, H9), 7.99–8.05 (*m*, 2H, H1 and H5); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 188.5 (C═O), 163.3 (C), 149.7 (C), 148.3 (C), 143.8 (CH), 131.2 (CH), 130.7 (CH), 129.5 (C), 125.0 (CH), 119.9 (CH), 113.8 (CH), 108.6 (CH), 106.6 (CH), 101.6 ($CH_2$), 55.5 ($CH_3$).

*Crystal data*

| | |
|---|---|
| $C_{17}H_{14}O_4$ | $Z = 2$ |
| $M_r = 282.28$ | $D_x = 1.408$ Mg m$^{-3}$ |
| Triclinic, $P\bar{1}$ | Cu $K\alpha$ radiation |
| $a = 6.0987$ (9) Å | Cell parameters from 25 reflections |
| $b = 7.463$ (1) Å | $\theta = 18.1$–44.5° |
| $c = 14.932$ (1) Å | $\mu = 0.83$ mm$^{-1}$ |
| $\alpha = 84.18$ (1)° | $T = 297$ (2) K |
| $\beta = 89.65$ (1)° | Prism, yellow |
| $\gamma = 79.90$ (1)° | 0.35 × 0.35 × 0.28 mm |
| $V = 665.60$ (14) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $\theta_{max} = 67.1°$ |
| non–profiled $\omega/2\theta$ scans | $h = -7 \rightarrow 7$ |
| Absorption correction: none | $k = -8 \rightarrow 8$ |
| 2435 measured reflections | $l = 0 \rightarrow 17$ |
| 2343 independent reflections | 2 standard reflections |
| 2234 reflections with $I > 2\sigma(I)$ | frequency: 120 min |
| $R_{int} = 0.009$ | intensity decay: <1% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.1073P)^2 + 0.1215P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.061$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2) = 0.165$ | $\Delta\rho_{max} = 0.41$ e Å$^{-3}$ |
| $S = 1.13$ | $\Delta\rho_{min} = -0.41$ e Å$^{-3}$ |
| 2343 reflections | Extinction correction: *SHELXL97* |
| 209 parameters | Extinction coefficient: 0.330 (19) |
| H atoms treated by a mixture of independent and constrained refinement | |

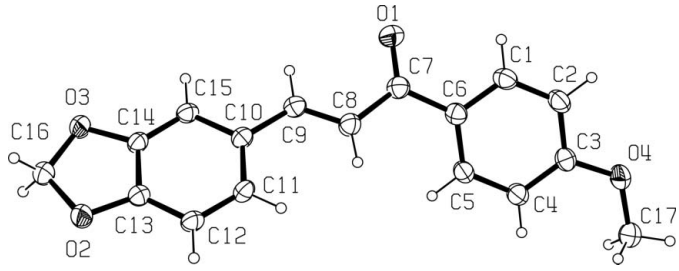



**Figure 1**
View of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

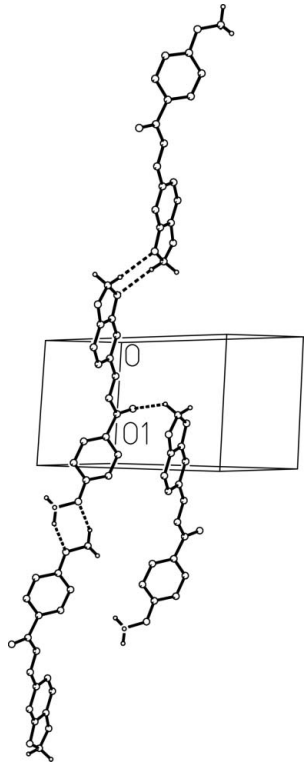



**Figure 2**
The packing, viewed almost perpendicular to the [100] axis; the [010] axis is vertical and [001] is horizontal pointing to the right. Intermolecular C—H···O hydrogen bonds are shown as dashed lines. A short contact is also shown between atom O1 and the neighbouring chain.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C7 | 1.224 (2) | O4—C17 | 1.412 (3) |
| O2—C13 | 1.367 (2) | C6—C7 | 1.485 (2) |
| O2—C16 | 1.428 (2) | C7—C8 | 1.481 (3) |
| O3—C14 | 1.377 (2) | C8—C9 | 1.330 (3) |
| O3—C16 | 1.424 (2) | C9—C10 | 1.466 (2) |
| O4—C3 | 1.358 (2) | | |
| C13—O2—C16 | 105.84 (13) | C8—C7—C6 | 118.28 (16) |
| C14—O3—C16 | 105.91 (13) | C9—C8—C7 | 121.63 (17) |
| C3—O4—C17 | 118.84 (15) | C8—C9—C10 | 127.33 (17) |
| C5—C6—C7 | 122.75 (16) | | |
| C1—C6—C7—O1 | 22.3 (3) | C7—C8—C9—C10 | −176.36 (15) |
| O1—C7—C8—C9 | 12.9 (3) | C8—C9—C10—C11 | 7.7 (3) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C16—H16$A$···O3$^{i}$ | 0.95 (2) | 2.56 (2) | 3.273 (2) | 132.0 (18) |
| C17—H17$A$···O4$^{ii}$ | 0.99 (2) | 2.43 (3) | 3.302 (3) | 146 (2) |

Symmetry codes: (i) $-x+2, -y-1, -z+1$; (ii) $-x+1, -y+3, -z$.

All H atoms, except those bonded to C9, C16 and C17, were positioned geometrically and allowed to ride on their parent atoms, with C—H distances constrained to 0.93 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C). The H atoms bonded to C16 and C17 were found in a difference map and their positions were refined with soft restraints H···H = 1.57 (4) Å and C—H = 0.96 (4) Å, and with $U_{iso}$(H16$A$/H16$B$) = 1.2$U_{eq}$(C16) and $U_{iso}$(H17$A$/H17$B$/H17$C$) = 1.5$U_{eq}$(C17). Additional restraints were applied for H16$A$ and H17$A$ which are involved in hydrogen bonds, in order to ensure a sensible geometry for the methylene and methyl groups: O3···H16$A$ = 1.95 (4) Å and O4···H17$A$ = 1.95 (4) Å. Finally, H9 was found in a difference map and its position refined freely; $U_{iso}$(H9) = 1.2$U_{eq}$(C9).

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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