

Table 1. Selected geometric parameters (Å, °)

S1—C10	1.738 (2)	O3—C12	1.209 (3)
S1—C14	1.798 (3)	N1—C13	1.145 (3)
O1—C2	1.369 (3)	C5—C8	1.468 (3)
O1—C1	1.429 (3)	C9—C15	1.508 (3)
O2—C12	1.369 (3)	C11—C13	1.435 (3)
O2—C8	1.374 (2)		
C10—S1—C14	108.08 (11)	C9—C10—S1	113.70 (15)
C2—O1—C1	117.8 (2)	C10—C11—C13	126.9 (2)
C12—O2—C8	123.27 (16)	C13—C11—C12	112.00 (19)
O1—C2—C3	124.6 (2)	O3—C12—O2	117.35 (19)
O1—C2—C7	115.4 (2)	O3—C12—C11	126.3 (2)
C9—C8—C5	129.82 (19)	O2—C12—C11	116.39 (18)
O2—C8—C5	108.77 (17)	N1—C13—C11	174.8 (3)
C11—C10—S1	126.93 (17)		
C1—O1—C2—C3	-3.2 (3)	C4—C5—C8—O2	137.6 (2)
C1—O1—C2—C7	176.6 (2)	C6—C5—C8—O2	-37.3 (3)
O1—C2—C3—C4	178.7 (2)	C14—S1—C10—C11	-14.7 (2)
O1—C2—C7—C6	-178.7 (2)	C14—S1—C10—C9	164.25 (17)
C12—O2—C8—C5	179.82 (18)	S1—C10—C11—C13	-7.6 (4)
C4—C5—C8—C9	-41.2 (3)	C10—C11—C13—N1	-146 (3)
C6—C5—C8—C9	143.8 (2)	C12—C11—C13—N1	31 (3)

The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different  $\varphi$  angle for the crystal and each exposure of 10 s covered 0.3° in  $\omega$ . The crystal to detector distance was 5.01 cm. Coverage of the unique set was over 97% complete to at least 26° in  $\theta$ . Crystal decay was monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: SMART (Siemens, 1994b). Cell refinement: SAINT (Siemens, 1995). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL/PC (Siemens, 1994a). Program(s) used to refine structure: SHELXL96 (Sheldrick, 1996). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXTL/PC.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher, McMeeking & Parkin, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1145). Services for accessing these data are described at the back of the journal.

## References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.  
 Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.  
 Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.  
 Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.

- Hatada, T., Sone, M., Tominaga, Y., Natsuki, R., Matsuda, Y. & Kobayashi, G. (1975). *Yakugaku Zasshi*, **95**, 623–628; *Chem. Abstr.* **83**, 206076.  
 Hussaini, F. A., Pragma, Ram, V. J., Singh, S. K., Nath, M., Shoeb, A. & Bhaduri, A. P. (1994). *J. Chem. Res. (S)*, p. 86.  
 Israili, Z. H. & Smisman, E. E. (1976). *J. Org. Chem.* **41**, 4070–4074.  
 Kretzschmar, R., Meyer, H. J., Teschendorf, H. J. & Zoellner, B. (1969). *Arch. Int. Pharmacodyn. Ther.* **180**, 475–491.  
 Kumar, A., Kumar, N., Parmar, V. S. & Errington, W. (1996). *Acta Cryst.* **C52**, 127–129.  
 Ram, V. J., Hussaini, F. A., Singh, S. K. & Shoeb, A. (1993). *J. Chem. Res. (S)*, pp. 110–111.  
 Ram, V. J., Mumtazuddin, S., Pragma, Haque, N., Hussaini, F. A., Shoeb, A. & Bhaduri, A. P. (1994). *J. Chem. Res. (S)*, pp. 354–355.  
 Ram, V. J., Verma, M., Hussaini, F. A. & Shoeb, A. (1991). *J. Chem. Res. (S)*, pp. 98–99.  
 Sheldrick, G. M. (1996). *SHELXL96. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Siemens (1994a). *SHELXTL/PC*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1994b). *SMART. Data Processing Software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1995). *SAINTE Software Reference Manual*. Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1997). **C53**, 1438–1440

## 1-(3,4-Dimethoxyphenyl)-3-(3-methylphenyl)prop-2-en-1-one

NAWAL K. SHARMA,<sup>a</sup> RAJESH KUMAR,<sup>a</sup> VIRINDER S. PARMAR<sup>a</sup> AND WILLIAM ERRINGTON<sup>b</sup>

<sup>a</sup>Department of Chemistry, University of Delhi, Delhi 110 007, India, and <sup>b</sup>Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk

(Received 21 January 1997; accepted 2 May 1997)

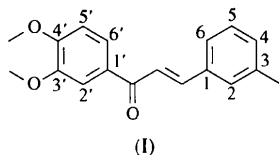
## Abstract

The synthesis and structure of the title compound, C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>, are described. The molecule is almost flat and the *ortho*-methoxy groups point away from each other.

## Comment

1,3-Diarylprop-2-enones (chalcones) constitute an important group of natural products and some have been found to possess a wide range of biological activities, viz antibacterial, antifungal and anti-inflammatory (Ahluwalia, Kaila & Bala, 1986; Bhat, Bhamaria, Patel, Bellare & Deliwala, 1972; Mathew, Subba Rao & Rambhav, 1984; Oganessian, Yakavenko, Khartyan,

Preshkov & Cherevatyi, 1986). Encouraged by these findings, we have synthesized a series of novel chalcones based on the 3,4-dioxygenation pattern in the benzoyl moiety for structure–activity studies. This paper reports the synthesis of a new chalcone, (I); its X-ray structure was determined to assign unambiguously its constitution.



The molecular structure of (I) is represented in Fig. 1. The molecule is approximately flat with a dihedral angle between the two aromatic planes of  $19.5(1)^\circ$ . The two methoxy groups are displaced slightly out of the plane of the phenyl group to which they are attached; this is illustrated by the torsion angles  $C17-O2-C3-C2$  and  $C16-O1-C2-C3$  of  $170.8(2)$  and  $-175.4(2)^\circ$ , respectively. The methyl groups in these methoxy groups point away from each other, presumably to reduce steric interactions. Bond lengths and angles are unremarkable.

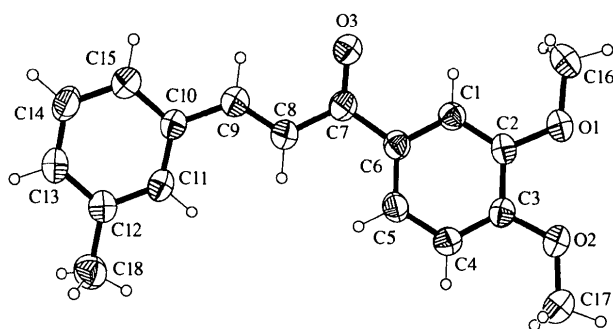


Fig. 1. A view of the title molecule showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The molecules pack together along two sets of parallel planes; these planes are inclined at an angle of approximately  $72^\circ$ . Adjacent molecules within a plane are aligned in a head-to-tail fashion. All intermolecular O—C distances exceed the sum of the van der Waals radii and thus significant hydrogen bonding is unlikely.

## Experimental

3,4-Dimethoxyacetophenone (980 mg, 5 mmol) was dissolved in ethanol (30 ml) and crushed KOH (2 g) was added; the flask was immersed in a bath of crushed ice, followed by the addition of 3-methylbenzaldehyde (600 mg, 5 mmol) dissolved in ethanol (5 ml). The reaction mixture was stirred at 300 K and completion of the reaction was monitored by TLC. Ice-cold water (50 ml) was added to the reaction mixture after 48 h

and the red–yellow solid that separated was filtered, washed with water and cold ethanol, dried and purified by column chromatography on silica gel (solvent system: ethyl acetate and petroleum ether, 1:9); the product (I) was isolated as a yellow crystalline solid, m.p. 377 K. IR (nujol)  $\nu_{\max}$ : 2950, 1660 (C=O), 1580, 1510, 1460, 1420, 1380, 1330, 1280, 1240, 1160, 1020, 850, 780, 720 and  $680\text{ cm}^{-1}$ . UV (MeOH)  $\lambda_{\max}$ : 215, 240 and 330 nm.  $^1\text{H NMR}$  (250 MHz,  $\text{CDCl}_3$ ):  $\delta$  2.41 (s, 3H,  $\text{CH}_3$ ), 3.97 (s, 3H,  $\text{OCH}_3$ ), 3.98 (s, 3H,  $\text{OCH}_3$ ), 6.93 (d,  $J = 8$  Hz, 1H, H-5'), 7.24–7.35 (m, 2H, H-4, H-5), 7.45 (d,  $J = 2$  Hz, 1H, H-2), 7.47–7.58 (m, 2H, H- $\alpha$ , H-6), 7.64 (d,  $J = 2$  Hz, 1H, H-2'), 7.70 (m, 1H, H-6'), 7.80 (d,  $J = 15.5$  Hz, 1H, H- $\beta$ ).  $^{13}\text{C NMR}$  (62.9 MHz,  $\text{CDCl}_3$ ):  $\delta$  21.2 ( $-\text{CH}_3$ ), 55.92 ( $2 \times -\text{OCH}_3$ ), 109.9 (C-2'), 110.9 (C-5'), 121.4 (C- $\alpha$ ), 122.9 (C-6'), 125.4 (C-6), 128.7 (C-2), 128.9 (C-4), 131.0 (C-5), 131.3 (C-1'), 134.95 (C-1), 138.4 (C-3), 144.0 (C- $\beta$ ), 149.2 (C-3'), 153.2 (C-4'), 188.4 ( $>\text{C}=\text{O}$ ). EIMS  $m/z$  (relative intensity): 282 ( $M^+$ ) (100), 281 (38), 267 (64), 254 (10), 251 (10), 239 (10), 165 (41), 145 (15).

## Crystal data

$\text{C}_{18}\text{H}_{18}\text{O}_3$   
 $M_r = 282.32$   
 Monoclinic  
 $P2_1/n$   
 $a = 9.1496(8)\text{ \AA}$   
 $b = 8.5119(6)\text{ \AA}$   
 $c = 19.9057(17)\text{ \AA}$   
 $\beta = 98.990(2)^\circ$   
 $V = 1531.2(4)\text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.225\text{ Mg m}^{-3}$   
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073\text{ \AA}$   
 Cell parameters from 3751 reflections  
 $\theta = 2.07\text{--}23.26^\circ$   
 $\mu = 0.082\text{ mm}^{-1}$   
 $T = 220(2)\text{ K}$   
 Block  
 $0.61 \times 0.40 \times 0.32\text{ mm}$   
 Yellow

## Data collection

Siemens SMART CCD area detector  
 $\omega$  scans  
 Absorption correction: none  
 6501 measured reflections  
 2187 independent reflections

1771 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.083$   
 $\theta_{\max} = 23.26^\circ$   
 $h = -10 \rightarrow 9$   
 $k = -9 \rightarrow 9$   
 $l = -22 \rightarrow 19$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.128$   
 $S = 1.066$   
 2187 reflections  
 202 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.5176P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.161\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.164\text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL96* (Sheldrick, 1996)  
 Extinction coefficient: 0.010(2)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C2	1.369(2)	C6—C7	1.488(3)
O1—C16	1.420(3)	C7—C8	1.484(3)
O2—C3	1.360(2)	C8—C9	1.331(3)
O2—C17	1.430(3)	C9—C10	1.472(3)
O3—C7	1.232(2)	C12—C18	1.505(3)

C2—O1—C16	117.70 (16)	O2—C3—C2	115.54 (17)
C3—O2—C17	117.61 (17)	C5—C6—C7	123.52 (18)
O1—C2—C1	125.72 (19)	C8—C9—C10	127.2 (2)
O1—C2—C3	114.45 (16)	C11—C12—C18	121.10 (19)
O2—C3—C4	124.84 (19)		
C16—O1—C2—C1	3.9 (3)	C6—C7—C8—C9	168.1 (2)
C16—O1—C2—C3	-175.38 (19)	C7—C8—C9—C10	-175.3 (2)
C17—O2—C3—C4	-8.4 (3)	C8—C9—C10—C15	174.0 (2)
C17—O2—C3—C2	170.8 (2)	C18—C12—C13—C14	178.0 (2)
O1—C2—C3—O2	1.1 (3)		

The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different  $\varphi$  angle for the crystal and each exposure of 10 s covered  $0.3^\circ$  in  $\omega$ . The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 95% complete to at least  $23^\circ$  in  $\theta$ . Crystal decay, monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections, was found to be negligible. Riding H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they were attached.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXL96* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXL96*. Molecular graphics: *SHELXTL/PC* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXTL/PC*.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher, McMeeking & Parkin, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993). RK thanks the Council for Scientific and Industrial Research (CSIR, New Delhi, India) for the award of a research fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1168). Services for accessing these data are described at the back of the journal.

## References

- Ahluwalia, V. K., Kaila, N. & Bala, S. (1986). *Indian J. Chem.* **25B**, 663–664.
- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
- Bhat, A. K., Bhamaria, R. P., Patel, M. R., Bellare, R. A. & Deliwala, C. V. (1972). *Indian J. Chem.* **10**, 694–698.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
- Mathew, J., Subba Rao, A. V. & Rambhav, S. (1984). *Curr. Sci.* **53**, 576–578.
- Oganessian, E. T., Yakavenko, V. I., Khartyan, M. M., Preshkov, S. R. & Cherevatyi, V. S. (1986). *Khim. Farm. Zh.* **20**, 696–702; *Chem. Abstr.* **105**, 126832.
- Sheldrick, G. M. (1995). *SHELXTL/PC*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1996). *SHELXL96. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Siemens (1995). *SMART and SAINT. Data Collection and Processing software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1997). **C53**, 1440–1442

## 1-(2-Acetoxy-4-methoxyphenyl)-2-phenylethanone†

HARI N. PATI,<sup>a</sup> VIRINDER S. PARMAR<sup>a</sup> AND WILLIAM ERRINGTON<sup>b</sup>

<sup>a</sup>Department of Chemistry, University of Delhi, Delhi 110 007, India, and <sup>b</sup>Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk

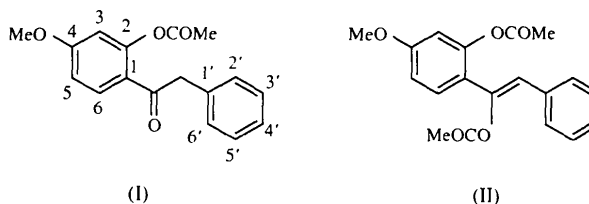
(Received 14 April 1997; accepted 20 May 1997)

## Abstract

The structure of the title compound, C<sub>17</sub>H<sub>16</sub>O<sub>4</sub>, obtained as the major product of the biotransformation of a diacetylated desoxybenzoin using porcine pancreatic lipase, has been confirmed. The molecule is twisted with an angle of  $79.0(1)^\circ$  between the two aromatic rings.

## Comment

Different classes of polyphenolics are known to exhibit a wide range of biological activities. Consequently, we have synthesized a series of differently substituted 1,2-diarylethanones (desoxybenzoin) and some of them have been shown to exhibit pronounced antiviral and antimicrobial activities (Parmar *et al.*, 1996). In addition, polyhydroxylated desoxybenzoin in different protected forms (as methyl/benzyl ethers or acyl derivatives) are used as building blocks for the synthesis of different bioactive polyphenolics (Gandhidasan, Nee-lakantan & Raman, 1982; Jain, Arya & Nayyar, 1984; Jain, Tyagi & Prasad, 1988). We have carried out biotransformation studies on several peracetylated desoxybenzoin (Parmar *et al.*, 1992, 1993). The title compound, (I), was obtained as the major product during the deacetylation of compound (II) by porcine pancreatic lipase (PPL) in organic solvents (Parmar, Pati, Prasad, Azim & Errington, 1997). The objective of this X-ray investigation was to confirm unambiguously the molecular structure of this biotransformation product.



The molecular structure of (I) is confirmed and illustrated in Fig. 1. Bond lengths and angles are

† Alternative systematic name: 5-methoxy-2-(phenylacetyl)phenyl acetate.