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 φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 95% complete to at least 23° in θ. 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*.

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.

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

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Abstract

The structure of the title compound, C$_{17}$H$_{16}$O$_4$, 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)° 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 (desoxybenzoins) and some of them have been shown to exhibit pronounced antiviral and antimicrobial activities (Parmar *et al.*, 1996). In addition, polyhydroxylated desoxybenzoins in different protected forms (as methyl/benzyl ethers or acyl derivatives) are used as building blocks for the synthesis of different bioactive polyphenolics (Gandhidasan, Neelakantan & Raman, 1982; Jain, Arya & Nayyar, 1984; Jain, Tyagi & Prasad, 1988). We have carried out biotransformation studies on several peracetylated desoxybenzoins (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.

(I)

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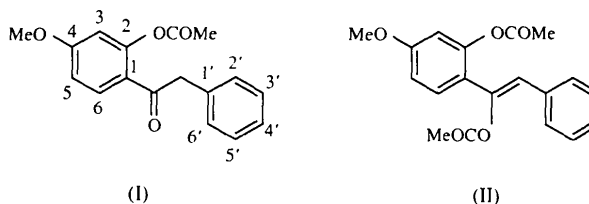
The molecular structure of (I) is confirmed and illustrated in Fig. 1. Bond lengths and angles are

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

unexceptional. In the solid state, the molecule is twisted so that the dihedral angle between the two aromatic rings is 79.0 (1)°. The C9—O3—C4—C5 torsion angle indicates that the methoxy group is twisted at 10.3 (3)° with respect to the attached phenyl ring; the O1—C7—C8 plane of the acetoxy group is aligned at 81.0 (1)° with respect to this same phenyl ring. There are no obvious short-range intermolecular interactions.
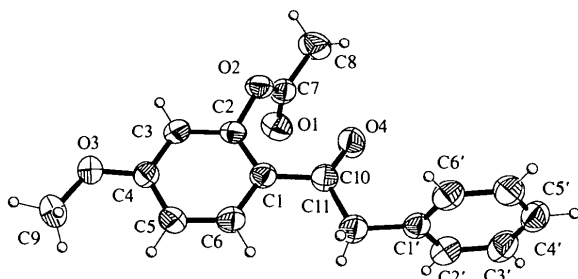

Fig. 1. View of the molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

To a solution of (II) (Parmar et al., 1997) in dry tetrahydrofuran (20 ml) containing n-butanol (0.5 ml), PPL (200 mg, procured from Sigma Chemical Co., USA, and dried over P_2O_5) was added and the suspension was stirred at 315–320 K for 48 h. The reaction was quenched by filtering off the enzyme, the solvent was removed to dryness in vacuo and the residue was subjected to column chromatography to afford (I), which recrystallized from chloroform as colourless needles, m.p. 403 K (literature m.p. 396–398 K; Donnelly & Maloney, 1979). IR (nujol) ν_{max}: 2995, 1760, 1680, 1620, 1580, 1460, 1380, 1200, 1120 and 860 cm^{-1}. UV (MeOH) λ_{max}: 298 and 289 nm. ^{1}H NMR (250 MHz, CDCl$_3$): δ 2.33 (s, 3H, OCOCH$_3$), 3.83 (s, 3H, OCH$_3$), 4.17 (s, 2H, –COCH$_2$–), 6.33 (s, 1H, H-3), 6.61 (m, 1H, H-5), 7.20 (m, 2H, H-3' and H-5'), 7.24 (m, 3H, H-2', H-4' and H-6') and 7.32 p.p.m. (d, 1H, J = 8 Hz, H-6). ^{13}C NMR (62.9 MHz, CDCl$_3$): δ 21.04, 47.33, 55.52, 108.68 (C-5), 111.44 (C-4'), 126.66 (C-3), 127.45 (C-1'), 128.43 (C-3' and C-5'), 129.26 (C-2' and C-6'), 130.50 (C-6), 132.20 (C-2), 151.42 (C-1), 163.46 (C-4), 169.36 and 129.26 p.p.m. (C=O). EIMS, m/z (%int.): 284 [M$^+$] (18), 242 (33), 193 (42), 151 (100), 91 (6) and 43 (12).

Crystal data

C$_{17}$H$_{16}$O$_4$	Mo $K\alpha$ radiation
M_r = 284.30	λ = 0.71073 Å
Monoclinic	Cell parameters from 4175
$P2_1/c$	reflections
a = 8.5946 (6) Å	θ = 2.17–23.28°
b = 18.775 (2) Å	μ = 0.092 mm^{-1}
c = 9.0289 (6) Å	T = 220 (2) K
β = 91.58 (4)°	Block
V = 1456.4 (2) Å^3	0.36 × 0.29 × 0.10 mm
Z = 4	Colourless
D_x = 1.297 Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART area-detector diffractometer	1695 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.046
Absorption correction: none	θ_{max} = 23.28°
6209 measured reflections	h = -7 → 9
2080 independent reflections	k = -20 → 19
	l = -10 → 10

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.136 e Å^{-3}
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$\Delta\rho_{min}$ = -0.135 e Å^{-3}
$wR(F^2)$ = 0.101	Extinction correction:
S = 1.121	SHELXL96 (Sheldrick,
2080 reflections	1996)
193 parameters	Extinction coefficient:
H atoms riding	0.032 (3)
$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 0.3608P]$	Scattering factors from International Tables for
where $P = (F_o^2 + 2F_c^2)/3$	Crystallography (Vol. C)
$(\Delta/\sigma)_{max}$ = 0.001	

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

O1—C7	1.198 (2)	O3—C4	1.364 (2)
O2—C7	1.361 (2)	O3—C9	1.434 (2)
O2—C2	1.406 (2)	O4—C10	1.218 (2)
O3—C4—C3	115.51 (16)	C1'—C11—C10	113.85 (16)
C7—O2—C2—C1	77.1 (2)	C2—C1—C10—C11	177.80 (18)
C9—O3—C4—C5	10.3 (3)	C1—C10—C11—C1'	-175.38 (16)
C2—O2—C7—O1	10.8 (3)	C10—C11—C1'—C2'	-100.6 (2)
C2—O2—C7—C8	-170.13 (17)	C10—C11—C1'—C6'	78.9 (2)
C2—C1—C10—O4	-2.5 (3)		

The temperature of the crystal was controlled using an 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 φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 99% complete to at least 23° in θ. Crystal decay was monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections, and was found to be negligible. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; each H atom were given an isotropic displacement parameter equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which it is attached.

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

$C_{17}H_{16}O_4$

Scientific and Industrial Research (CSIR, New Delhi, India) for the award of a research fellowship.

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3-Cyano-6-(2-methoxyphenyl)-4-methylthio-2H-pyran-2-one

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Abstract

The molecule of the title compound, $C_{14}H_{11}NO_3S$, is approximately planar with the two six-membered rings inclined at an angle of $10.99(12)°$.

Comment

Compounds containing the pyrone nucleus, in particular the 4-hydroxy-2H-pyran-2-ones and their ether derivatives, have been found to have a variety of pharmacological properties (Israili & Smissman, 1976; Kretzschmar, Meyer, Teschendorf & Zoellner, 1969). Tominaga and other workers have extensively investigated the addition of enolate anions derived from ketones to doubly active ketene dithioacetals, which are versatile reagents and have been extensively utilized in organic synthesis to afford 4-methylthio-2H-pyran-2-ones (Hatada *et al.*, 1975). We have synthesized several 3-cyano-4-methyl-thio-6-aryl-2H-pyran-2-ones (δ-lactones) as synthons for the synthesis of different classes of heterocycles, such as pyrazoles, isoxazoles and pyrazolo/isoxazolo coumarins and triazoles (Singh *et al.*, 1995; Kumar, Kumar, Parmar & Errington, 1996). However, lactones having the aryl group possessing an *ortho* substituent are usually difficult to make due to steric hindrance. This paper reports the first preparation of a lactone containing an *ortho*-methoxyphenyl substituent, (I).

(I)   (II)
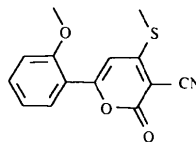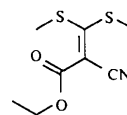
The molecular structure of the title compound is illustrated in Fig. 1. The bond lengths and angles are largely unexceptional (Allen *et al.*, 1987). As previously reported (Azim, Parmar & Errington, 1997), the C_{sp^2}—S distance $[1.734(3) Å]$ is shorter than the C_{sp^3}—S distance $[1.796(3) Å]$ and is indicative of some double bonding in the sp^2 case. The molecule is approximately planar, with the dihedral angle between the two six-membered rings equal to $10.99(12)°$. The ring substituents are approximately aligned with the ring planes as illustrated by torsion angles C7—S1—C3—C2 and C14—O3—C13—C12 of $-177.22(19)$ and
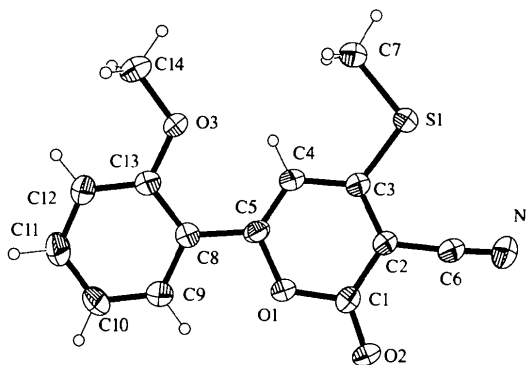

Fig. 1. View of the molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.