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1-(2,4-Diacetoxyphenyl)-2-phenylethanone†

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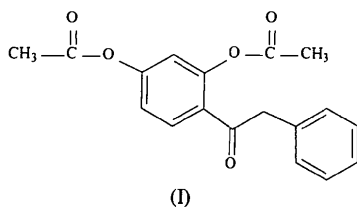
Abstract

The synthesis and structure of the title compound, C₁₈H₁₆O₅, are reported. The two phenyl rings are inclined at an angle of 66.9(1)° with respect to each other; the substituent acetoxy groups are also twisted relative to the central phenyl group.

Comment

The use of differently substituted 1,2-diarylethanones (desoxybenzoins) as starting materials for the synthesis of a variety of biologically active polyphenolics is well documented (Jain, Tyagi & Prasad, 1988). Many of the desoxybenzoins have shown quite significant antimicrobial and antiviral properties (Parmar *et al.*, 1996). Consequently, we have synthesized a series of peracetylated desoxybenzoins for biotransformation studies.

The bond lengths and angles of the title compound, (I), are unexceptional (Allen *et al.*, 1987). The two C—O bond lengths to the phenyl ring are identical within experimental error [1.387(3) and 1.388(3) Å] and are significantly longer than the O4—C17 [1.345(3) Å] and O2—C15 [1.358(3) Å] distances. This is indicative of some double bonding in the latter two cases; the conformation of the acetoxy groups, however, precludes any π interactions with the phenyl ring.



The overall picture is of a very angular molecule; several necessarily planar fragments may be identified, but none of these are coplanar with any other. For example, the best planes through the two phenyl rings are inclined at an angle of 66.9(1)°. Both acetoxy

† Alternative name: 4-(benzylcarbonyl)-1,3-phenylene diacetate.

groups are planar, as expected, but are inclined at angles of 58.9(1) and 82.9(1)° with respect to the central phenyl ring. Finally, the ethanone unit is twisted in relation to the phenyl groups: at 28.1(2)° with respect to the central unit and at 85.4(2)° relative to the peripheral phenyl. Packing diagrams do not reveal any obvious intermolecular hydrogen bonding or π -stacking interactions.

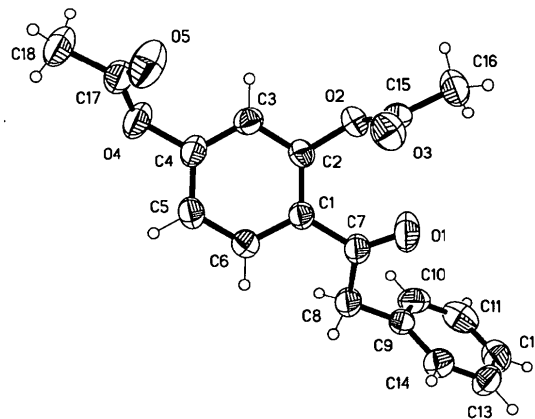


Fig. 1. View of the title molecule showing the crystallographic numbering system. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

A mixture of 2,4-dihydroxydesoxybenzoins (Jain, Arya & Nayyar, 1984) (5 mmol, 1.14 g), acetic anhydride (11 mmol, 1 ml) and concentrated H₂SO₄ (0.1 ml) was stirred at 300 K for 24 h; it was then poured onto crushed ice (50 g) containing concentrated HCl (0.2 ml). The product (I) was obtained as a white solid which recrystallized from chloroform–petrol (1:1) as white needles (1.50 g, 96% yield), m.p. 401 K. IR (Nujol mull) ν_{\max} : 3000, 1770, 1700, 1610, 1460, 1190, 1100, 915 and 720 cm⁻¹. UV (MeOH) λ_{\max} : 386 and 291 nm. ¹H NMR (250 MHz, CDCl₃): δ 2.30 (s, 6H, 2 × OCOCH₃), 4.19 (s, 2H, -COCH₂), 6.96 (d, 1H, *J* = 2 Hz, H₃), 7.06 (m, 1H, H₅), 7.19 (m, 2H, H_{3'} and H_{5'}), 7.28 (m, 3H, H_{2'}, H_{4'} and H_{6'}) and 7.84 (d, 1H, *J* = 8 Hz, H₆). ¹³C NMR (62.9 MHz, CDCl₃): δ 21.01 (2 × OCOCH₃), 47.82 (-COCH₂), 117.35 (C₃), 118.95 (C_{4'}), 126.90 (C₅), 127.77 (C_{1'}), 128.54 (C_{3'} and C_{5'}), 129.37 (C_{2'} and C_{6'}), 130.95 (C₆), 133.83 (C₁), 149.98 (C₂), 153.71 (C₄), 168.20 (OCOCH₃), 168.98 (OCOCH₃) and 196.15 (C=O). EIMS, *m/z* (% int.): 312 [*M*⁺] (72), 270 (100), 228 (63), 179 (20) and 43 (15).

Crystal data

C₁₈H₁₆O₅
M_r = 312.31
 Triclinic
 P $\bar{1}$
a = 8.034(6) Å
b = 9.039(7) Å
c = 11.493(9) Å

Mo K α radiation
 λ = 0.71073 Å
 Cell parameters from 27 reflections
 θ = 5–11°
 μ = 0.100 mm⁻¹
T = 293(2) K

$\alpha = 106.15 (6)^\circ$
 $\beta = 92.75 (6)^\circ$
 $\gamma = 106.74 (6)^\circ$
 $V = 760.2 (10) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.364 \text{ Mg m}^{-3}$
 D_m not measured

Block
 $0.46 \times 0.25 \times 0.20 \text{ mm}$
 Colourless

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1147). Services for accessing these data are described at the back of the journal.

Data collection

Siemens P3R3 diffractometer $\theta_{\max} = 25.05^\circ$
 ω - 2θ scans $h = 0 \rightarrow 9$
 Absorption correction: none $k = -10 \rightarrow 10$
 2905 measured reflections $l = -13 \rightarrow 13$
 2694 independent reflections 3 standard reflections
 1926 reflections with every 200 reflections
 $I > 2\sigma(I)$ intensity decay: none
 $R_{\text{int}} = 0.023$

Refinement

Refinement on F^2 $\Delta\rho_{\max} = 0.26 \text{ e \AA}^{-3}$
 $R[F^2 > 2\sigma(F^2)] = 0.043$ $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$
 $wR(F^2) = 0.124$ Extinction correction:
 $S = 1.043$ SHELXL96 (Sheldrick, 1996)
 2694 reflections Extinction coefficient:
 211 parameters 0.089 (7)
 H atoms not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.1752P]$ Scattering factors from
 where $P = (F_o^2 + 2F_c^2)/3$ International Tables for
 $(\Delta/\sigma)_{\max} = 0.010$ Crystallography (Vol. C)

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

O1—C7	1.196 (3)	O4—C17	1.345 (3)
O2—C15	1.358 (3)	O4—C4	1.388 (3)
O2—C2	1.387 (3)	O5—C17	1.189 (3)
O3—C15	1.188 (3)		
C15—O2—C2—C3	106.3 (2)	C2—C1—C7—O1	28.9 (3)
C15—O2—C2—C1	-81.5 (2)	O1—C7—C8—C9	-15.0 (3)
C17—O4—C4—C3	-60.3 (3)	C1—C7—C8—C9	163.98 (19)

The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). 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: P3/PC Diffractometer Program (Siemens, 1989). Cell refinement: P3/PC Diffractometer Program. Data reduction: SHELXTL-Plus (Sheldrick, 1991). Program(s) used to solve structure: SHELXTL-Plus. Program(s) used to refine structure: SHELXL96 (Sheldrick, 1996). Molecular graphics: SHELXTL-Plus. Software used to prepare material for publication: SHELXL96.

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Dimethyl 9,10-Dihydro-9,10-ethenoanthracene-11-carboxylate-12-S-carbonothioate

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Abstract

The title molecule, C₂₀H₁₆O₄S, contains a dibenzo-barrelene skeleton, with normal geometry and dimensions. The carbomethoxy substituent is almost fully conjugated with the C11=C12 double bond [O=C—C=C torsion angle 171.3 (2)° and cos²(angle) 0.98].

Comment

Previous crystal structure studies of 11,12-derivatives of dibenzobarrelene have given detailed information

† Alternative name: methyl 9,10-dihydro-12-methoxycarbonylthio-9,10-ethenoanthracene-11-carboxylate.