Monoclinic
$P2_1/c$
a = 12.9274 (1) Å
b = 10.7353 (1) Å
c = 20.1194 (2) Å
β = 92.849 (1)°
V = 2788.71 (4) Å^3
Z = 4
D_x = 1.374 Mg m^{-3}
D_m not measured

Cell parameters from 6993 reflections
θ = 1–25°
μ = 0.176 mm^{-1}
T = 143 K
Cube
0.20 × 0.20 × 0.20 mm
Colourless

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996*a*)
T_{min} = 0.733, T_{max} = 1.000
41 669 measured reflections

5723 independent reflections
3841 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 26.52°
$h = -16 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.121
S = 1.063
5723 reflections
393 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.9665P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.269 e Å^{-3}
$\Delta\rho_{min}$ = −0.281 e Å^{-3}
Extinction correction: *SHELXL*96
Extinction coefficient: 0.0059 (7)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond angles (°)*

C1—N1—C12	117.3 (2)	C30—N4—C31	117.6 (2)
C10—N2—C11	117.3 (2)	C41—N5—C52	116.8 (2)
C21—N3—C32	117.2 (2)	C50—N6—C51	121.7 (2)

The data collection nominally covered a sphere of reciprocal space, by a combination of seven sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.95 cm. Coverage of the unique set is over 99% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. The data were corrected for Lorentz and polarization effects. All H atoms were located by difference Fourier synthesis and the H atoms bonded to carbon were refined with fixed individual displacement parameters [U(H) = 1.2U_{eq}(C)] using a riding model with C—H = 0.95 Å. The H6N atom was refined isotropically without any constraints or restraints.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*b*). Molecular graphics: *XP* (Siemens, 1994).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1259). Services for accessing these data are described at the back of the journal.

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6-Acetyl-3,4-dihydro-2,2-dimethyl-2*H*-benzopyran-3,7-diyl Diacetate

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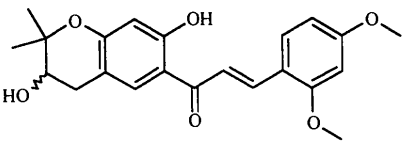
Abstract

The title compound, $C_{17}H_{20}O_6$, is an important precursor in the synthesis of a biologically active chalcone. It contains the dihydropyran unit in a distorted chair conformation, with the 3-acetoxy group arranged in an axial position.

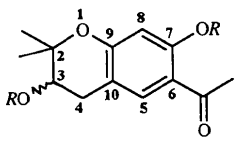
Comment

3-Methyl-2-butenylated (prenylated) phenolics and their cyclic analogues are known to possess a variety of biological activities, *viz* antibacterial (Ahluwalia *et al.*, 1989), antifungal (Bhakuni & Chaturvedi, 1984),

antimicrobial (Ahluwalia *et al.*, 1987), anticancer (Gschwendt *et al.*, 1984), anti-ulcer (Kyogoku *et al.*, 1979) and antifeedant (Simmonds *et al.*, 1990). Recently, we have found that the diarylpropenone (I), carrying an oxidatively cyclized prenyl group, displays very strong anti-invasive activity *in vitro* against MCF 7/6 human mammary carcinoma cell lines (Parmar *et al.*, 1994); however, it was cytotoxic at 100 μM concentrations. As the test molecule was racemic, it was desirable to test the hypothesis that one enantiomer exhibits the desired activity whilst the other is cytotoxic. To isolate optically pure forms of (I), it was proposed that both enantiomers of 6-acetyl-3,7-dihyroxy-2,2-dimethyl-2*H*-benzopyran, (II), should first be prepared by the asymmetric deacetylation of 6-acetyl-3,4-dihydro-2,2-dimethyl-2*H*-benzopyran-3,7-diyl diacetate, (III), using lipase in an organic solvent.



(I)



(II) R = H
(III) R = CH_3CO

This paper confirms the molecular structure of the precursor compound (III); it is represented in Fig. 1 together with the atomic numbering scheme. The bond lengths and angles are unexceptional. The dihydropyran ring has a distorted chair conformation, with the O2 and C11 substituents in axial positions and the C12 methyl group in an equatorial alignment. The best plane (r.m.s. deviation 0.185 Å) through the dihydropyran ring is aligned at 6.3 (1)° with respect to the plane of the aromatic ring. The molecules pack together in the lattice with the aromatic rings arranged in parallel layers. No significant short-range intermolecular contacts were noted.

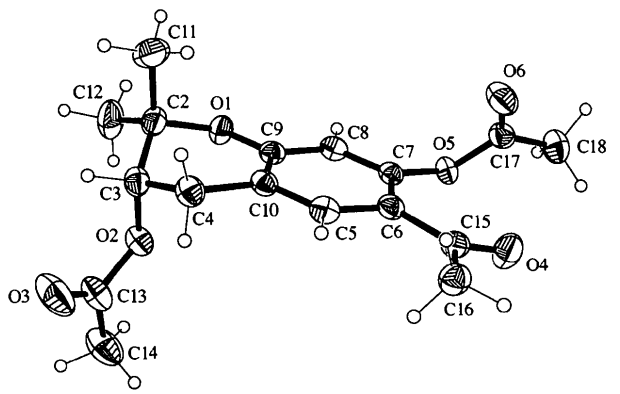


Fig. 1. View of the title molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms have small arbitrary radii for clarity.

Experimental

Compound (II) was prepared according to our procedure reported earlier (Parmar *et al.*, 1989), wherein its m.p. is erroneously reported as 439–440 K; the corrected m.p. of (II) is 396–397 K. A mixture of compound (II) (2.36 g, 0.01 mol), pyridine (2 ml) and acetic anhydride (2.20 ml) was stirred for 20 h at 300 K. The contents were poured onto crushed ice (50 g) and stirred for 30 min, whereupon compound (III) separated out as a white solid; it was recrystallized from chloroform as colourless needles (2.75 g, 86% yield), m.p. 393 K. Elemental analysis: found C 63.63, H 6.30%; $C_{17}H_{20}O_6$ requires C 63.74, H 6.29%. IR (KBr) ν_{max}: 3000, 1780, 1745, 1680, 1625, 1580, 1500, 1480, 1280, 1260, 1200, 1130, 1040, 955 and 880 cm^{-1}. UV (MeOH) λ_{max}: 230 and 275 nm. ^1H NMR (300 MHz, $CDCl_3$): δ 1.33 and 1.36 (2*s*, 3H each, 2 × C-2 CH_3), 2.07 (*s*, 3H, C-6 $COCH_3$), 2.34 (*s*, 3H, C-3 $OCOCH_3$), 2.49 (*s*, 3H, C-7 $OCOCH_3$) 2.82 and 3.15 (2*dd*, 1H each, J = 17.2, 5.0 Hz, C-4 H_a and H_b), 5.05 (*t*, 1H, J = 5.0 Hz, H-3), 6.56 (*s*, 1H, H-8) and 7.59 (*s*, 1H, H-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.41 (C-2 CH_3), 21.58 (C-2 CH_3), 23.58 (C-6 $COCH_3$), 25.33 (C-3 $OCOCH_3$), 28.19 (C-7 $OCOCH_3$), 29.40 (C-4), 70.58 (C-3), 76.96 (C-2), 112.77 (C-8), 116.15 (C-10), 123.45 (C-6), 133.18 (C-5), 150.03 (C-7), 157.67 (C-9), 169.88 (C-3, $OCOCH_3$), 170.79 (C-7, $OCOCH_3$) and 196.02 (C-6 $COCH_3$). MS *m/z* (%int): 320 ($[M]^+$, 4), 260 (20), 250 (63), 236 (28), 218 (46), 217 (44), 203 (69), 180 (40), 179 (70), 165 (61), 147 (23), 107 (10), 59 (24) and 43 (100).

Crystal data

$C_{17}H_{20}O_6$
M_r = 320.33
Triclinic
$P\bar{1}$
a = 5.5650 (7) Å
b = 11.9277 (14) Å
c = 12.7732 (15) Å
α = 77.935 (3)°
β = 84.163 (3)°
γ = 79.965 (3)°
V = 814.6 (2) Å^3
Z = 2
D_x = 1.306 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 2236 reflections
θ = 1.77–25.00°
μ = 0.099 mm^{-1}
T = 190 (2) K
Plate
0.44 × 0.19 × 0.08 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
4234 measured reflections
2797 independent reflections

2019 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25°
h = −6 → 6
k = −14 → 14
l = −14 → 15

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.140$
$S = 1.043$
2797 reflections
213 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 0.2075P]$ where $P = F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C9	1.359 (3)	O5—C17	1.376 (3)
O1—C2	1.459 (3)	O5—C7	1.394 (2)
O2—C13	1.355 (3)	O6—C17	1.197 (3)
O2—C3	1.455 (3)	C2—C3	1.529 (3)
O3—C13	1.201 (3)	C3—C4	1.515 (3)
O4—C15	1.218 (3)	C4—C10	1.504 (3)
C9—O1—C2	118.48 (17)	O1—C2—C3	109.97 (18)
C13—O2—C3	116.12 (19)	C4—C3—C2	112.23 (19)
C17—O5—C7	118.13 (17)	C10—C4—C3	110.88 (19)
O1—C2—C12	104.53 (19)	O1—C9—C10	123.29 (19)
O1—C2—C11	108.16 (19)	C9—C10—C4	120.6 (2)
C9—O1—C2—C3	−40.6 (3)	O1—C9—C10—C4	0.2 (3)
O1—C2—C3—C4	57.2 (2)	C3—C4—C10—C9	16.7 (3)
C2—C3—C4—C10	−44.8 (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 88% complete to at least 25° in θ. The absence of crystal decay was established by repeating the initial frames at the end of the data collection and analysing 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 (1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached. S.u.'s on C—C distances do not exceed 0.004 Å. The bond-length distribution confirms the bond orders shown in the Scheme.

Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994*b*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). 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 *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1179). Services for accessing these data are described at the back of the journal.

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1-(3,4-Dimethoxy-α,β-dihydrocinnamoyl)-pyrrole,† a Novel Amide from *Piper brachystachyum*

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Abstract

The isolation and structure of the title compound, $C_{15}H_{17}NO_3$, are described. The molecule is twisted so that the two ring systems are oriented at an angle of

† Systematic name: 1-[3-(3,4-dimethoxyphenyl)propanoyl]pyrrole.