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5-Acetylgoniothalamin

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Key indicators

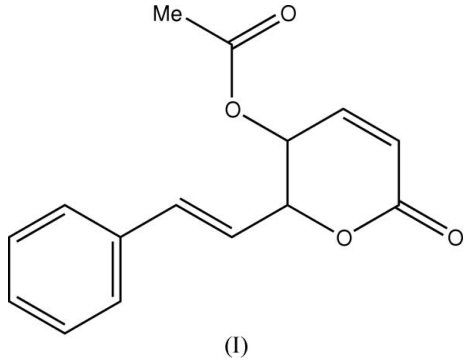
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.106
Data-to-parameter ratio = 8.5

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

In the title compound, $C_{13}H_{10}O_2$, the pyrone ring adopts a modified screw-boat conformation and a *trans* configuration about the C=C double bond. The styryl fragment is pseudo-equatorial on the pyrone ring. The molecules are stabilized by intermolecular C—H···O interactions and form a one-dimensional zigzag chain parallel to [100].

Comment

Biologically active styrylpyrone derivatives such as (+)-goniothalamin (Jewers *et al.*, 1972) and (+)-goniothalamin epoxide (Sam *et al.*, 1987) are among the dominant compounds originally isolated from the genuses *Cryptocarya* (Hlubucek & Robertson, 1967) and '*Goniothalamus*' (Airy-Shaw, 1966). 5-Acetyl goniothalamin (Fig. 1), a styryl-dihydropyrone, was first reported as the second major component after goniothalamin, isolated from the species *G. uvaroides* King, collected from Sabah, Malaysia (Ahmad *et al.*, 1991).



(I)

In the present structural investigation, the title compound, (I), was isolated from the same genus but of species *G. ridleyi* King. The molecular structure of (I) is typical of $\alpha\beta$-unsaturated δ-lactones and similar to 5,6-dihydro-6-styryl-2-pyrone, (II) (Clarke & Pauling, 1975). The replacement of an H atom by the acetyl group at atom C5 of the pyrone ring showed no significant effect on the bond lengths of the ring except that the C4—C5—C6, C3—C4—C5 and C4—C3—C2 bond angles (Table 1) are slightly smaller than those in (II) [110.7 (4), 120.6 (5) and 122.7 (4)°, respectively]. The conformation of the ring is a modified screw-boat as indicated by the non-planarity of the ring, with a maximum deviation of 0.328 (3) Å for atom C6 from the least-squares plane of the O1/C2–C6 fragment. Atom C3 deviates by 0.161 (3) Å on the same side as atom C6. Atoms C5 [deviation 0.275 (3) Å], O1 [deviation 0.127 (2) Å] and C2 [deviation 0.115 (2) Å] are on the oppo-

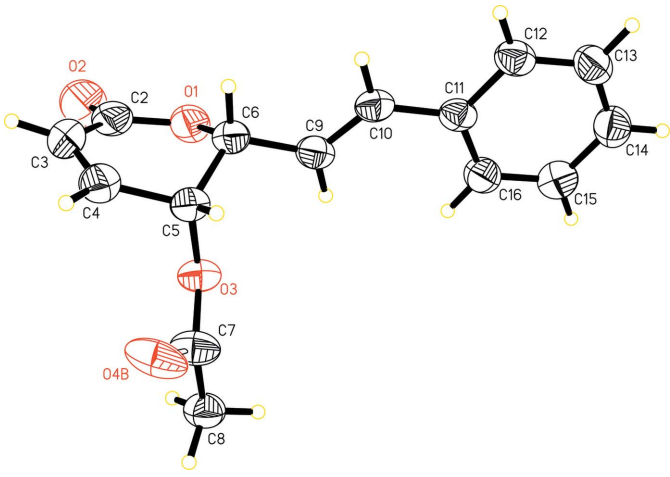


Figure 1
Molecular structure of (I), with 50% probability displacement ellipsoids. The minor component of the disorder has been omitted.

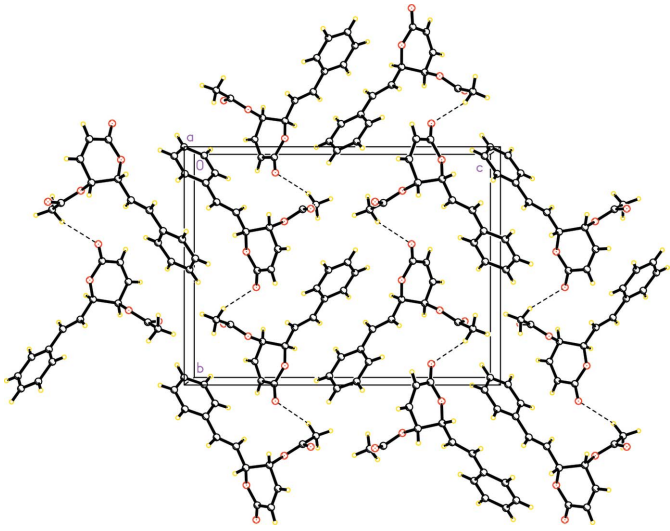


Figure 2
Packing diagram of the title complex, viewed down the *a* axis. The dashed lines denote C—H···O hydrogen bonds.

site side of the least-squares plane. The strain-relieving factor of the $\alpha\beta$-unsaturated δ-lactone indicated by the torsion angle O2—C2—C3—C4 of 157.3 (4)° is less than that in (II) [169.3 (6)°]. The molecule maintains its *trans* configuration about the C9=C10 bond and the pseudo-equatorial position of the planar styryl ring C9—C16 [maximum deviation 0.022 (2) Å for C10 atom] on the pyrone ring. The two rings are approximately perpendicular. This is in contrast to dehydrogoniothalamin (Din *et al.*, 2004), where the whole molecule is essentially planar. The crystal structure is stabilized by weak C—H···O molecular interactions (Table 2), forming zigzag polymeric chains parallel to the *b* axis (Fig. 2).

Experimental

Goniothalamus ridleyi King was collected from Gua Musang, Kelantan, East of Peninsular Malaysia. Stem bark powder (420 g) was extracted in petroleum ether (2 l) in a Soxhlet apparatus for 36 h. Yellow crystals were obtained at the bottom of the Soxhlet flask. The crystals were washed with petroleum ether and recrystallized from ethyl acetate to afford colourless crystals (1.2 g, 0.3%), with melting point 391–392 K. ^{1}H NMR ($CDCl_3$, p.m.m.): δ 7.26 (*m*, 5 H, aromatic), 6.90 (*dd*, *J* = 10 and 5 Hz, H-4), 6.80 (*dd*, *J* = 15 and 1 Hz, H-8), 6.20 (*dd*, *J* = 10 and 2 Hz, H-3), 6.16 (*dd*, *J* = 15 and 6 Hz, H-7), 5.26 (*dd*, *J* = 6 and 3 Hz, H-6), 5.15 (*dd*, *J* = 5 and 3 Hz, H-5) and 2.02 (*s*, 3 H, –OMe). ^{13}C NMR ($CDCl_3$, p.m.m.): δ 170.2 (*s*, ester carbonyl), 162.6 (*s*, C-2), 140.8 (*d*, C-4), 135.7 (*d*, C-9), 135.0 (*d*, C-7), 128.8 (*d*, C-11 dan C-13), 128.7 (*d*, C-12), 126.9 (*d*, C-10 and C-14), 125.0 (*d*, C-8), 121.2 (*d*, C-3), 79.2 (*d*, C-6), 64.0 (*d*, C-5) and 20.7 (*q*, Ac).

Crystal data

$C_{15}H_{14}O_4$
M_r = 258.26
Orthorhombic, $P2_12_12_1$
a = 5.3798 (12) Å
b = 13.659 (3) Å
c = 18.153 (4) Å
V = 1333.9 (5) Å^3
Z = 4
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colourless
0.49 × 0.33 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.955, T_{max} = 0.983
7462 measured reflections
1551 independent reflections
1234 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.106
S = 1.12
1551 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.207P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.347 (4)	C4—C5	1.487 (4)
O1—C6	1.459 (4)	C5—C6	1.505 (4)
O2—C2	1.201 (5)	C6—C9	1.484 (4)
C2—C3	1.469 (6)	C7—C8	1.457 (5)
C3—C4	1.308 (5)	C9—C10	1.312 (4)
C4—C3—C2	121.3 (3)	C4—C5—C6	108.6 (3)
C3—C4—C5	119.8 (4)		
C6—O1—C2—O2	174.5 (3)	C2—C3—C4—C5	6.3 (6)
C6—O1—C2—C3	−9.1 (4)	C3—C4—C5—C6	30.1 (5)
O1—C2—C3—C4	−18.9 (6)	C4—C5—C6—O1	−54.3 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O4*B*^{i}	0.96	2.59	3.120 (8)	115
C8—H8*B*···O4*B*^{ii}	0.96	2.59	3.382 (6)	140
C8—H8*C*···O2^{iii}	0.96	2.43	3.315 (4)	153

Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

Atom O4 is disordered and the site-occupation factors of the disorder components O4*A* and O4*B* refined to 0.49 (2) and 0.51 (2), respectively. In the absence of significant anomalous scattering, Friedel pairs were averaged. H atoms were positioned geometrically with C—H = 0.93–0.98 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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