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Dehydrogoniothalamin

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The title compound, $C_{13}H_{10}O_2$, has a *trans* configuration about the C=C double bond of the phenylethylene side chain and the molecule is nearly planar, with a dihedral angle between the pyrone and benzene rings of 8.54 (6)°. The crystal packing is stabilized by intermolecular C—H···O interactions, which form one-dimensional zigzag chains parallel to [100].

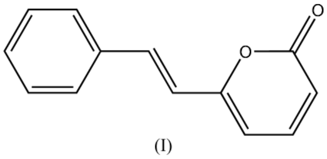
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.126
Data-to-parameter ratio = 16.9

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

Comment

Most of the α-pyrone derivatives extracted from plants have either an epoxidized or an unepoxidized side chain attached to the pyrone moiety. For an unepoxidized side chain, the hydroxyl and acetoxyl groups are the common substituents (Boalino *et al.*, 2003; Aycard *et al.*, 1993; Pereda-Miranda *et al.*, 1993). Strictifolione [$C_{19}H_{24}O_4$, 6-(4′,6′dihydroxy-8′-phenyloct-1′-enyl)-5,6-dihydro-2*H*-pyran-2-one] extracted from *Cryptocarya strictifolia* (Juliawaty *et al.*, 2000) is an example of a 6-substituted 5,6-dihydro-2-pyrone. The title compound, (I), $C_{13}H_{10}O_2$, is also a 6-substituted 2-pyrone but with phenylethylene as the side-chain substituent (Fig. 1) and a C5=C6 bond with double-bond character [1.3440 (19) Å]. The whole molecule is essentially planar, with a maximum deviation from the mean plane of 0.141 (2) Å for atom C11. The pyrone group, including atom C7, *i.e.* O1/O2/C2–C7, is planar [maximum deviation of 0.058 (1) Å for atom C3] and makes a dihedral angle of 8.54 (6)° with the phenyl ring (C9–C14). The bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987) and in agreement with those of α-pyrone (Blake *et al.*, 1994) and other 6-substituted derivatives (Thailambal & Pattabhi, 1985). The molecule has a *trans* configuration about the C7=C8 double bond. In the crystal structure, the molecules are linked by intermolecular C—H···O interactions (Table 2) to form zigzag chains parallel to the *a* axis (Fig. 2).



(I)

Experimental

A *Goniothalamus tortilipetalus* plant was collected from The National Park, Marapoh, Pahang, Peninsular Malaysia, and the voucher specimen (KMS 4732) was deposited at the herbarium of Universiti Kebangsaan Malaysia (UKMB). The air-dried and powdered stembark of *G. tortilipetalus* (dry weight 1 kg) was percolated exhaustively in MeOH (2.0 l) over a period of 5 d at room temperature. The

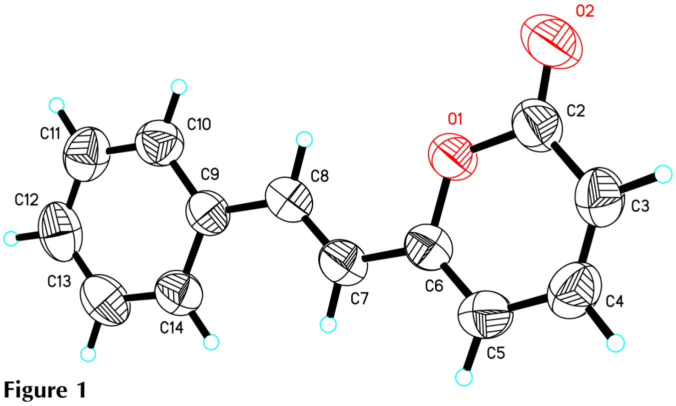


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids.

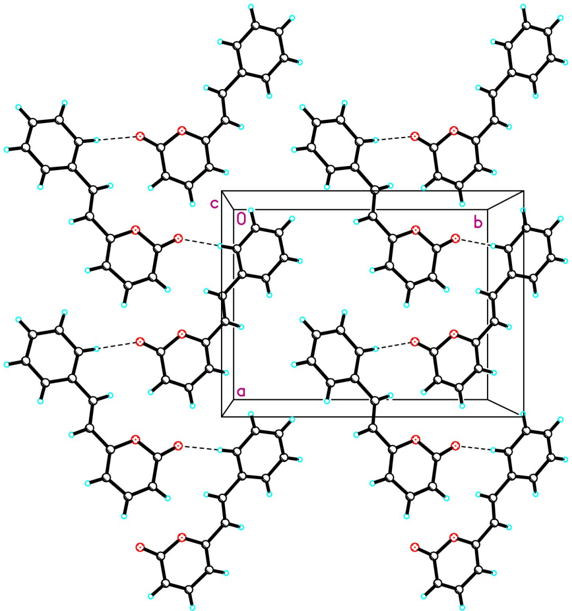


Figure 2
A packing diagram of (I), viewed down the *c* axis. The dashed lines indicate short intermolecular C—H···O contacts.

mixture was filtered and concentrated under reduced pressure, to give a crude extract (100 g, 10%). The methanol extract was subjected to flash chromatography followed by column chromatography using silica as the stationary phase and a mixture of petroleum ether and ethyl acetate as eluant. All the fractions containing dehydrogoniothalamin (R_f 0.8, petroleum ether–ethyl acetate 7:3) were combined and concentrated by rotary evaporation to give a yellow crystalline solid (1.2 g, 0.12%). Crystals suitable for X-ray investigation were obtained by recrystallization from ethyl acetate. The melting point of 388–390 K is in agreement with the literature value of 389 K (Hlubecek & Robertson, 1967).

Crystal data

$C_{13}H_{10}O_2$
$M_r = 198.21$
Orthorhombic, *Pbca*
$a = 10.0406\ (15)$ Å
$b = 13.420\ (2)$ Å
$c = 15.040\ (2)$ Å
$V = 2026.5\ (5)$ Å^3
$Z = 8$
$D_x = 1.299$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3875 reflections
$\theta = 2.7$–$27.5°$
$\mu = 0.09$ mm^{-1}
$T = 273\ (2)$ K
Block, colourless
0.50 × 0.46 × 0.43 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.957$, $T_{\max} = 0.963$
11 115 measured reflections

2304 independent reflections
1819 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 8$
$k = -17 \rightarrow 16$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.126$
$S = 1.05$
2304 reflections
136 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.3022P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.3650 (15)	C3—C4	1.335 (2)
O1—C2	1.3897 (16)	C4—C5	1.414 (2)
O2—C2	1.2016 (17)	C5—C6	1.3440 (19)
C2—C3	1.426 (2)	C7—C8	1.3311 (19)
C6—O1—C2—O2	178.88 (13)	C3—C4—C5—C6	−0.7 (2)
C6—O1—C2—C3	−0.99 (19)	C4—C5—C6—C7	−177.73 (13)
O2—C2—C3—C4	−177.94 (16)	C2—O1—C6—C5	−0.77 (19)
O1—C2—C3—C4	1.9 (2)		

Table 2
Short C—H···O contacts (A, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1	0.93	2.43	2.7806 (16)	102
C10—H10*A*···O2$^{\rm i}$	0.93	2.45	3.381 (2)	176

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

After their location in a difference Fourier map, all H atoms were placed in calculated positions, with C—H distances of 0.93 Å and with $U_{\rm iso}$(H) values constrained to be $1.2U_{\rm eq}$ of the carrier atom.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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