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8,9-Dihydro-1,6-dimethylphenanthro[1,2-*b*]-furan-10,11-dione

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Key indicators

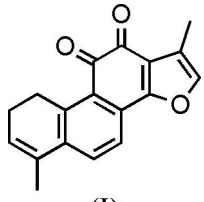
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.126
Data-to-parameter ratio = 15.2

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

The title compound, $C_{18}H_{14}O_3$, also known as 1,2-dihydrotanshinquinone, contains a four-ring system. No classical hydrogen bonds are found in the structure.

Comment

1,2-Dihydrotanshinquinone is one of the tanshinones isolated from the root of the Chinese traditional herb, *Salvia miltiorrhiza*, which has broad pharmacological activities, such as antibacterial (Fang *et al.*, 1976), antitumour (Ryu *et al.*, 1997) and antiplatelet aggregation (Onitsuka *et al.*, 1983). Tanshinones have been used to treat coronary diseases (Chang *et al.*, 1991), cerebrovascular and neurasthenic insomnia problems (Yagi *et al.*, 1989). The structure of 1,2-dihydrotanshinquinone (or 8,9-dihydro-1,6-dimethylphenanthro[1,2-*b*]furan-10,11-dione), (I), was previously elucidated on the basis of spectroscopic analysis (Feng & Li, 1980). We report here its crystal structure.



(I)

The X-ray study of (I) confirms the previously proposed structure based on the spectroscopic data. The asymmetric unit of (I) consists of one independent molecule (Fig. 1). No classical hydrogen bonds are found. The molecular skeleton consists of a four-ring system in which the furan ring is essentially coplanar with the fused six-membered ring, with a torsion angle C15—C13—C14—O1 = −0.75 (15)° (Table 1), while the terminal six-membered ring is in a twist-boat form, with torsion angles C10—C1—C2—C3 = −46.82 (16)° and C3—C4—C5—C10 = −17.63 (19)° (Table 1).

Experimental

Compound (I) was isolated from the root of *Salvia miltiorrhiza*. The root was extracted with EtOH and concentrated *in vacuo*. The residue was subjected to silica-gel column chromatography, eluting with EtOAc–petroleum ether (5:95 *v*/*v*), yielding the title compound, (I). Crystals of (I) were obtained by slow evaporation of an EtOAc–petroleum ether solution. The compound identity was confirmed by NMR and FAB–MS spectroscopy. ^{1}H NMR (500 MHz; $CDCl_3$): 7.55 (*d*, 1H, J = 8 Hz, H6), 7.42 (*d*, 1H, J = 8 Hz, H7), 7.22 (*s*, *br*, 1H, H16), 6.05 (*t*, *br*, 1H, J = 4 Hz, H3), 3.33 (*t*, 2H, J = 8 Hz,

H1), 2.29 (*m*, 2H, H2), 2.27 (*s*, *br*, 3H, H17), 2.06 (*d*, 3H, *J* = 1 Hz, H18). FAB–MS *m*/*z*: 279 (*M* + 1).

Crystal data

$C_{18}H_{14}O_3$	$Z = 2$
$M_r = 278.29$	$D_x = 1.382$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.9225$ (11) Å	Cell parameters from 999 reflections
$b = 8.0119$ (10) Å	$\theta = 2.8$–$26.0°$
$c = 11.7565$ (14) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 83.979$ (2)°	$T = 293$ (2) K
$\beta = 86.251$ (2)°	Block, red
$\gamma = 64.422$ (2)°	0.50 × 0.41 × 0.21 mm
$V = 669.22$ (15) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2897 independent reflections
	2409 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.013$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.1°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.955$, $T_{max} = 0.980$	$k = -10 \rightarrow 10$
7564 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.1042P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	
$wR(F^2) = 0.127$	
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2897 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.3545 (14)	C13—C14	1.3617 (17)
O1—C16	1.3779 (17)	C13—C15	1.4365 (19)
C8—C7	1.3875 (16)	C13—C12	1.4434 (17)
C8—C9	1.4198 (15)	C11—C12	1.5628 (19)
C8—C14	1.4394 (18)	O2—C12	1.2126 (15)
C9—C10	1.4032 (17)	C6—C7	1.3722 (18)
C9—C11	1.4949 (16)	C1—C2	1.5174 (19)
O3—C11	1.2092 (15)	C4—C3	1.3375 (19)
C10—C5	1.4220 (16)	C4—C18	1.4982 (19)
C10—C1	1.5111 (16)	C2—C3	1.4865 (19)
C5—C6	1.3960 (16)	C16—C15	1.343 (2)
C5—C4	1.4749 (18)	C15—C17	1.495 (2)
C14—O1—C16	105.68 (10)	O2—C12—C11	118.76 (11)
O3—C11—C9	123.76 (12)	C3—C4—C5	119.47 (12)
O1—C14—C13	110.26 (11)	C3—C2—C1	111.73 (11)
O1—C14—C8	121.36 (10)	C4—C3—C2	121.47 (13)
C10—C1—C2	112.52 (10)	C15—C16—O1	112.25 (12)
O2—C12—C13	125.18 (13)		
C10—C5—C4—C3	−17.63 (19)	C5—C4—C3—C2	−2.3 (2)
C10—C1—C2—C3	−46.82 (16)		

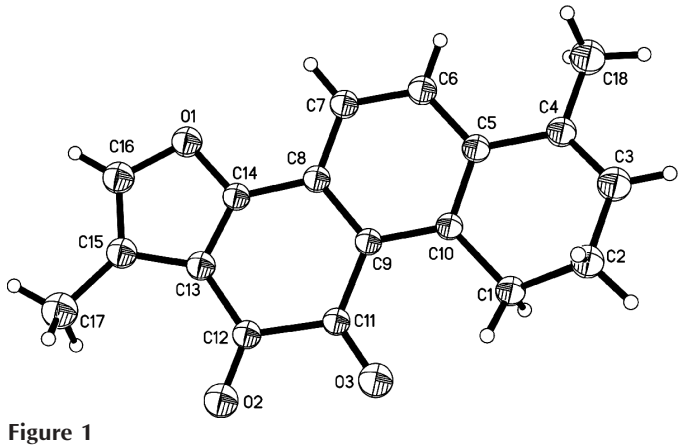


Figure 1
Molecular structure of the asymmetric unit of the title compound, showing 30% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

H atoms were positioned geometrically and were refined as riding on the parent C atoms, with C—H distances of 0.93–0.97 Å [U_{iso}(H) = 1.2U_{eq}(C)].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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