Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Zhi-Yun Du,[a] Jiang-Ke Qin,[a] Xue-Pu Mao,[a] Lin Ma[a] and Lian-Quan Gu[a,b]*

[a]School of Chemistry and Chemical Engineering, Sun Yat-sen University, Guangzhou 510275, People's Republic of China, and [b]School of Pharmaceutical Sciences, Sun Yat-sen University, Guangzhou 510080, People's Republic of China

Correspondence e-mail: jianlonchem@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.104
Data-to-parameter ratio = 7.1

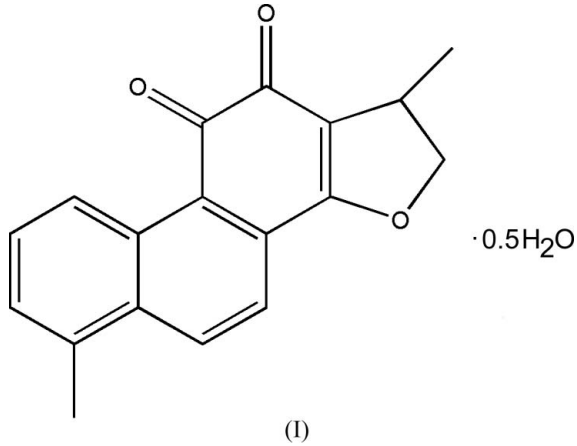
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 August 2005
Accepted 9 August 2005
Online 17 August 2005

In the title compound, $C_{18}H_{14}O_3\cdot0.5H_2O$, also known as dihydrotanshinone hemihydrate, the molecular skeleton is a nearly planar. The two molecules in the asymmetric unit are linked by O—H···O hydrogen bonds involving the carbonyl groups and the solvent water molecule, forming moieties which are, in turn, linked *via* intermolecular π–π stacking interactions into a two-dimensional sheet.

Comment

Dihydrotanshinone is one of the tanshinones isolated from the root of the Chinese traditional herb *Salvia miltiorrhiza* and it has broad pharmacological activities, such as antibacterial (Fang *et al.*, 1976), antitumor (Ryu *et al.*, 1997) and antiplatelet aggregation (Onitsuka *et al.*, 1983). Tanshinones have been used to treat coronary heart disease (Chang *et al.*, 1991), cerebrovascular and neurasthenic insomnia (Yagi *et al.*, 1989). The structure of dihydrotanshinone was elucidated on the basic of spectroscopic analysis (Yang *et al.*, 1981). We have previously reported the crystal structure of 1,2-dihydrotanshinquinone, another tanshinone isolated from the root of *Salvia miltiorrhiza* (Qin *et al.*, 2005). We report here the crystal structure of dihydrotanshinone hemihydrate, (I).



(I)

The X-ray crystallographic study of (I) confirms the molecular structure previously proposed on the basis of spectroscopic data. The asymmetric unit of (I) consists of two independent molecules and one water molecule (Fig. 1). In the crystal structure, the carbonyl group O atoms of two adjacent molecules are linked to one water molecule *via* two O—H···O hydrogen bonds [O···O = 3.070 (4) and 2.991 (4) Å]. Additionally, π–π stacking interactions in the crystal structure result in the formation of infinite two-dimensional sheets (Fig. 2).

Experimental

Dried powder, obtained from the root of *Salvia miltiorrhiza*, was exacted with EtOH and and the extract concentrated *in vacuo*. The residue was subjected to silical-gel coloumn chromatography, eluting with petroleum ether–ethyl acetate (90:10 (*v*/*v*), to yield the title compound. The identity of (I) was confirmed by ^{1}H NMR and FAB mass spectra. Crystals of (I) were obtain by evaporation of a petroleum ether–ethyl acetate solution. ^{1}H NMR in $CDCl_3$ (500 MHz): δ 9.28 (*d*, 9 Hz, 1H), 8.30 (*d*, 9 Hz, 1H), 7.76 (*d*, 9 Hz, 1H), 7.57 (*m*, 1H), 7.40 (*d*, 7 Hz, 1H), 4.97 (*t*, 9.5 Hz, 1H), 4.43 (*q*, 1H), 3.66 (*m*, 1H), 2.70 (*s*, 3H), 1.41 (*d*, 6.5 Hz, 3H). The FAB mass spectrum showed M^{+1} ions at 279.

Crystal data

$C_{18}H_{14}O_3\cdot0.5H_2O$
M_r = 287.30
Monoclinic, $P2_1$
a = 7.287 (2) Å
b = 26.569 (9) Å
c = 7.294 (3) Å
β = 95.590 (7)°
V = 1405.6 (8) Å^3
Z = 4
D_x = 1.358 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 984 reflections
θ = 2.9–26.2°
μ = 0.09 mm^{-1}
T = 293 (2) K
Plate, red
0.48 × 0.42 × 0.17 mm

Data collection

Bruker SMART 1000 CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.984
7831 measured reflections
2782 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°
$h = -4 \rightarrow 8$
$k = -32 \rightarrow 32$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.104
S = 1.03
2782 reflections
392 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.1165P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O5	0.89	2.12	2.991 (4)	164
O7—H7B···O2	0.88	2.26	3.070 (4)	152

In the absence of any significant anomalous scattering, Friedel equivalents were merged prior to the final refinement and the absolute configuration could not be determined. The H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances in the range 0.93–0.98 Å. H atoms attached to water O atoms were located in difference Fourier maps and constrained to ride on their carrier atoms, with O—H distances in the range 0.88–0.89 Å, and with U_{iso}(H) = 1.5U_{eq}(O).

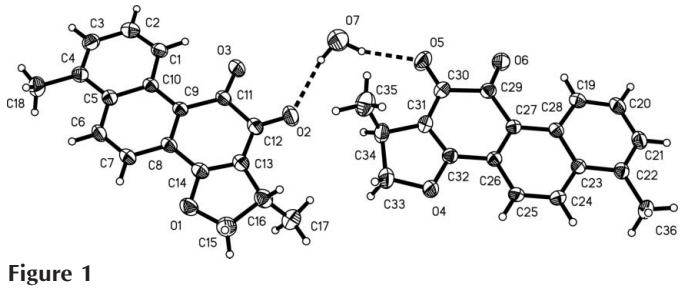


Figure 1
View of the molecules in the asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level.

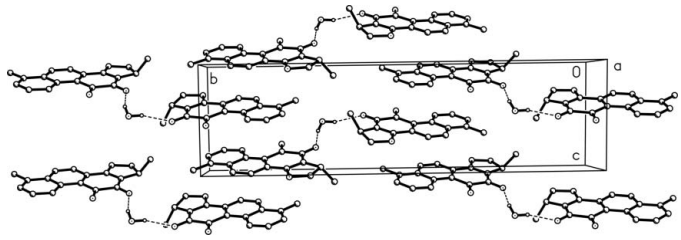


Figure 2
The molecular packing of (I), viewed down the *a* axis; hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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

This work was supported by Guanggong Provincical Science Foundation.

References

Bruker (1999). *SMART* (Version 5.054), *SAINT-Plus* (Version 6.45) and *SHELXTL* (Version 6.14). Bruker AXS Inc, Madison, Wisconsin, USA.
Chang, H. M., Chui, K. Y., Tan, F. W. L., Yang, Y., Zhong, Z. P., Lee, C. M., Sham, H. L. & Wang, H. N. C. (1991). *J. Med. Chem.* **34**, 1675–1692.
Fang, C. N., Chang, P. L. & Hsu, T. P. (1976). *Acta Chim. Sinica.* **34**, 197–209.
Onitsuka, M., Fujiu, M., Shinma, N. & Maruyama, H. B. (1983). *Chem. Pharm. Bull.* **31**, 1670–1675.
Qin, J.-K., Liao, C.-Z., Wang, Y.-T., Bu, X.-Z., Huang, Z.-S. & Gu, L.-Q. (2005). *Acta Cryst.* E**61**, o804–o805.
Ryu, S. Y., Lee, C. O. & Choi, A. U. (1997). *Planta Med.* **63**, 339–342.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Yagi, A., Fujimoto, K., Tanonaka, K., Hirai, K. & Takeo, S. (1989). *Planta Med.* **55**, 51–54.
Yang, B. J., Qian, M. K., Qin, G. W. & Chen, Z. X. (1981). *Acta Pharm. Sinica.* **16**, 837–847.