Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

10-Acetonyl-10-hydroxy-1,6-dimethyl-1,2,10,11-tetrahydrophenanthro[1,2-*b*]-furan-11-one

Ying Chang,[a] Gang Xu,[b] Qin-Shi Zhao,[b] Yang Lu[a]* and Qi-Tai Zheng[a]

[a]Institute of Materia Medica, Chinese Academy of Medical Sciences & Peking Union Medical College, 1 Xiannong tan street, Beijing 100050, People's Republic of China, and [b]State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences,Kunming 650204,Yunnan, People's Republic of China

Correspondence e-mail: luy@imm.ac.cn

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.065
wR factor = 0.179
Data-to-parameter ratio = 8.5

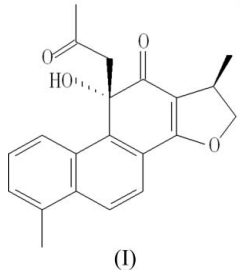
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 June 2006
Accepted 1 July 2006

In the crystal structure of the title compound, $C_{21}H_{20}O_4$, an intramolecular O—H···O hydrogen bond is found between a hydroxyl H atom and a carbonyl O atom.

Comment

The title compound (danshenol A), (I), was extracted from *Salvia yunnanensis* (Xu *et al.*, 2006) and recrystallized from ethanol. As it shows strong aldose reductase (AR) inhibitory activity (Tezuka *et al.*, 1997) we have determined its structure.



(I)

The molecular structure of (I) is shown in Fig. 1. All four rings are coplanar and an intramolecular O—H···O hydrogen bond is found between a hydroxyl H atom and a carbonyl O atom (Fig. 2).

Experimental

The dried and powdered roots of *S. yunnanensis* were extracted three times over 24 h with Me_2CO at room temperature and afterwards the

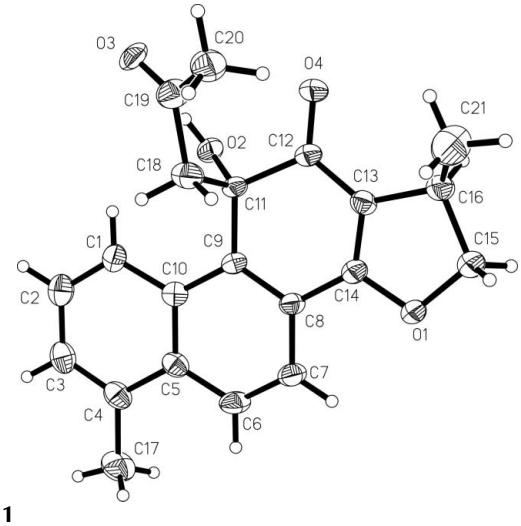


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

solvent was removed *in vacuo*. The residue was subjected to column chromatography over DM-130 porous resin and eluted with MeOH–H_2O (1:1) and 90% MeOH–H_2O (9:1). The residue of the MeOH–H_2O (9:1) fraction was partitioned between H_2O and EtOAc. The EtOAc part was subjected to silica-gel column chromatography. Mixtures of petroleum ether/EtOAc (1:0, 9:1, 8:2, 7:3, 6:4, 5:5 and 0:1) of increasing polarity were used as eluants. Seven fractions were collected and combined by monitoring with thin-layer chromatography. In this procedure, a mixture of danshenol A and danshenol C was obtained from the third fraction by silica-gel column chromatography using petroleum ether–$CHCl_3$–EtOAc (70/25/5) as eluants. Both compounds were separated by semi-preparative high-performance liquid chromatography using 85% MeOH–H_2O as eluant. Crystals of Danshenol A suitable for data collection were obtained by slow evaporation of an ethanol solution over a period of two weeks.

Crystal data

$C_{21}H_{20}O_4$
M_r = 336.37
Monoclinic, $P2_1$
a = 7.5180 (15) Å
b = 6.7150 (13) Å
c = 17.161 (3) Å
β = 102.75 (3)°
V = 845.0 (3) Å^3
Z = 2
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
Column, colorless
0.30 × 0.15 × 0.10 mm

Data collection

MAC DIP 2030K diffractometer
ω scans
Absorption correction: none
7044 measured reflections
1930 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.179
S = 1.09
1930 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1047P)^2 + 0.3521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.081 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O3	0.82	2.24	2.969 (6)	148

The methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, allowed to rotate but not tip and were refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C). All other C—H H atoms were placed in geometrically idealized positions with C—H = 0.92–0.98 Å and were refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C). The O—H H atom was placed in an ideal position such that the O—H vector points in the direction of the nearest acceptor atom and afterwards it was refined using a riding model with U_{iso}(H) = 1.5U_{eq}(O). In the absence of significant anomalous cattering effects, Friedel pairs were averaged.

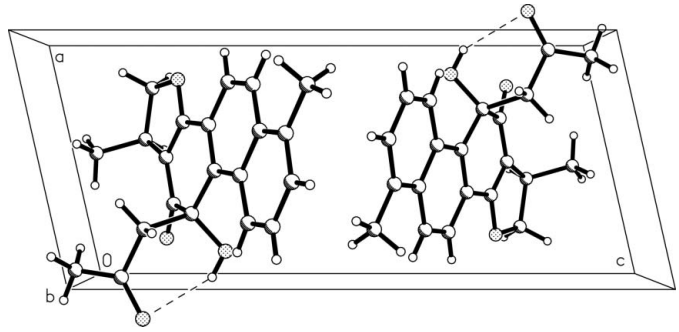


Figure 2
The molecular packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

We acknowledge the financal support of the International Centre for Diffraction Data (ICDD), Pennsylvania, USA.

References

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tezuka, Y., Kasimu, R., Basnet, P., Namba, T. & Kadota, S. (1997). *Chem. Pharm. Bull.* **45**, 1306–1311.
Xu, G., Peng, L. Y., Lu, L., Weng, Z. Y., Zhao, Y., Li, X. L., Zhao, Q. S. & Sun, H. D. (2006). *Planta Med.* **72**, 84–86.