

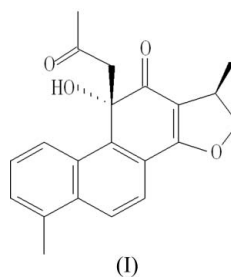
10-Acetyl-10-hydroxy-1,6-dimethyl-  
1,2,10,11-tetrahydrophenanthro[1,2-*b*]-  
furan-11-oneYing Chang,<sup>a</sup> Gang Xu,<sup>b</sup> Qin-Shi  
Zhao,<sup>b</sup> Yang Lu<sup>a\*</sup> and  
Qi-Tai Zheng<sup>a</sup><sup>a</sup>Institute of Materia Medica, Chinese Academy  
of Medical Sciences & Peking Union Medical  
College, 1 Xiannong tan street, Beijing 100050,  
People's Republic of China, and <sup>b</sup>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

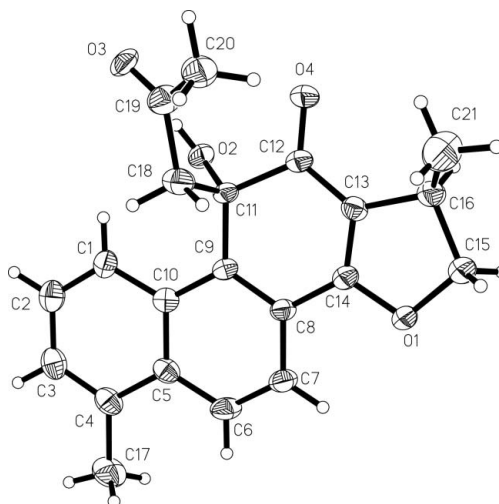
## Key indicators

Single-crystal X-ray study  
*T* = 295 K  
Mean  $\sigma(\text{C}-\text{C})$  = 0.006 Å  
*R* factor = 0.065  
*wR* factor = 0.179  
Data-to-parameter ratio = 8.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the crystal structure of the title compound,  $\text{C}_{21}\text{H}_{20}\text{O}_4$ , an  
intramolecular O—H···O hydrogen bond is found between a  
hydroxyl H atom and a carbonyl O atom.Received 26 June 2006  
Accepted 1 July 2006

## 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.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  $\text{Me}_2\text{CO}$  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<sub>2</sub>O (1:1) and 90% MeOH–H<sub>2</sub>O (9:1). The residue of the MeOH–H<sub>2</sub>O (9:1) fraction was partitioned between H<sub>2</sub>O 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 eluents. 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<sub>3</sub>–EtOAc (70/25/5) as eluents. Both compounds were separated by semi-preparative high-performance liquid chromatography using 85% MeOH–H<sub>2</sub>O 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<sub>21</sub>H<sub>20</sub>O<sub>4</sub> Z = 2  
M<sub>r</sub> = 336.37 D<sub>x</sub> = 1.322 Mg m<sup>-3</sup>  
Monoclinic, P2<sub>1</sub> Mo Kα radiation  
a = 7.5180 (15) Å μ = 0.09 mm<sup>-1</sup>  
b = 6.7150 (13) Å T = 295 (2) K  
c = 17.161 (3) Å Column, colorless  
β = 102.75 (3)° 0.30 × 0.15 × 0.10 mm  
V = 845.0 (3) Å<sup>3</sup>

Data collection

MAC DIP 2030K diffractometer 1930 independent reflections  
ω scans 1894 reflections with I > 2σ(I)  
Absorption correction: none R<sub>int</sub> = 0.048  
7044 measured reflections θ<sub>max</sub> = 27.2°

Refinement

Refinement on F<sup>2</sup> w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.1047P)<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.065 + 0.3521P  
wR(F<sup>2</sup>) = 0.179 where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
S = 1.09 (Δ/σ)<sub>max</sub> = 0.002  
1930 reflections Δρ<sub>max</sub> = 0.28 e Å<sup>-3</sup>  
227 parameters Δρ<sub>min</sub> = -0.22 e Å<sup>-3</sup>  
H-atom parameters constrained 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

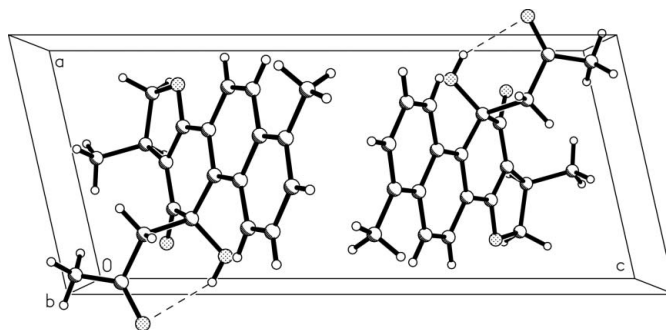


Figure 2 The molecular packing of the title compound, viewed along the b axis. Hydrogen bonds are shown as dashed lines.

riding model, with U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(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<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(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<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(O). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

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 financial 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.