

**(S)-Methyl 6,7,8,9,10,11-hexahydro-1,6-dimethyl-10,11-dioxophenanthro[1,2-*b*]-furan-6-carboxylate (methyl tashinonate)****Jiang-Ke Qin,<sup>a</sup> Ming-Hua Zeng<sup>a\*</sup> and Seik Weng Ng<sup>b</sup>**<sup>a</sup>Department of Chemistry, Guangxi Normal University, Guilin 541000, Guangxi, People's Republic of China, and <sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

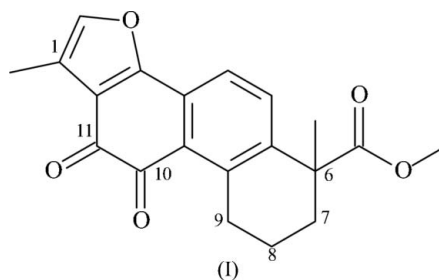
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**Key indicators**Single-crystal X-ray study  
 $T = 295\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.037  
 $wR$  factor = 0.107  
Data-to-parameter ratio = 7.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The approximately planar molecules of methyl tashinonate,  $\text{C}_{20}\text{H}_{18}\text{O}_5$ , are stacked over each other along the  $b$  axis of the monoclinic unit cell. There are two independent molecules in the asymmetric unit.

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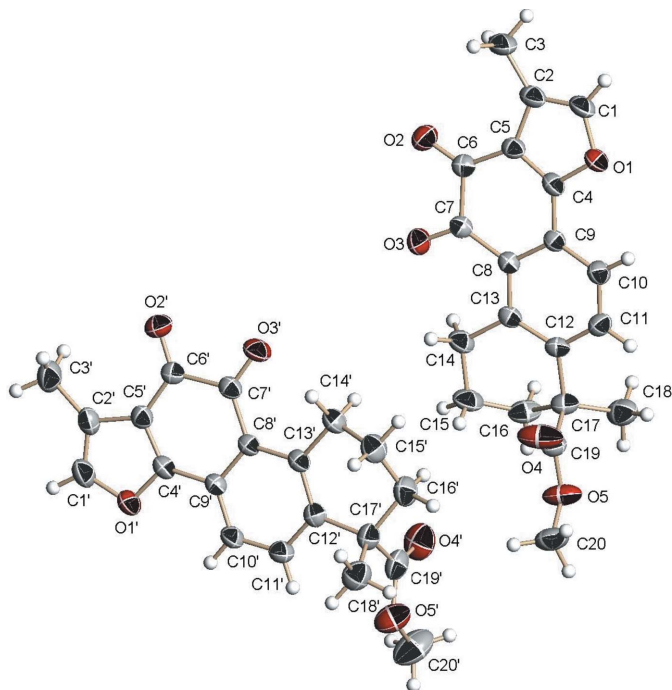
A recent report on the tanshinone isolated from the root of *Salvia miltiorrhiza* details the structure of 8,9-dihydro-1,6-dimethylphenanthro[1,2-*b*]furan-10,11-dione. The red compound exists as a nearly planar molecule that has no chiral C atoms (Qin *et al.*, 2005). The present compound, (I), was also isolated from the same source. As the C atoms at the 6- and 7-positions in (I) are saturated, the compound can be regarded as being the product of the addition of methyl formate across the 6,7 C=C double bond of 8,9-dihydro-1,6-dimethylphenanthro[1,2-*b*]furan-10,11-dione. The structure of methyl tashinonate was first elucidated by spectroscopic means by Kakisawa *et al.* (1968) who assigned an *S* configuration to the chiral carbon.



The compound crystallizes as two independent molecules (Fig. 1) having similar bond dimensions. In the unit cell, the molecules are stacked over each other (Fig. 2), the distance between the nearly planar molecules being about 3.5 Å. The distances between nearly planar molecules of two kinds of independent molecules are similar.

**Experimental**

The root of *Salvia miltiorrhiza* Bunge was dried and then crushed into powder. The powdered root was extracted with ethanol; evaporation of the solvent gave a residue that was subjected to silica-gel column chromatography. The compound was eluted with petroleum ether/ethyl acetate (92:8 v/v). Recrystallization from this solvent system yielded red crystals. The formula was established from the FAB-MS  $m/z$  peak of 339 for the  $[M + 1]^+$  ion, and it was further confirmed by solution NMR measurements.



**Figure 1**  
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

*Crystal data*

$C_{20}H_{18}O_5$   
 $M_r = 338.34$   
 Monoclinic,  $P2_1$   
 $a = 9.184 (1) \text{ \AA}$   
 $b = 13.384 (2) \text{ \AA}$   
 $c = 13.638 (2) \text{ \AA}$   
 $\beta = 105.720 (2)^\circ$   
 $V = 1613.7 (4) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.393 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 853 reflections  
 $\theta = 2.2\text{--}27.1^\circ$   
 $\mu = 0.10 \text{ mm}^{-1}$   
 $T = 295 (2) \text{ K}$   
 Block, red  
 $0.48 \times 0.41 \times 0.34 \text{ mm}$

*Data collection*

Bruker SMART area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 8266 measured reflections  
 3603 independent reflections

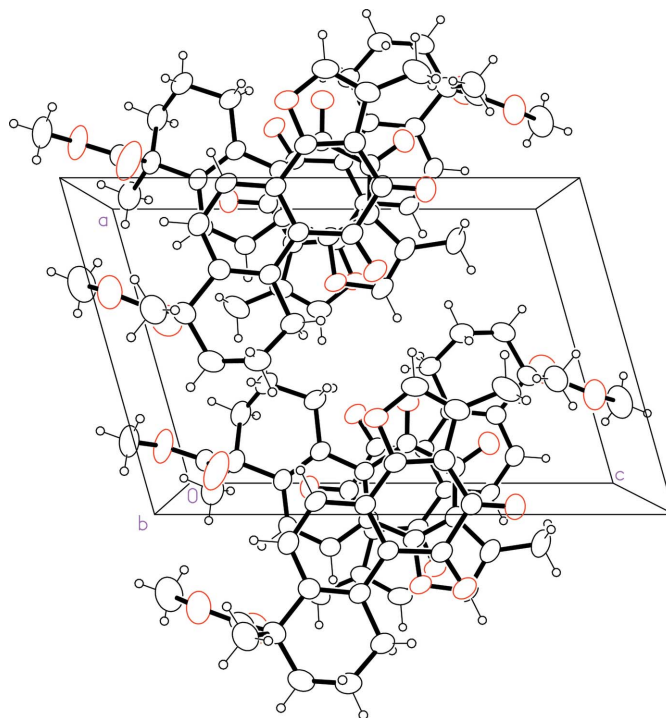
3071 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.021$   
 $\theta_{max} = 27.1^\circ$   
 $h = -11 \rightarrow 8$   
 $k = -16 \rightarrow 17$   
 $l = -14 \rightarrow 17$

*Refinement*

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.107$   
 $S = 1.02$   
 3603 reflections  
 458 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.2301P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.20 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.22 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.023 (2)

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. H atoms were positioned geometrically ( $C-H =$



**Figure 2**  
*ORTEP* (Johnson, 1976) plot illustrating the stacking of the molecules of (I).

0.93 Å for the aromatic atoms, 0.97 Å for the methylene atoms and 0.96 Å for the methyl atoms) and were included in the refinement in the riding-model approximation, with  $U_{iso}(H)$  values set at  $1.2U_{eq}(C)$  for the aromatic and methylene atoms, and to  $1.5U_{eq}(C)$  for the methyl atoms. The methyl groups were rotated to fit the electron density.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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