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**Key indicators**

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
 R factor = 0.038  
 wR factor = 0.096  
 Data-to-parameter ratio = 8.1

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

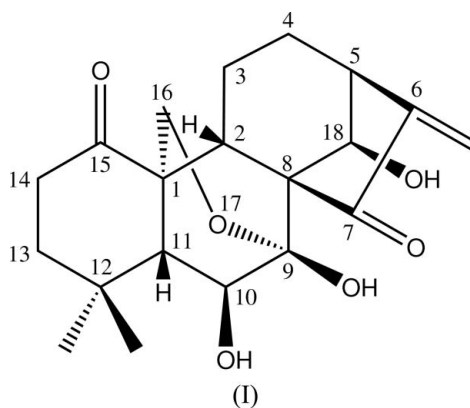
**(1S\*,2S\*,5S\*,9S\*,10S\*,11R\*,18R\*)-9,10,18-  
 Trihydroxy-12,12-dimethyl-6-methylene-  
 17-oxapentacyclo[7.6.2.1<sup>5,8</sup>.0<sup>1,11</sup>.0<sup>2,8</sup>]-  
 octadecane-7,15-dione**

The title compound,  $\text{C}_{20}\text{H}_{26}\text{O}_6$ , was prepared from the natural diterpenoid oridonin and is composed of one five-membered ring with an envelope conformation and five six-membered rings, four with boat conformations and one with a twist-boat conformation. The molecules are assembled into chains *via* hydrogen bonding.

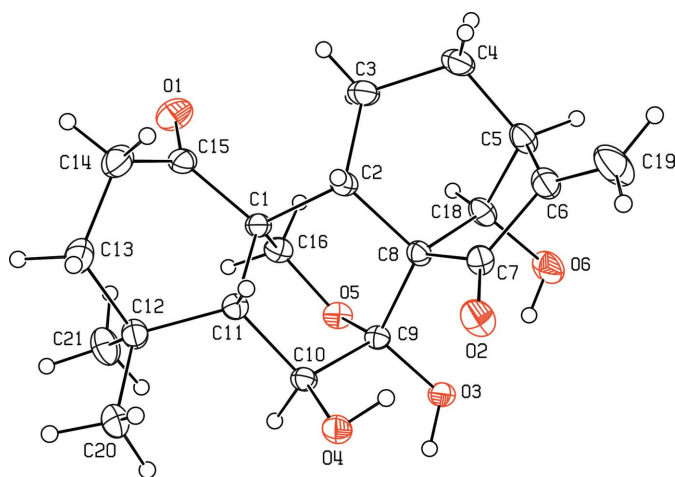
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**Comment**

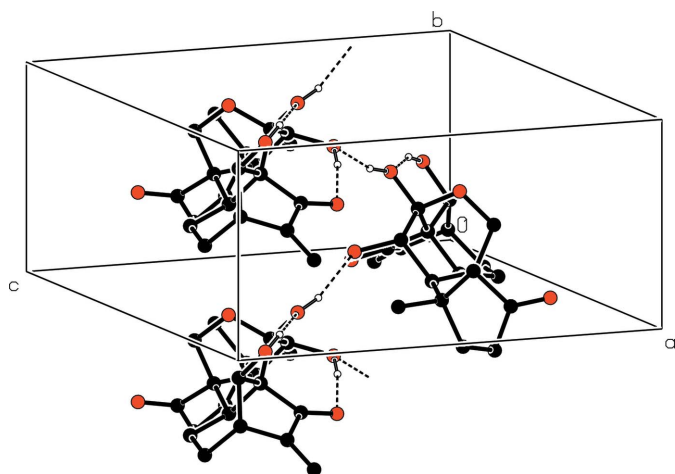
The natural diterpenoid oridonin (Lu *et al.*, 1995; Shi *et al.*, 1992) was found to have antitumor activity (Zuo *et al.*, 2005). In order to investigate the relationship between its structure and activity, a derivative of oridonin, which has an  $\alpha$ -methylene cyclopentanone function, was synthesized and its structure characterized by X-ray diffraction.



The molecule of the title compound, (I) (Fig. 1), is built up from five six-membered rings and one five-membered ring. The cyclohexane ring (C1/C11–C15) adopts a twist-boat conformation, with puckering parameters (Cremer & Pople, 1975)  $Q = 0.701$  (3)  $\text{Å}$ ,  $\theta = 96.7$  (2) and  $\varphi = 273.9$  (2)°, ring C1/C2/C8–C11 exists in a boat conformation [ $Q = 0.838$  (2)  $\text{Å}$ ,  $\theta = 97.73$  (16) and  $\varphi = 178.96$  (17)°], ring C1/C16/O5/C9/C10/C11 adopts a boat conformation [ $Q = 0.805$  (2)  $\text{Å}$ ,  $\theta = 96.23$  (16) and  $\varphi = 242.75$  (16)°], ring C1/C16/O5/C9/C8/C2 adopts a boat conformation [ $Q = 0.854$  (2)  $\text{Å}$ ,  $\theta = 90.77$  (15) and  $\varphi = 68.02$  (15)°], and ring C2–C5/C18/C8 adopts a boat conformation [ $Q = 0.818$  (3)  $\text{Å}$ ,  $\theta = 101.19$  (18) and  $\varphi = 54.78$  (19)°]. The five-membered ring (C5–C8/C18) is a conjugated  $\alpha$ -methylene cyclopentanone ring and adopts an envelope conformation, with puckering parameters  $q_2 = 0.477$  (3)  $\text{Å}$  and  $\varphi_2 = 320.6$  (3)° (envelope on C18). It has been found that this ring in rabdosa diterpenes is highly reactive toward sulfhydryl (thiol) groups essential to the enzyme function (Yamaguchi *et al.*, 1977). As a result of the conjugated  $\alpha$ -methylene cyclo-



**Figure 1**  
Perspective view of the title compound, shown with 30% probability displacement ellipsoids.



**Figure 2**  
The crystal packing diagram for the title compound. Hydrogen bonds are indicated by dashed lines.

pentanone ring, six atoms (C5–C8, C19 and O2) are roughly coplanar, the largest deviation from the mean plane being 0.0234 (15) Å for atom C5.

The three hydroxy groups are involved in hydrogen bonding (Fig. 2 and Table 1) although only atom O6 establishes an intermolecular hydrogen bond with a neighbouring molecule and thus assembles the molecules in chains (running along the *b* axis). The intramolecular hydrogen bonds involving the other hydroxy groups are important for inhibitory activity (Yamaguchi *et al.*, 1977).

## Experimental

Jones reagent (0.5 ml) was added to a solution of oridonin (200 mg; isolated from *Rubdosin rubescens*) in acetone (35 ml). After stirring for 30 min at room temperature, the solution was filtered and added to 24 ml 15% NaHCO<sub>3</sub> in water. The mixture was extracted with 3 ×

30 ml ethyl acetate, washed with 3 × 30 ml water, and dried with anhydrous MgSO<sub>4</sub>. Evaporation of the solvent *in vacuo* left a white residue, which was purified by recrystallization with methanol to give the title compound as colorless crystals.

## Crystal data

C<sub>20</sub>H<sub>26</sub>O<sub>6</sub>  
*M<sub>r</sub>* = 362.41  
 Monoclinic, *P*2<sub>1</sub>  
*a* = 10.8260 (11) Å  
*b* = 6.5434 (7) Å  
*c* = 13.0040 (13) Å  
 $\beta$  = 111.774 (2)°  
*V* = 855.47 (15) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.407 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.10 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism, colorless  
 0.51 × 0.42 × 0.19 mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  
*T<sub>min</sub>* = 0.778, *T<sub>max</sub>* = 1.000

5077 measured reflections  
 2029 independent reflections  
 1850 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.033  
 $\theta_{\max}$  = 27.0°

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.038  
*wR* (*F*<sup>2</sup>) = 0.096  
*S* = 1.03  
 2029 reflections  
 249 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0631P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4...O2	0.89 (3)	1.79 (3)	2.669 (2)	172 (2)
O6—H6...O3	0.822 (19)	1.91 (2)	2.669 (2)	153 (4)
O3—H3...O4 <sup>i</sup>	0.88 (3)	1.92 (3)	2.780 (2)	164 (4)

Symmetry code: (i)  $-x + 1, y + \frac{1}{2}, -z + 1$ .

H atoms of the hydroxy groups were located in a difference map and refined freely. The remaining H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.96 (CH<sub>3</sub>), 0.97 (CH<sub>2</sub>), 0.98 (CH) and 0.93 Å (terminal CH<sub>2</sub>), and with *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(CH<sub>3</sub>) and 1.2*U*<sub>eq</sub>(CH<sub>2</sub> and CH). In the absence of significant anomalous scattering effects, Friedel pairs were merged; only the relative stereochemistry is shown in the title, scheme and figures.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

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