

## Epivernadol diacetate

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## Key indicators

Single-crystal X-ray study

T = 291 K

Mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$ 

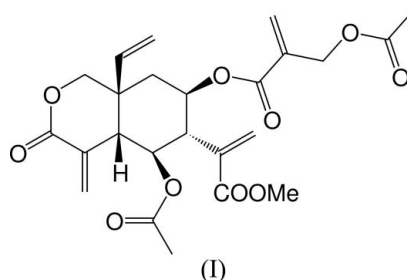
R factor = 0.043

wR factor = 0.115

Data-to-parameter ratio = 8.3

For 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, (4*aR*,5*R*,6*S*,7*S*,8*aS*)-methyl 8*a*-ethenyloctahydro-5-acetoxy-7-[[2-(acetoxymethyl)-1-oxo-2-propenyl]oxy]- $\alpha$ ,4-bis(methylene)-3-oxo-1*H*-2-benzopyran-6-acetate,  $\text{C}_{24}\text{H}_{28}\text{O}_{10}$ , the molecule has a *cis* configuration at the junction of the two six-membered rings and not a *trans* configuration as was assigned on the basis of NMR data in solution [Asaka *et al.* (1977). *Phytochemistry*, **16**, 1838–1839].



## Experimental

Epivernadolol was isolated from an ethanolic extract and acetylated according to the method of Koul *et al.* (2003). It was crystallized from ethyl acetate/petroleum ether (1:4) (m.p. 381 K). MS (diacetate of 2):  $m/z$  at  $M^+$  476 (5), 474 (19), 435 (5), 357 (3), 329 (5), 316 (8), 291 (54), 273 (24), 244 (33), 214 (25), 182 (26), 154 (27), 136 (9), 127 (100).

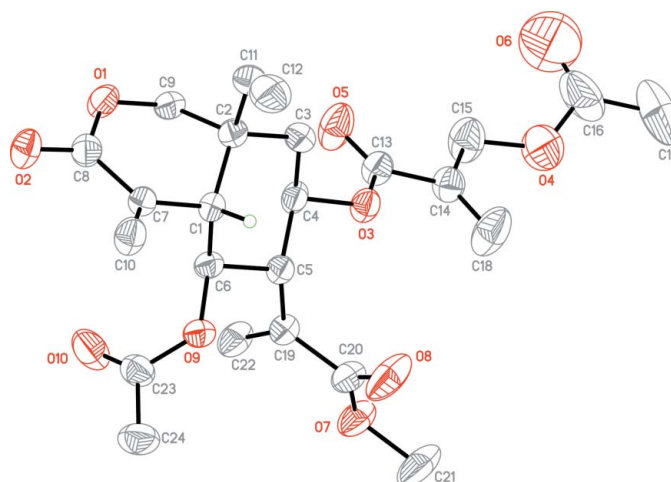


Figure 1

View of the title compound (XP; Sheldrick, 1991), showing the labelling of all non-H atoms. Displacement ellipsoids are drawn at the 30% probability level. H atoms, except H1, have been omitted for clarity.

Crystal data

C<sub>24</sub>H<sub>28</sub>O<sub>10</sub>  
*M<sub>r</sub>* = 476.46  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 8.0887 (2) Å  
*b* = 12.8750 (4) Å  
*c* = 23.8205 (8) Å  
*V* = 2480.71 (13) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.276 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 14723 reflections  
 $\theta$  = 3.0–25.3°  
 $\mu$  = 0.10 mm<sup>-1</sup>  
*T* = 291 (1) K  
 Needle, colourless  
 0.20 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 14723 measured reflections  
 2589 independent reflections  
 1373 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.069  
 $\theta_{\text{max}}$  = 25.3°  
*h* = -9 → 9  
*k* = -15 → 15  
*l* = -28 → 28

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.043  
*wR* (*F*<sup>2</sup>) = 0.115  
*S* = 0.87  
 2589 reflections  
 311 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 ( $\Delta/\sigma$ )<sub>max</sub> < 0.001  
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0057 (15)

H atoms were placed in calculated positions (C–H = 0.93–0.97 Å) with *U*<sub>iso</sub> values constrained to be 1.5*U*<sub>eq</sub> of the carrier atom for the methyl H atoms and 1.2*U*<sub>eq</sub> for the remaining H atoms. The methyl groups were allowed to rotate but not to tip. In the absence of

significant anomalous dispersion effects, Friedel pairs were merged and the absolute configuration was assigned arbitrarily.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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