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(19*R*)-5,19-Bis(acetyloxy)-10,13-dideoxy-10-oxo-5β-enmein

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The title compound, $C_{24}H_{28}O_9$, was prepared from macrocalyxin A and is built up from five fused rings, *viz.* three six-membered rings and two five-membered rings. Two unique molecules are present in the asymmetric unit; both independent molecules have the same absolute configuration, which was deduced from the chirality of the starting material.

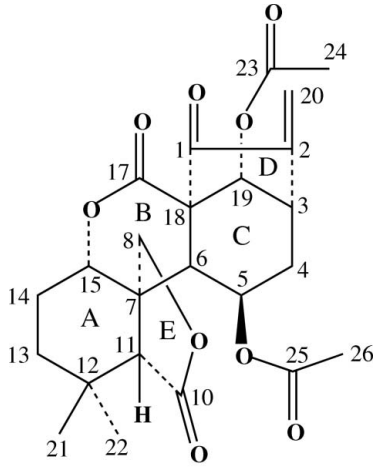
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.103
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.

Comment

Since the natural diterpenoid macrocalyxin A is found to have inhibitory action on *Staphylococcus*, *Bacillus subtilis* and *Candida albicans* (Wang *et al.*, 1984) and exhibits cytotoxicity *in vitro* against cultures of HeLa cells, with a rate of check back of 80% at the concentration of 4 µg ml^{-1} (Cheng *et al.*, 1984), we have derived the title compound from it.



(I)

Two unique molecules are present in the asymmetric unit; both molecule 1 (Fig. 1) and molecule 2 (Fig. 2) are built up from five fused rings, *viz.* three six-membered and two five-membered rings. Some geometrical features of these rings were investigated using *PLATON* (Spek, 2003).

For molecule 1, cyclohexane ring *A* (C7/C11–C15) adopts a chair conformation with puckering parameters (Cremer & Pople, 1975) Q = 0.526 (3) Å, θ = 156.2 (4)° and φ = 23.6 (9)°, ring *B* (O16/C15/C7/C6/C18/C17) exists in a screw-boat conformation [Q = 0.676 (3) Å, θ = 72.1 (3)° and φ = 82.7 (3)°], and ring *C* (C3–C6/C18/C19) adopts a boat conformation [Q = 0.808 (3) Å, θ = 103.4 (2) and φ = 124.1 (2)°]. For the two five-membered rings, ring *D* (C1–C3/C19/C18) adopts an envelope conformation with puckering parameters $Q2$ = 0.445 (3) Å, and $\varphi2$ = 288.0 (5)° (envelope on C19), while ring *E* (O9/C10/

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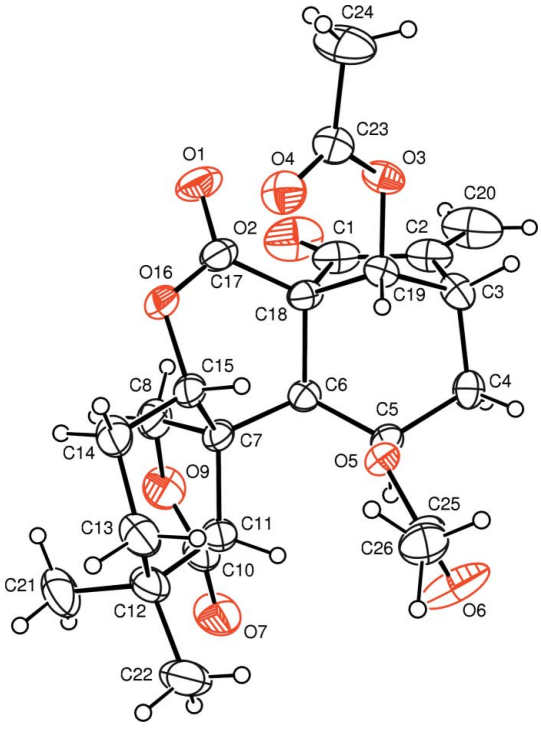

Figure 1
Perspective view of molecule 1 of the title compound, shown with 30% probability displacement ellipsoids.

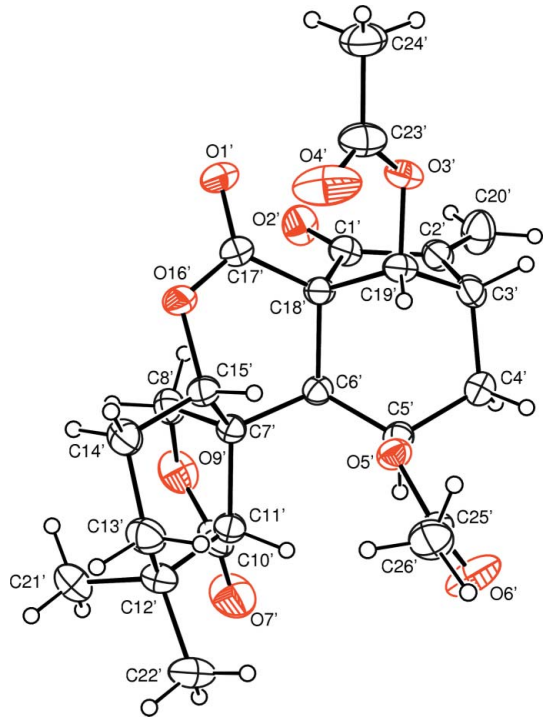

Figure 2
Perspective view of molecule 2 of the title compound, shown with 30% probability displacement ellipsoids.

C11/C7/C8) adopts an envelope conformation with puckering parameters $Q2 = 0.328$ (4) Å and $\varphi2 = 256.4$ (6)° (envelope on C7).

For molecule 2, cyclohexane ring A' (C7′/C11′–C15′) adopts a chair conformation with puckering parameters $Q = 0.520$ (3) Å, $\theta = 158.7$ (3)° and $\varphi = 29.0$ (9)°, ring B' (O16′/C15′/C7′/C6′/C18′/C17′) exists in a screw-boat conformation [$Q = 0.635$ (3) Å, $\theta = 69.3$ (2)° and $\varphi = 85.9$ (3)°], and ring C' (C3′–C6′/C18′/C19′) adopts a boat conformation [$Q = 0.813$ (3) Å, $\theta = 102.06$ (20)° and $\varphi = 128.2$ (2)°]. For the two five-membered rings, ring D' (C1′–C3′/C19′/C18′) adopts an envelope conformation with puckering parameters $Q2 = 0.466$ (3) Å and $\varphi2 = 292.9$ (4)° (envelope on C19′), while ring E' (O9′/C10′/C11′/C7′/C8′) adopts an envelope conformation with puckering parameters $Q2 = 0.337$ (3) Å and $\varphi2 = 259.9$ (6)° (envelope on C7′).

Since the title compound was prepared from the same starting materials (*i.e.* macrocalyxin A) as in the reference (Shi *et al.*, 2003), the configuration can be deduced from the known chirality of the starting material and thus Figs. 1 and 2 represent the correct absolute configuration.

Experimental

With cooling in an ice–water bath, Jones reagent (0.2 ml) was added to a solution of macrocalyxin A (400 mg; isolated from *Rabdosia macrocalyx*) in acetone (15 ml). After stirring for 20 min, the solution was filtered and 240 ml 15% $NaHCO_3$ in water was added. The mixture was extracted three times with diethyl ether (90 ml). After evaporation of the solvent, a white residue was obtained. Recrystallization from methanol gave the title compound as colourless crystals. Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a solution in methanol at room temperature.

Crystal data

$C_{24}H_{28}O_9$
$M_r = 460.46$
Monoclinic, $P2_1$
$a = 13.4851$ (13) Å
$b = 12.7578$ (12) Å
$c = 13.6537$ (13) Å
$\beta = 104.796$ (2)°
$V = 2271.1$ (4) Å^3
$Z = 4$
$D_x = 1.347$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4846 reflections
$\theta = 4.5$–47.6°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.50 × 0.49 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.778$, $T_{max} = 1.000$
13256 measured reflections
5153 independent reflections
3988 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 27.0°$
$h = -17 \rightarrow 16$
$k = -16 \rightarrow 16$
$l = -8 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.103$
$S = 0.93$
5153 reflections
603 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.050$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.96 Å (CH_3), 0.93 and 0.97 Å (CH_2), and 0.98 Å (CH), and with $U_{iso}(H) = 1.5U_{eq}(C)$ (CH_3) and

$1.2U_{eq}$(C) (CH_2 and CH). The two independent molecules have the same absolute configuration, although this could not be determined reliably from the X-ray data, and Friedel reflections were merged.

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); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (1999). *SMART* (Version 5.611), *SAINT* (Version 6.02a) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, P. Y., Lin, Y. L. & Xu, G. Y. (1984). *Yaoxue Xuebao*, **19**, 593–598.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shi, H., Guo, Y. W., Yu, J. L. & Sun, J. (2003). *Z. Kristallogr. New Cryst. Struct.* **218**, 328–330.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wang, X. R., Wang, Z. Q., Dong, J. G. & Xue, Z. W. (1984). *Acta Bot. Sin.* **26**, 425–431.