

Hao Shi

College of Pharmaceutical Science, Zhejiang
University of Technology, Hangzhou 310014,
People's Republic of China

Correspondence e-mail: shihao@126.com

Key indicators

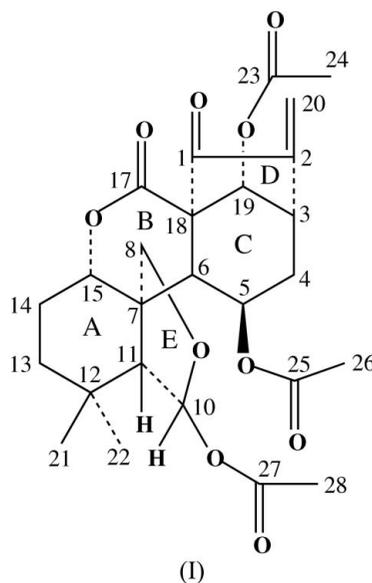
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.045
 wR factor = 0.111
Data-to-parameter ratio = 9.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(3*S*,5*R*,6*S*,7*S*,10*S*,11*R*,15*S*,18*R*,19*R*)-12,12-Dimethyl-
2-methylene-1,17-dioxo-9,16-dioxapentacyclo-
[14.2.1.0^{6,18}.0^{7,11}.0^{7,15}]nonadeca-5,10,19-triyl
triacetate**The title compound, $\text{C}_{26}\text{H}_{32}\text{O}_{10}$, was prepared from macrocalyxin A and is built up from five fused rings, three six-membered and two five-membered. The relative absolute configuration was deduced from the known configuration of the starting material.

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Comment

The molecule of the title compound, (I) (Fig.1), is built up from five fused rings, three six-membered and two five-membered. Some geometrical features of these rings were investigated using *PLATON* (Spek, 2003).

Cyclohexane ring *A* (C7/C11–C15) adopts a chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q = 0.5196$ (33) Å, and $\theta = 158.98$ (35) and $\varphi = 23.1$ (10)°. Ring *B* (O16/C15/C7/C6/C18/C17) exists in a screw-boat conformation, with $Q = 0.6494$ (31) Å, and $\theta = 68.34$ (27) and $\varphi = 89.0$ (3)°. Ring *C* (C3–C6/C18/C19) adopts a boat conformation, with $Q = 0.8173$ (33) Å, and $\theta = 102.02$ (23) and $\varphi = 130.0$ (2)°. For the two five-membered rings, ring *D* (C1–C3/C19/C18) adopts an envelope conformation, with puckering parameters $Q_2 = 0.4560$ (34) Å and $\varphi_2 = 295.2$ (4)°, and ring *E* (O9/C10/C11/C7/C8) adopts an envelope conformation, with puckering parameters $Q_2 = 0.3672$ (32) Å and $\varphi_2 = 260.1$ (5)°.

Since the title compound was prepared from the same starting materials (*i.e.* macrocalyxin A) as the compound reported by Shi *et al.* (2003), the absolute configuration can be deduced from the known configuration of the starting material, and thus Fig. 1 shows this configuration.

Experimental

Macrocalyxin A (50 mg; isolated from *Rabdosia macrocalyx*) was dissolved in a mixture of pyridine (1.5 ml) and Ac_2O (1.5 ml) and the solution was stirred for 24 h at room temperature. MeOH (5 ml) was then added to the mixture and the solution was concentrated *in vacuo* to give a residue that was purified by column chromatography (solvent $\text{CH}_2\text{Cl}_2:\text{CH}_3\text{COCH}_3$ 40:1) to give the title compound. Crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a solution in ethanol at room temperature.

Crystal data

$\text{C}_{26}\text{H}_{32}\text{O}_{10}$

$M_r = 504.52$

Orthorhombic, $P2_12_12_1$

$a = 11.2682$ (7) Å

$b = 14.1695$ (9) Å

$c = 16.0841$ (10) Å

$V = 2568.1$ (3) Å³

$Z = 4$

$D_x = 1.305$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 3033

reflections

$\theta = 4.4\text{--}42.6^\circ$

$\mu = 0.10$ mm⁻¹

$T = 293$ (2) K

Prism, colourless

$0.43 \times 0.41 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 1999)

$T_{\min} = 0.890$, $T_{\max} = 1.000$

15224 measured reflections

3147 independent reflections

2344 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 27.0^\circ$

$h = -14 \rightarrow 13$

$k = -17 \rightarrow 18$

$l = -14 \rightarrow 20$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.111$

$S = 0.96$

3147 reflections

330 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.007$

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.13$ e Å⁻³

H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.96 (CH₃), 0.97 (CH₂) or 0.98 Å (CH), and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{CH}_3)$ or $1.2U_{\text{eq}}(\text{CH}_2, \text{CH})$. In the absence of significant anomalous scattering, Friedel pairs were merged; the absolute configuration has been assigned on the basis of the known configuration of the starting material.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

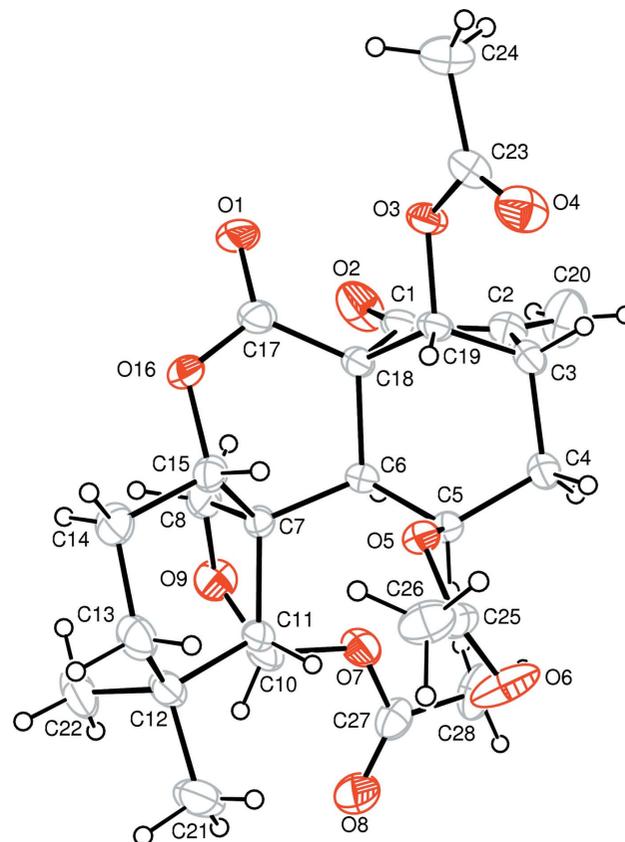


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids.

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