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Alisol B monoacetate from the rhizome of *Alisma orientale*

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.086
Data-to-parameter ratio = 11.5

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

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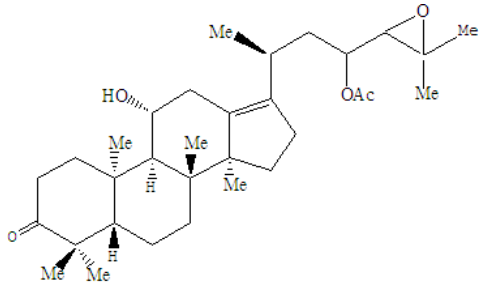
The title compound, alisol B monoacetate or 11-hydroxy-24,25-epoxy-3-oxoprotost-13(17)-en-23-yl acetate, $C_{32}H_{50}O_5$, is a protostane-type triterpenoid which was isolated from the Chinese herbal medicine Alismatis Rhizoma (the rhizome of *Alisma orientalis Juzep*.). The molecule contains three six-membered rings, two of which adopt slightly distorted half-chair conformations while the third exhibits a chair conformation, and one five-membered ring, which adopts an envelope conformation. There is an intermolecular hydrogen bond between the hydroxy and epoxy groups, forming zigzag molecular chains along the *b* axis.

Comment

Alismatis Rhizoma is a crude drug prepared from the dried rhizome of *Alisma orientale Juzep*., and has been used as a folk medicine for diabetes and swellings (diuretics), and as an important component crude drug for several Chinese preparations (Nakajima, 1994). Since 1960, many groups have made phytochemical investigations of the crude drug, including its physiological active principles. The triterpenoids from Alismatis Rhizoma show a relaxant effect on the contraction of isolated aortic or bladder smooth muscles, inhibitory activities for experimental model of type I–IV allergies (Matsuda *et al.*, 1999), and the inhibition of nitrite (a product of NO) accumulation in LPS-activated macrophages. To investigate the bioactive natural products from Alismatis Rhizoma, we investigated its triterpene constituents, which led to the isolation of the title compound, alisol B monoacetate, (I). Its structure was elucidated by spectroscopic investigations, and confirmed by a single-crystal X-ray diffraction analysis.



(I)

The relative stereochemistry of (I) has been determined (Fig. 1 and Table 1). The molecule contains three six-membered rings and one five-membered ring. Rings *A* (C1–C5/C10) and *B* (C5–C9/C10) adopt slightly distorted half-chair conformations owing to the ketone group at C3, and ring *C*

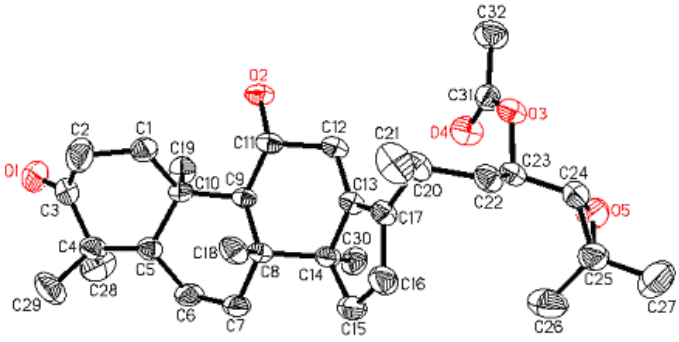


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms have been omitted.

(C8/C9/C11–14) exhibits a chair conformation. Ring *D* (C13–C17) adopts an envelope conformation. All rings are *trans*-fused. The hydroxy and epoxy groups serve as hydrogen-bond donors and acceptors (Table 2), forming zigzag molecular chains along the *b* axis.

Experimental

Dried powder (7.5 kg) of the rhizome of *Astilbe chinensis* was extracted three times with 95% EtOH at room temperature. The solvent was removed by evaporation at reduced pressure, and the residue was successively fractioned with petroleum ether and chloroform. The residue of the chloroform fraction was subjected to column chromatography on silica gel. The column was eluted with a petroleum ether–EtOAc mixture. The crude compound was purified by column chromatography on silica gel with an acetone–chloroform mixture and recrystallized from MeOH to afford 40 mg of the pure title compound, (I). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of an MeOH solution at room temperature (m.p.: 443–445 K). ^{13}C NMR (125 MHz, $CDCl_3$, p.p.m): 220.3 (C3), 170.2 (C31), 138.3 (C17), 134.5 (C13), 70.7 (C23), 70.5 (C11), 65.3 (C24), 58.7 (C25), 57.3 (C14), 48.7 (C9), 48.7 (C7), 47.2 (C4), 41.0 (C8), 37.2 (C22), 37.0 (C10), 34.8 (C12), 34.4 (C1), 33.97 (C2), 31.2 (C16), 30.9 (C15), 29.8 (C29), 29.4 (C7), 28.1 (C20), 25.9 (C21), 25.00 (C27), 24.1 (C19), 23.4 (C18), 21.4 (C6), 20.4 (C28), 20.3 (C30), 20.3 (C32), 19.7 (C26).

Crystal data

$C_{32}H_{50}O_5$
M_r = 514.72
Orthorhombic, $P2_12_12_1$
a = 7.716 (1) Å
b = 14.495 (2) Å
c = 27.526 (5) Å
V = 3078.6 (8) Å^3
Z = 4
D_x = 1.111 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 reflections
θ = 3.1–14.9°
μ = 0.07 mm^{-1}
T = 296 (2) K
Prisms, colorless
0.56 × 0.56 × 0.46 mm

Data collection

Siemens *P*4 diffractometer
ω scans
4120 measured reflections
3990 independent reflections
1961 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 27.5°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 18$
$l = -1 \rightarrow 35$
3 standard reflections
every 97 reflections
intensity decay: 2.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.086
S = 0.80
3990 reflections
346 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0071 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.208 (3)	O5—C24	1.438 (3)
O2—C11	1.427 (3)	O5—C25	1.447 (3)
O3—C31	1.335 (3)	C13—C17	1.325 (3)
O3—C23	1.451 (3)	C24—C25	1.461 (4)
O4—C31	1.200 (3)		
C31—O3—C23	119.0 (2)	O3—C23—C24	107.95 (19)
C24—O5—C25	60.83 (17)	O3—C23—C22	106.53 (19)
C3—C2—C1	117.0 (3)	O5—C24—C25	59.90 (17)
O1—C3—C2	121.2 (3)	O5—C24—C23	117.3 (2)
O1—C3—C4	122.9 (3)	C25—C24—C23	124.9 (3)
C6—C7—C8	116.1 (2)	O5—C25—C24	59.28 (18)
C8—C9—C10	116.34 (17)	O5—C25—C26	113.7 (3)
C5—C10—C1	105.94 (18)	C24—C25—C26	120.9 (3)
C17—C13—C12	130.4 (2)	C24—C25—C27	120.0 (3)
C13—C14—C15	101.81 (19)	O4—C31—O3	124.4 (3)
C17—C16—C15	104.9 (2)	O4—C31—C32	124.7 (3)
C13—C17—C20	127.8 (2)	O3—C31—C32	110.9 (3)
C20—C17—C16	121.5 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O5^{i}	0.82	2.06	2.870 (3)	170

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

After location of H atoms in difference density maps, all H atoms were positioned geometrically and allowed to ride on their attached atoms. The U_{iso} of the hydroxy H atom (H_2O) was refined. The absolute configuration could not be determined because of the absence of significant anomalous effects. Friedel pairs were merged before the final cycles of refinement.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL*/*PC* (Siemens, 1991); software used to prepare material for publication: *SHELXTL*/*PC*.

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