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5,11,11-Trimethyl-16-oxatetracyclo[6.6.2.0^{1,10}.0^{2,7}]-hexadeca-2,4,6-trien-4-ol

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
R factor = 0.084
wR factor = 0.155
Data-to-parameter ratio = 9.7

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

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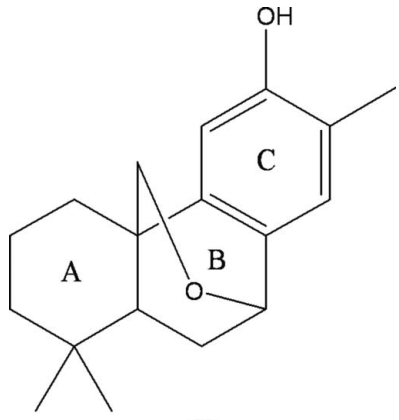
In the title compound, $C_{18}H_{24}O_2$, one of the cyclohexane rings adopts a chair conformation and the other adopts a boat conformation. Molecules are linked by intermolecular O—H···O hydrogen bonds, forming ribbons along the *b* axis.

Comment

Savia przewalskii Maxim is a species endemic to northwestern China which has been used as a substitute in Chinese folk medicine for 'Tan-Shen' (*S. miltiorrhiza*). The title compound, (I), was first acquired from the etheral part of *S. przewalskii*, and its structure was elucidated mainly on the basis of two-dimensional NMR studies (Li *et al.*, 1991). In our case, the title compound, (I), przewalskin, was obtained from *S. yunnanensis*, and the unambiguous crystal structure is reported here for the first time.



(I)

The structure of the title compound (I) is presented in Fig. 1. Bond lengths and angles in (I) are consistent with normal values (Allen *et al.*, 1987). The *A* (C1–C5/C10) and *B* (C5–C10) cyclohexane rings adopt chair [Q_T = 0.520 (7) Å, θ = 10.2 (8)° and φ = 97 (4)° (Cremer & Pople, 1975)] and boat conformations [Q_T = 0.820 (6) Å, θ = 87.8 (4)° and φ = 304.7 (4)°], respectively.

In the structure, molecules of (I) pack in ribbons along the *b* axis, linked by intermolecular O—H···O hydrogen bonds (Table 1). The presence of a number of intermolecular hydrogen bonds and van der Waals forces is responsible for the stability of the structure.

Experimental

The dried and powdered roots (4.7 kg) of *S. yunnanensis* were extracted with Me_2CO (3 × 25 l) at room temperature. The solvent

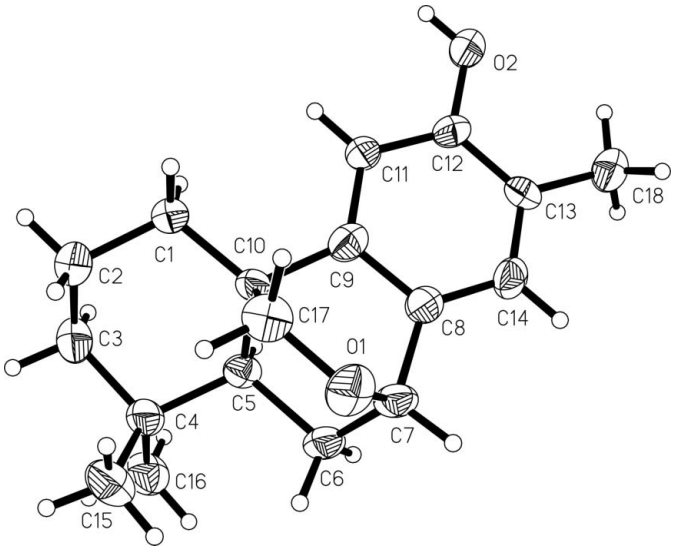


Figure 1
View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

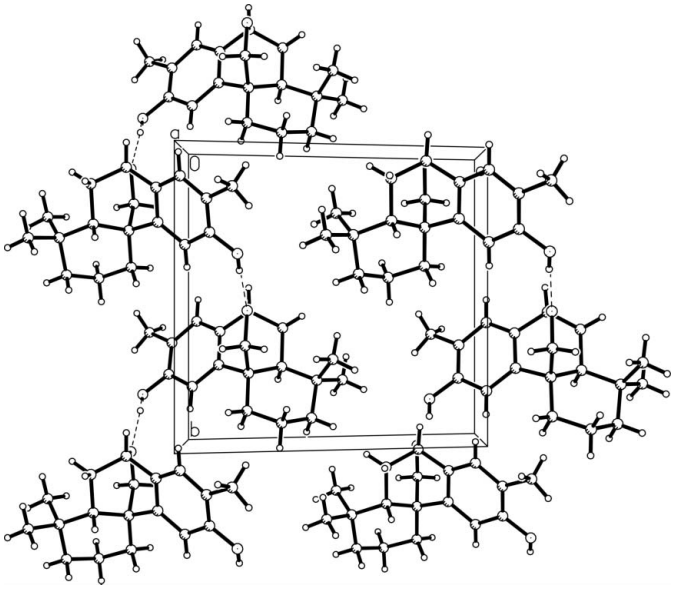


Figure 2
The molecular packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

was removed under vacuum. The gummy residue (200 g) was subjected to column chromatography (12 × 150 cm) over DM-130 porous resin and eluted with MeOH-H_2O (50 and 90%). The residue of the 90% MeOH–H_2O fraction was partioned between H_2O (2.5 l) and EtOAc (2.5 l). The EtOAc part (65 g) was subjected to silica gel column chromatography (9 × 120 cm). Mixtures of petroleum ether/EtOAc (1:0, 9:1, 8:2, 7:3, 6:4, 5:5, and 0:1, each 5 l) of increasing polarity were used as eluants. Seven fractions were collected and combined by monitoring with thin-layer chromatography. The fifth fraction (9 g) was repeatedly chromotographed over RP-18 (7 × 80 cm) using MeOH–H_2O (8:2, 8 l) to give the crude przewalskin, which wasa purified with Sephadex LH-20 (2 × 150 cm) (CH_3OH–$CHCl_3$, 1:1, 0.5 l) to give pure przewalskin (50 mg). Crystals suitable for data collection were obtained by slow evaporation of an ethanol solution at 283 K over a period of 10 d.

Crystal data

$C_{18}H_{24}O_2$
M_r = 272.37
Monoclinic, $P2_1$
a = 6.1900 (12) Å
b = 10.979 (2) Å
c = 11.622 (2) Å
β = 104.88 (3)°
V = 763.3 (3) Å^3
Z = 2
D_x = 1.185 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
Block, colourless
0.60 × 0.40 × 0.20 mm

Data collection

MAC DIP-2030K diffractometer
ω scans
Absorption correction: none
4631 measured reflections
1772 independent reflections
1770 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 27.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.155
S = 1.16
1772 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0114P)^2 + 0.9069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.198 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1^{i}	0.82	1.93	2.750 (6)	174

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

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). The hydroxyl H atom was constrained to an ideal geometry with O—H distances of 0.82 Å and U_{iso}(H) = 1.5U_{eq}(C). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.92–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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