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Key indicators

Single-crystal X-ray study

$T = 295$ K

Mean $\sigma(C-C) = 0.008$ 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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5,11,11-Trimethyl-16-oxatetracyclo[6.6.2.0^1,10.0^2,7^]hexadeca-2,4,6-trien-4-ol

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

Savina 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 ethereal 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.

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, $\theta = 10.2 (8)^\circ$ and $\psi = 97 (4)^\circ$ (Cremer & Pople, 1975)] and boat conformations [$Q_T = 0.820 (6)$ Å, $\theta = 87.8 (4)^\circ$ and $\psi = 304.7 (4)^\circ$], 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$_2$CO (3 × 25 l) at room temperature. The solvent
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-H2O (50 and 90%). The residue of the 90% MeOH–H2O fraction was partitioned between H2O (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 (10, 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 chromatographed over RP-18 (7 × 80 cm) using MeOH–H2O (8:2, 8:1) to give the crude przewalskin, which was purified with Sephadex LH-20 (2 × 150 cm) (CH3OH–CHCl3, 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

\[
\begin{align*}
C_{18}H_{24}O_2 &

\text{Mr} & = 272.37

\text{Monoclinic}, P & 2_1

a & = 6.1900 (12) \text{ Å}

b & = 10.979 (2) \text{ Å}

c & = 11.622 (2) \text{ Å}

\beta & = 104.88 (3)

\text{V} & = 763.3 (3) \text{ Å}^3

Z & = 2

\mu & = 0.08 \text{ mm}^{-1}

T & = 298 (2) \text{ K}

\text{Block, colourless}

\end{align*}
\]

Data collection

\[
\begin{align*}
&\text{MAC DIP-2030K diffractometer} \\
&\text{Absorption correction: none} \\
&1772 \text{ independent reflections} \\
&1770 \text{ reflections with } I > 2 \sigma(I)

\end{align*}
\]

Refinement

\[
\begin{align*}
R(F^2) & = 0.045 \\
\omega R(F^2) & = 0.155 \\
S & = 1.16

\end{align*}
\]

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 \( U_{eq}(C) \). The hydroxyl H atom was constrained to an ideal geometry with O–H distances of 0.82 Å and \( U_{eq}(C) \). 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 \( U_{eq}(C) \).

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

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References


