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Key indicators
Single-crystal X-ray study
T = 288 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 8.3

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

11α,12β-Acetonyltenacissoside F acetone solvate: a new polyoxypregnane glycoside from the stems of Marsdenia tenacissima

The title compound (11α,12β-acetonyltenacigenin B 3-O-deoxy-3-O-methyl-β-d-allopyranosyl-(1→4)-β-d-oleandropyranoside acetone solvate), C38H60O12.C3H6O, was isolated from the stems of Marsdenia tenacissima. It contains five six-membered rings in chair and distorted half-chair conformations, two five-membered rings exhibiting envelope conformations and a three-membered ring. The unit cell (the asymmetric unit) also contains a disordered acetone molecule. The molecules are linked by two intermolecular O—H⋯O hydrogen-bonds.

Comment

The stems of Marsdenia tenacissima (Roxb.) Wight et Arn. is a perennial climber of the family Asclepiadaceae. It is indigenous to the southwest of China and has been shown to have some activity against inflammation, asthma and cancer (Jiangsu New College of Medicine, 1986). Previous chemical investigations on this plant were focused on its polyoxypregnane derivatives (Deng et al., 2005). Some of its glycosides have also been tested for cytotoxicity (Luo et al., 1993; Wang et al., 2006). Our continuing investigation and careful examination of M. tenacissima led to the isolation of a new polyoxypregnane glycoside, (I), that was first characterized in solution by 1H, 13C and two-dimensional NMR experiments and then in the solid state by this X-ray crystallographic study (Fig. 1).

The aglycone unit contains three six-membered rings (A, atoms C1–C5/C10, B, C5–C10 and C, C8/C9/C11–C14), two five-membered rings (D, C13–C17 and F, C11/C12/O3/C22/O2) and a three-membered ring (E, C8/C14/O1). The sugar unit contains two six-membered rings (G, atoms C25–C29/O6 and H, C30–C34/O8). Rings A and B adopt a chair conformation and ring C a distorted half-chair conformation, while the rings of the sugar unit have normal chair conformations. Ring D exhibits an envelope conformation with C14 the out-of-plane atom [0.089 (5) Å]. Ring F also exhibits an envelope conformation with C11 the out-of-plane atom [0.722 (4) Å]. All rings are trans fused except ring E.
In the crystal structure of (I), the molecules are linked by two intermolecular O—H···O hydrogen-bonds (Table 1).

Experimental

The stems of *M. tenacissima* (10 kg), were collected in Yunnan province of China in September 2005. The plant was identified by Professor Zuo-Cheng Zhao (Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, P. R. China). They were boiled in water for 2 h and the water was then evaporated in vacuo. The brown mass (10 kg), were collected in Yunnan province of China in September 2005. The plant was identified by Professor Zuo-Cheng Zhao (Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, P. R. China). They were boiled in water for 2 h and the water was then evaporated in vacuo. The brown mass (10 kg), were collected in Yunnan province of China in September 2005. The plant was identified by Professor Zuo-Cheng Zhao (Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, P. R. China). They were boiled in water for 2 h and the water was then evaporated in vacuo. The brown mass

Crystal data

$$\begin{align*}
\text{Cs}_{13}\text{H}_{60}\text{O}_{15}\cdot \text{C}_{3}\text{H}_{6}\text{O} & \quad \text{M}_0 = 766.94 \\
\text{Triclinic, } P\bar{1} & \quad a = 6.328 (1) \text{ Å} \\
b = 10.558 (2) \text{ Å} & \quad c = 17.553 (4) \text{ Å} \\
\alpha = 74.57 (2)^\circ & \quad \beta = 81.76 (2)^\circ \\
\gamma = 79.03 (2)^\circ & \quad V = 1104.5 (4) \text{ Å}^3 \\
Z & = 1 \\
D_0 & = 1.153 \text{ Mg m}^{-3} \\
\text{Mo } K\alpha \text{ radiation} & \quad \mu = 0.09 \text{ mm}^{-1} \\
T & = 298 (2) \text{ K} \\
\text{Block, colourless} & \quad 0.54 \times 0.54 \times 0.50 \text{ mm} \\
\end{align*}$$

Table 1

<table>
<thead>
<tr>
<th>Hydrogen-bond geometry (Å, °)</th>
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<tr>
<td>D—H···A</td>
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<tr>
<td>O10—H10O···O6i</td>
</tr>
<tr>
<td>O12—H12O···O4ii</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) x + 1, y, z; (ii) x + 1, y − 1, z + 1.

All H atoms were positioned geometrically, with C—H = 0.93—0.98 Å, O—H = 0.82 Å, and $U_{w}(H) = 1.2U_{eq}(C)$. The cocrystallized acetone solvent molecule was severely disordered. Thus we divided the cocrystallized acetone molecules into two parts, each of them restrained within 1.22 Å. In the absence of significant anomalous scattering, Friedel pairs were merged; the absolute configuration was assigned by reference to the chiral molecule of known absolute configuration (Mamoru et al., 1995; Luo et al., 1982) resemble tenuigenin-A.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL*.

We are grateful to the staff of the analytical group of Chengdu Institute of Biology, Chinese Academy of Sciences, for measuring the NMR spectra.

References


Crystal data

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\end{align*}$$