Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2β,24-Dihydroxyurs-12-en-27-oic acid methanol solvate from the rhizome of *Astilbe chinensis*

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.109
Data-to-parameter ratio = 8.2

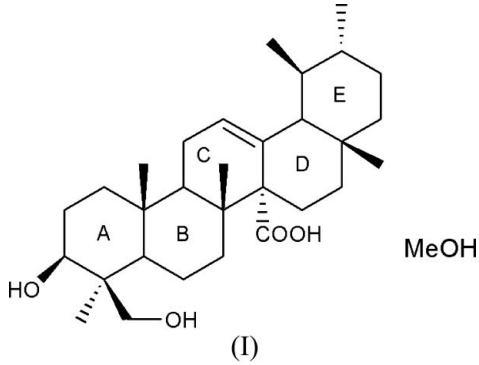
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 24 March 2007
Accepted 28 March 2007

The title compound, a novel triterpenoid, $C_{30}H_{48}O_4\cdot CH_4O$, was isolated from the rhizome of *Astilbe chinensis* (Maxim.) Franch. et Sav. It contains five fused six-membered rings, which adopt half-chair conformations. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules, forming an extensive network, which may be effective in the stabilization of the structure.

Comment

Astilbe chinensis (Maxim.) Franch. et Sav. is a perennial herbaceous plant growing in China, Russia, Japan and Korea. Its rhizome has been used for headache, arthralgia, chronic bronchitis and stomachalgia in traditional Chinese medicine (Pan, 1985, 1995). Pharmacological studies indicated extracts of *A. chinensis* to have antineoplastic and immunopotentiating activities (Chen *et al.*, 1996). Six flavonoids and four triterpenes have been isolated from this plant (Chen *et al.*, 2004; Sun *et al.*, 2003). The study of the bioactive constituents from *A. chinensis* led to the isolation of a novel triterpene, 2β,24-dihydroxyurs-12-en-27-oic acid, (I), from the petroleum ether extract. The structure of (I) was elucidated by extensive spectroscopic analysis, including two-dimensional NMR spectroscopy, and confirmed by single-crystal X-ray diffraction analysis.



(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). It contains five fused six-membered rings [*A* (C1–C5/C10), *B* (C5–C10), *C* (C8/C9/C11–C14), *D* (C13–C18) and *E* (C17–C22)] which are not planar, having total puckering amplitudes, Q_T, of 0.527 (2), 0.576 (3), 0.513 (3), 0.535 (2) and 0.535 (3) Å, respectively, and half-chair conformations [φ = 170.6 (1) and θ = 174.50 (2)° for ring *A*, φ = −162.9 (1) and θ = 170.38 (2)° for ring *B*, φ = −150.9 (2) and θ = 127.05 (2)° for ring *C*, φ = −134.1 (2) and θ = 12.0 (2)° for ring *D*, and φ = −157.5 (2) and

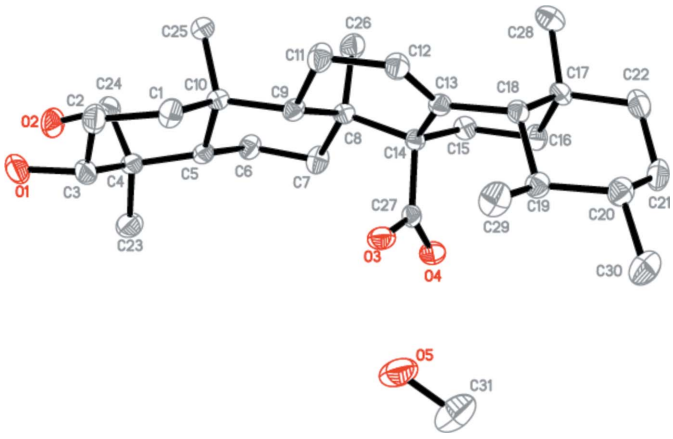


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

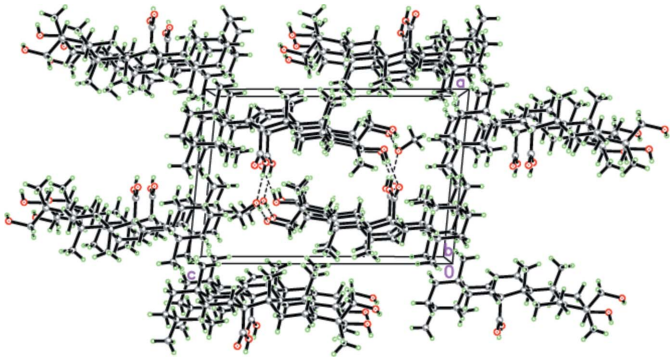


Figure 2
A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

θ = 5.03 (2)° for ring *E*] (Cremer & Pople, 1975). All rings are *trans* fused except for the *D*/*E* junction, which is *cis* fused.

As can be seen from Fig. 2, intermolecular O—H···O hydrogen bonds (Table 1) link the molecules, forming an extensive network, which may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

Astilbe chinensis (Maxim.) Franch. et Sav. was collected in Hefeng, Hubei province of China in November 2004 and identified by Professor Ding-Rong Wan, School of Life Sciences, South-Central University for Nationalities. A voucher specimen (D20040903) has been deposited at the School of Pharmacy, Tianjin Medical University. The rhizomes of *A. chinensis* were dried at room temperature in the dark. The material (4300 g) was extracted three times with ethanol (95%) under reflux. The ethanol extract (1115 g, 95%) was suspended in water, and then extracted with petroleum ether, ethyl acetate and *n*-butanol successively. The petroleum ether layer (49 g) was absorbed on silica gel (130 g) and chromatographed on a silica gel (1000 g) column eluted with petroleum ether–EtOAc with increased polarity to give 15 fractions. Fraction 8 was further separated on a Toyopear HW-40 preparative HPLC-ODS to afford the title compound, (I) (yield 0.038 g; m.p. 508–510 K). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a solution in methanol at room temperature. ^{13}C NMR (300 MHz, CD_3OD): δ 39.9 (C_1), 28.5 (C_2), 81.3 (C_3), 43.7 (C_4), 57.2 (C_5), 19.9 (C_6), 38.5 (C_7), 40.9 (C_8), 48.4 (C_9), 38.0 (C_{10}), 24.1 (C_{11}), 129.5 (C_{12}), 135.1 (C_{13}), 57.5 (C_{14}), 23.7 (C_{15}), 30.5 (C_{16}), 34.8 (C_{17}), 62.0 (C_{18}), 41.1 (C_{19}), 38.9 (C_{20}), 31.8 (C_{21}), 42.3 (C_{22}), 23.3 (C_{23}), 65.4 (C_{24}), 17.4 (C_{25}), 18.8 (C_{26}), 179.1 (C_{27}), 29.8 (C_{28}), 18.6 (C_{29}), 21.7 (C_{30}).

Crystal data

$C_{30}H_{48}O_4\cdot CH_4O$
M_r = 504.73
Monoclinic, $P2_1$
a = 11.068 (3) Å
b = 7.9037 (18) Å
c = 16.677 (4) Å
β = 95.487 (4)°
V = 1452.2 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
0.32 × 0.28 × 0.20 mm

Data collection

Bruker CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.985
7442 measured reflections
2764 independent reflections
2013 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.110
S = 1.02
2764 reflections
336 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4^{i}	0.82	1.91	2.728 (3)	173
O5—H5A···O2^{i}	0.82	1.85	2.667 (4)	178
O2—H2···O1	0.82	1.97	2.645 (5)	138
O3—H3···O5	0.82	1.80	2.618 (3)	173

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

The absolute configuration could not be established because of the absence of significant anomalous effects and has been assigned arbitrarily; Friedel pairs were merged. H atoms were positioned geometrically, with O—H = 0.82 Å (for OH) and C—H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,O), where x = 1.5 for hydroxy and methyl, and x = 1.2 for all other H atoms.

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

This project was sponsored by the Scientific Research Foundation of Tianjin Medical University.

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