Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

($3R^*,6S^*,6aR^*,9S^*,9aS^*$)-6,6a,9-Trihydroxy-3,6,9a-trimethylperhydroazuleno[1,2-*b*]furan-2-one

G. Y. S. K. Swamy,[a]* K. Ravikumar,[a] Biswanath Das[b] and G. Mahender[b]

[a]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [b]Organic Chemistry Division I, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: swamy@ins.iictnet.com

Received 7 December 2004
Accepted 8 December 2004
Online 18 December 2004

Key indicators

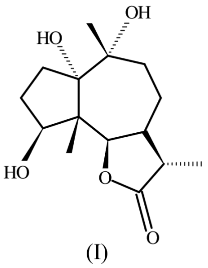
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.087
Data-to-parameter ratio = 8.8

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

The title compound, $C_{15}H_{24}O_5$, was isolated from the flowers of *Parthenium hysterophorus*. The crystal structure determination confirms the relative stereochemistry unambiguously. The packing is stabilized by O—H···O hydrogen bonding.

Comment

Parthenium hysterophrous Linn, an obnoxious weed, is known for its allelopathic effects (Kanchan, 1975; Patel & Hedge, 1988). Furthermore, parthenin is known to possess significant cytotoxic properties (Kupchan *et al.*, 1971). In a continuation of our work (Ramesh *et al.*, 2003) on the constituents of the herb, we report here the crystal structure of a new pseudoguanolide, histerone (I).



(I)

Compound (I) has a cyclopentane ring *trans*-fused and a γ-lactone ring *cis*-fused to a cycloheptane ring (Fig. 1). The relative configurations at the asymmetric centres are *C*1(*R*), *C*4(*R*), *C*5(*S*), *C*8(*R*), *C*9(*R*), *C*11(*R*) and *C*15(*R*). The cyclopentane ring, atoms C1 through C4 and C15, is envelope-

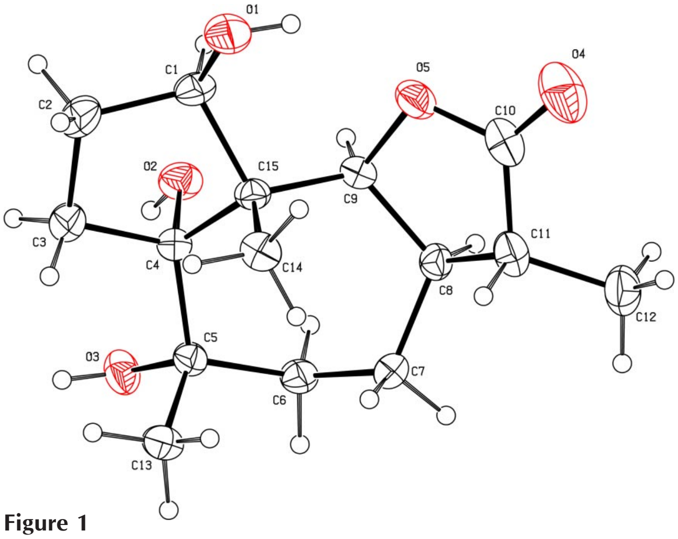


Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

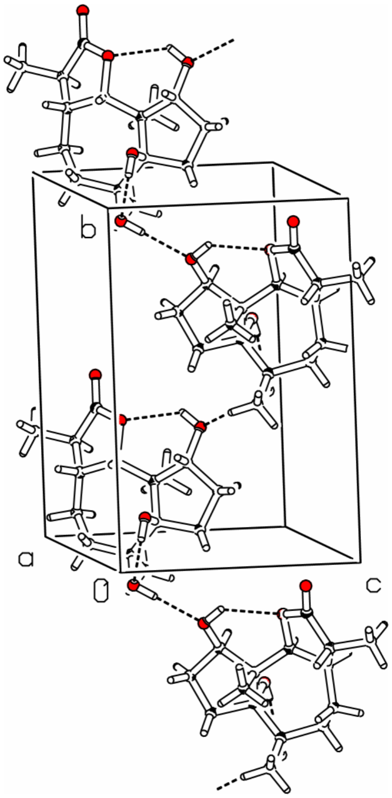


Figure 2
A partial packing diagram of (I), showing the O—H···O hydrogen-bonded (dashed lines) molecules forming helical chains.

shaped, with C15 at the flap. The average torsion angle magnitude is 0.41° and the ring asymmetry parameter (Duax & Norton, 1975) ΔC_s(C15) is 7.44°. The γ-lactone ring is also in an envelope conformation, with C8 at the flap. The ring asymmetry parameter ΔC_s(C8) is 5.5°. The average torsion angle magnitude in the lactone ring is 0.4°. The torsion angles of the cycloheptane ring have C_2 symmetrical values and the ring is therefore in a twist-chair conformation. The pseudo-diad axis bisects the C4—C5 bond and passes through C8. The asymmetry parameter ΔC_2 has a value of 12.3° and the average torsion angle magnitude is 5.1°.

Screw-related histerone molecules are joined by O—H···O intermolecular hydrogen bonds (Fig. 2) into helical chains. Hydroxy atom O1 participates in intra- (as a donor to atom O5) and intermolecular (as an acceptor from the O3 hydroxy group) interactions (Table 2). The structure is further stabilized by intra- and weak intermolecular C—H···O hydrogen bonding.

Experimental

The air-dried powdered plant material (2 kg) was extracted with CH_2Cl_2–MeOH (1:1) at room temperature for 120 h. The extract was concentrated under reduced pressure to afford a residue (30 g). This was subjected to column chromatography over silica gel (1.5 kg), using solvents of increasing polarity from *n*-hexane through EtOAc to obtain the title compound, (I). Colourless crystals were obtained from methanol by slow evaporation.

Crystal data

$C_{15}H_{24}O_5$
$M_r = 284.34$
Monoclinic, $P2_1$
$a = 7.0046$ (8) Å
$b = 13.1146$ (14) Å
$c = 8.4316$ (9) Å
$\beta = 109.753$ (2)°
$V = 728.97$ (14) Å^3
$Z = 2$
$D_x = 1.295$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3140 reflections
$\theta = 6.0$–56.0°
$\mu = 0.10$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.24 × 0.20 × 0.14 mm

Data collection

Bruker Smart APEX CCD area-detector diffractometer
ω scans
4433 measured reflections
1649 independent reflections
1615 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\rm max} = 27.6°$
$h = -8 \rightarrow 9$
$k = -16 \rightarrow 17$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.087$
$S = 1.16$
1649 reflections
187 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—C5	1.448 (2)	O2—C4	1.439 (2)
O5—C10	1.357 (3)	O1—C1	1.427 (3)
O5—C9	1.468 (2)		
C6—C5—C4—C15	54.7 (2)	C3—C4—C15—C1	45.09 (19)
C10—O5—C9—C8	12.1 (2)	C8—C7—C6—C5	87.2 (2)
C11—C8—C9—O5	−22.9 (2)	C4—C5—C6—C7	−68.5 (2)
C7—C8—C9—C15	−27.8 (3)	O5—C10—C11—C8	−19.2 (3)
C9—C8—C7—C6	−45.8 (3)	C9—C8—C11—C10	25.2 (2)
C9—O5—C10—C11	4.4 (3)	C15—C4—C3—C2	−32.7 (2)
C8—C9—C15—C4	76.1 (2)	C15—C1—C2—C3	22.7 (3)
C2—C1—C15—C4	−41.8 (2)	C4—C3—C2—C1	6.3 (3)
C5—C4—C15—C9	−70.8 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1$^{\rm i}$	0.82	2.01	2.818 (2)	167
O2—H2···O3	0.82	2.12	2.605 (2)	118
O1—H1···O5	0.82	2.19	2.815 (2)	133
C6—H6*A*···O2	0.97	2.56	2.940 (3)	104
C9—H9···O2	0.98	2.30	2.762 (2)	108
C11—H11···O2$^{\rm ii}$	0.98	2.51	3.265 (3)	134

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

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.96–0.98 Å, and with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl H atoms and $1.2U_{\rm eq}$(C) for other H atoms. The methyl and hydroxy groups were allowed to rotate but not to tip. The absolute configuration could not be determined because of the absence of a significant anomalous scattering effect; Friedel pairs were merged before the final cycles of the refinement.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

o122 Swamy *et al.* • $C_{15}H_{24}O_5$ *Acta Cryst.* (2005). E**61**, o121–o123

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

The authors thank Dr J.-S. Yadav, Director, IICT, Hyderabad, for his kind encouragement.

References

Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.

Duax, W. L. & Norton, D. A. (1975). In *Atlas of Steroid Structures*, Vol. I. New York: Plenum.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Kanchan, S. D. (1975). *Curr. Sci.* **44**, 358–361.

Kupchan, S. M., Eakin, M. A. & Thomas, A. M. (1971). *J. Med. Chem.* **14**, 1147–1152.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Patel, T. M. & Hedge, B. A. (1988). *Curr. Sci.* **57**, 1178–1181.

Ramesh, C., Ravindranath, N., Das, B., Prabhakar, A., Jagadeesh, B., Ravikumar, K., Kashinatham, A. & McMorris, T. C. (2003). *Phytochemistry*, , 841–844.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Acta Cryst. (2005). E**61**, o121–o123 Swamy *et al.* • $C_{15}H_{24}O_5$ **o123**