Acta Cryst. (1998). C**54**, 113–114

8α-Hydroxy-11α,13-dihydrozaluzanin C

Hiroko Kakuda,[a] Amr M. Helal,[b] Norio Nakamura[c] and Masao Hattori[c]

[a]*Laboratory of Chemistry, Toyama Medical and Pharmaceutical University, Sugitani 2630, Toyama 930-0194, Japan,* [b]*Department of Pharmacognosy, Faculty of Pharmacy, Cairo University, Kasr-El Aini Street, Cairo 11562, Egypt, and* [c]*Department of Cell-Resources Engineering, Research Institute for Wakan-Yaku, Toyama Medical and Pharmaceutical University, Sugitani 2630, Toyama 930-0194, Japan. E-mail: chemk@ms.toyama-mpu.ac.jp*

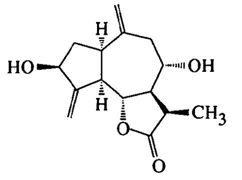
(*Received 4 September 1997; accepted 1 October 1997*)

Abstract

In the structure of the title compound [3a,4,5,6,6a,7,8,9,9a,9b-decahydro-4,8-dihydroxy-3-methyl-6,9-bis(methylene)azuleno[4,5-*b*]furan-2(3*H*)-one, $C_{15}H_{20}O_4$], the secondary hydroxyl groups participate in intermolecular hydrogen bonds both as donors and acceptors.

Comment

The title molecule, (I), was isolated previously from *Ainsliaea fragrans* Champ. as an oily substance (Bohlmann & Chen, 1982). Recently, we obtained the same compound from *Centaurea scoparia* Sieb. as colourless crystals (Helal *et al.*, 1997). The X-ray crystal and molecular structure determinations confirm the relative stereochemistry unambiguously.



(I)

The title compound has a cyclopentane ring *cis*-fused and a γ-lactone ring *trans*-fused to a cycloheptane ring (Fig. 1). The γ-lactone ring has a half-chair conformation with a pseudo-twofold axis running through the C12 carbonyl atom. The cyclopentane ring exhibits a distorted half-chair conformation with the C4 atom on the approximate twofold axis. The torsion angles of the cycloheptane ring have C_2 symmetrical values and the ring is therefore in a twist-chair conformation. The pseudo-twofold axis bisects the C7—C8 bond and passes through C1.

The secondary hydroxyl groups participate in intermolecular hydrogen bonds both as donors and acceptors. Atom O3 donates a hydrogen bond to O8 and also accepts a hydrogen bond from O8 (Table 2). Thus, the molecules are linked together by the latter hydrogen bond to form chains running around the 3_2 axes with each chain connected by the former hydrogen bond to form a network (Fig. 2).

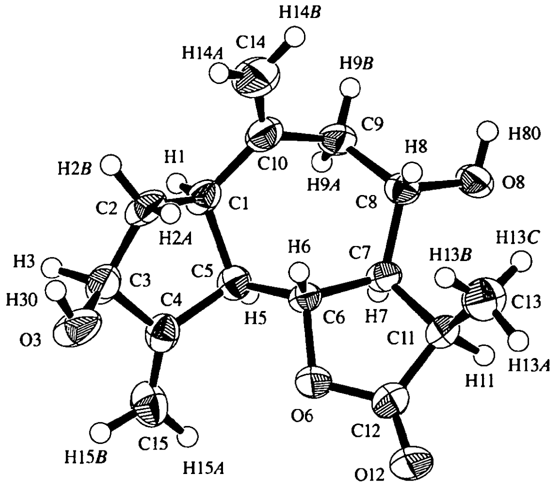


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

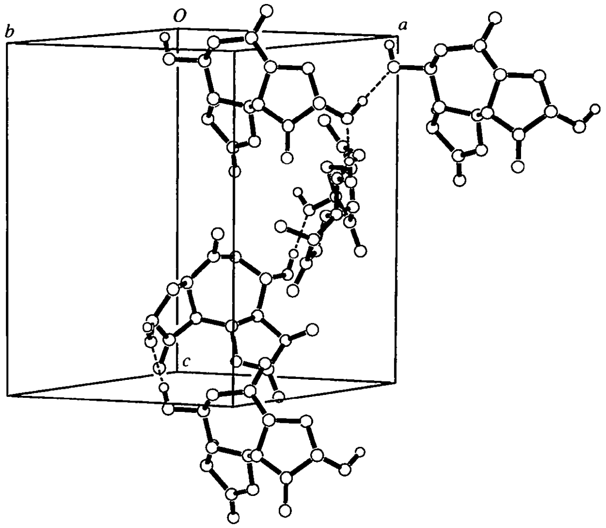


Fig. 2. The packing and hydrogen bonding of the molecule in the unit cell. All H atoms, except for those of the hydroxyl groups, have been omitted for clarity.

Experimental

The title compound was isolated as reported previously (Helal *et al.*, 1997). Crystals suitable for X-ray analysis were recrystallized from chloroform (m.p. 442 K).

Crystal data

$C_{15}H_{20}O_4$ Mo $K\alpha$ radiation
$M_r = 264.32$ $\lambda = 0.71069$ Å

Trigonal
$P3_2$
$a = 9.221\,(4)$ Å
$c = 14.006\,(6)$ Å
$V = 1031.4\,(9)$ Å^3
$Z = 3$
$D_x = 1.277$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 10.2–12.0°$
$\mu = 0.092$ mm^{-1}
$T = 293$ K
Prismatic
0.34 × 0.31 × 0.17 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: none
1780 measured reflections
1636 independent reflections
1150 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.029$
$\theta_{\rm max} = 27.48°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 18$
3 standard reflections every 150 reflections intensity decay: 0.68%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.121$
$S = 0.926$
1636 reflections
172 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.011$
$\Delta\rho_{\rm max} = 0.156$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.161$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.520 (6)	C2—C3	1.513 (7)
C1—C5	1.566 (5)	C9—C10	1.519 (6)
C1—C10	1.510 (7)		
C2—C1—C5	103.3 (3)	C5—C6—C7	114.4 (3)
C2—C1—C10	115.9 (4)	C6—C7—C8	114.5 (3)
C5—C1—C10	116.5 (3)	C6—C7—C11	103.1 (3)
O3—C3—C2	114.5 (4)	C8—C7—C11	117.3 (3)
O3—C3—C4	110.0 (3)	O8—C8—C7	105.4 (3)
C2—C3—C4	105.1 (3)	O8—C8—C9	109.9 (3)
C1—C5—C4	102.3 (3)	C7—C8—C9	112.5 (3)
C1—C5—C6	108.6 (3)	C7—C11—C12	102.2 (3)
C4—C5—C6	116.1 (3)	C7—C11—C13	117.1 (3)
O6—C6—C5	110.8 (3)	C12—C11—C13	109.4 (4)
O6—C6—C7	103.4 (3)		
O6—C6—C7—C11	−32.3 (4)	C5—C1—C10—C9	38.0 (5)
O6—C12—C11—C7	−12.4 (5)	C5—C6—C7—C8	78.6 (4)
C1—C2—C3—C4	28.8 (4)	C6—O6—C12—C11	−8.6 (5)
C1—C5—C4—C3	−17.3 (4)	C6—C5—C1—C10	39.6 (4)
C1—C5—C6—C7	−91.0 (4)	C6—C7—C8—C9	−54.7 (4)
C1—C10—C9—C8	−89.4 (5)	C6—C7—C11—C12	26.9 (4)
C2—C1—C5—C4	34.6 (4)	C7—C6—O6—C12	25.9 (4)
C2—C3—C4—C5	−6.6 (4)	C7—C8—C9—C10	71.6 (4)
C3—C2—C1—C5	−39.7 (4)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H30···O8$^{\rm i}$	0.917	1.873	2.739 (5)	156.5
O8—H80···O3$^{\rm ii}$	0.880	1.912	2.790 (5)	175.3

Symmetry codes: (i) $1 + x, y, z$; (ii) $1 - y, x - y, z - \frac{1}{3}$.

All H atoms were obtained from difference Fourier synthesis but not refined. Isotropic displacement parameters of the H atoms were set equal to $1.05U_{\rm eq}$ of the bonded non-H atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995*b*). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *CHARON* (Lauher, 1989). Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1327). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.

Bohlmann, F. & Chen, Z.-L. (1982). *Phytochemistry*, **21**, 2120–2122.

Helal, A. M., Nakamura, N., Meselhy, M. R., El-Fishawy, A. M., Hattori, M. & Mahran, G. H. (1997). *Phytochemistry*, **45**, 551–554.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lauher, J. W. (1989). *CHARON. A Graphics Program for Postscript Printers.* The Research Foundation of the State University of New York, USA.

Molecular Structure Corporation (1995*a*). *MSC/AFC Diffractometer Control Software.* Version 5.3.2c. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1995*b*). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1998). **C54**, 114–116

Diethyl 3,7-Hypoxanthyldiacetate

GEETA SOOD, CARL H. SCHWALBE AND WILLIAM FRASER

Pharmaceutical Sciences Institute, Aston University, Aston Triangle, Birmingham B4 7ET, England. E-mail: c.h.schwalbe@aston.ac.uk

(*Received 4 June 1997; accepted 8 September 1997*)

Abstract

The title compound, diethyl 6-oxo-6,7-dihydro-3*H*-purine-3,7-diacetate, $C_{13}H_{16}N_4O_5$, was obtained by direct alkylation of hypoxanthine using ethyl bromoacetate, in the presence of potassium carbonate; the two alkyl substituents are attached to the heterocycle at positions N3 and N7. Both side chains avoid steric hindrance with the heterocycle, as demonstrated by the torsion angles C2—N3—C15—C16 −68.9 (2) and C8—N7—C10—C11 106.8 (2)°.