Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\text{eq}} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.7028 (3)	0.1345	0.2978 (2)	0.0505 (1)
C2	0.8586 (4)	0.0572 (4)	0.3971 (3)	0.0473 (1)
N3	0.7758 (3)	−0.0860 (3)	0.4239 (2)	0.0540 (1)
C4	0.5611 (4)	−0.1265 (4)	0.3273 (2)	0.0457 (1)
C5	0.4877 (4)	0.0418 (4)	0.2706 (2)	0.0448 (1)
C6	0.3890 (4)	−0.2107 (5)	0.3927 (3)	0.0660 (1)
C7	0.3696 (4)	0.0488 (4)	0.1208 (2)	0.0461 (1)
C8	0.4690 (5)	−0.0169 (4)	0.0202 (3)	0.0613 (1)
C9	0.3508 (6)	−0.0141 (5)	−0.1163 (3)	0.0744 (1)
C10	0.1370 (7)	0.0537 (6)	−0.1525 (3)	0.0804 (1)
C11	0.0393 (5)	0.1204 (6)	−0.0538 (4)	0.0858 (1)
C12	0.1537 (4)	0.1188 (5)	0.0825 (3)	0.0645 (1)
O13	1.0419 (3)	0.1189 (3)	0.4493 (2)	0.0645 (1)

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.355 (3)	C5—C7	1.502 (3)
O1—C5	1.463 (3)	C7—C8	1.381 (4)
C2—N3	1.328 (4)	C7—C12	1.383 (4)
C2—O13	1.211 (3)	C8—C9	1.385 (4)
N3—C4	1.457 (3)	C9—C10	1.362 (5)
C4—C5	1.524 (4)	C10—C11	1.363 (5)
C4—C6	1.499 (4)	C11—C12	1.376 (4)
C2—O1—C5	108.6 (2)	C4—C5—C7	115.8 (2)
O1—C2—N3	109.9 (2)	C5—C7—C8	121.6 (2)
O1—C2—O13	121.4 (2)	C5—C7—C12	119.4 (2)
N3—C2—O13	128.7 (3)	C8—C7—C12	119.0 (2)
C2—N3—C4	112.4 (2)	C7—C8—C9	119.9 (3)
N3—C4—C5	100.0 (2)	C8—C9—C10	120.5 (3)
N3—C4—C6	113.8 (2)	C9—C10—C11	119.8 (3)
C5—C4—C6	114.5 (2)	C10—C11—C12	120.6 (3)
O1—C5—C4	103.9 (2)	C7—C12—C11	120.1 (3)
O1—C5—C7	111.2 (2)		

The scan rate was 3° min^{-1} (in ω). Weak reflections [$I < 10\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to improve accuracy. H atoms were placed at assumed positions (C,N—H = 0.95 Å, $U = 1.2 \times U_{\text{eq}}$ of the associated atom) and fixed. The correct enantiomer was chosen on the basis of the known configuration of the starting material [(−)-(1*R*,2*S*)-norephedrine].

Financial support from the National Cancer Institute (CA 51794) is acknowledged.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55982 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1047]

References

Adams, W. J., Geise, H. J. & Bartell, L. S. (1970). *J. Am. Chem. Soc.* **92**, 5013-5019.

Fodor, G., Stefanovsky, J. & Kurtev, B. (1967). *Monatsh. Chem.* **98**, 1027-1042.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Kano, S., Yokomatsu, T., Iwasawa, H. & Shibuya, S. (1987). *Tetrahedron Lett.* **28**, 6331-6334.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1985). *SHELXS*86. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175-189. Oxford Univ. Press.

Spassov, S. L., Stefanovsky, J. N., Kurtev, B. J. & Fodor, G. (1972). *Chem. Ber.* **105**, 2462-2466.

Acta Cryst. (1993). C**49**, 1240–1242

Structure of Vicogenin

J. Sivaraman and K. Subramanian

Department of Physics, Anna University, Madras-25, India

D. Velmurugan and E. Subramanian

Department of Crystallography and Biophysics, University of Madras, Madras-25, India

K. Balakrishna

Captain Srinivasamurthy Drug Research Institute for Ayurveda, Madras-106, India

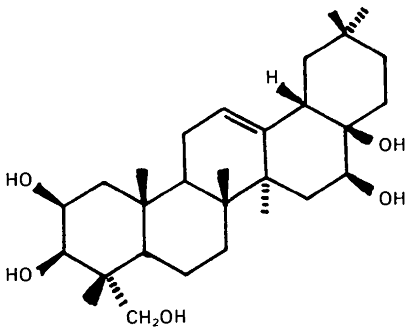
(*Received* 2 *April* 1992; *accepted* 23 *December* 1992)

Abstract

Vicogenin is a new 28-nortriterpenoid. Its structure has been elucidated as 2α,3β,16α,17β,23-pentahydroxy-28-norolean-12-ene. In the crystal there are two water molecules per vicogenin molecule. Hydrogen bonds link the vicogenin molecules to each other and to the water molecules.

Comment

The title compound (I) has been isolated for the first time from the chloroform extract of the plant *Vicoa indica* DC(composital). The plant is said to exhibit antifertility activity when administered during the post-partum period in albino rats (Gandhi,



(I)

Sankaranarayanan, Ramesh Lal & Mathur, 1983) and anti-implantation activity (Mohana, Susan & Purushothaman, 1984). The configuration at the *D*/*E* ring junction was tentatively fixed as *cis* on the basis of biogenetic considerations and ^{13}C NMR spectra and the molecular formula was established by high-resolution mass spectrometry. In order to confirm the structure, particularly the nature of the configuration at the *D*/*E* ring junction, an X-ray crystallographic investigation was undertaken. 28-Noroleananes are relatively rare in nature. There is only one report of the extraction of 17-hydroxy-28-norolean-12-ene, a natural product (Linde, 1979).

The C—C bond lengths range from 1.507 (11) to 1.600 (11) Å with a mean value of 1.54 (2) Å. The endocyclic bond angles for tetrahedral atoms in rings *A*, *B*, *C*, *D* and *E* vary from 104.9 (6) to 125.4 (7)° with a mean value of 111.81 (7)°, which compares well with that of related compounds (Hoge & Nordman, 1974; Subramanian, Selladurai, Sivakumar, Ponnuswamy & Sukumar, 1989). The rings *A*, *B*, *D* and *E* possess chair conformations and the conformation of ring *C* is intermediate between half-chair and sofa (Gzella, Zaprutko, Wrzeciono & Jaskólski, 1987) with the best rotational axis bisecting the C(8)—C(9) and C(13)—C(12) bonds $\{\Delta C_2[C(8)—C(9)] = 16.5°\}$. The ring junctions *A*/*B*, *B*/*C* and *C*/*D* are *trans*, but the *D*/*E* ring junction is *cis* with both the hydroxyl group and the H atom pointing upwards, thus showing a *cis* di-β-configuration. The angles between the mean planes of the rings are *A*/*B* 15.20, *B*/*C* 165.49, *C*/*D* 165.11 and *D*/*E* 121.46° (Maurin & Krygowski, 1987), making a 'zigzag' along the length of the molecule. The molecules are held together by a network of hydrogen bonds (Table 3) involving the hydroxyl groups and the water molecules.

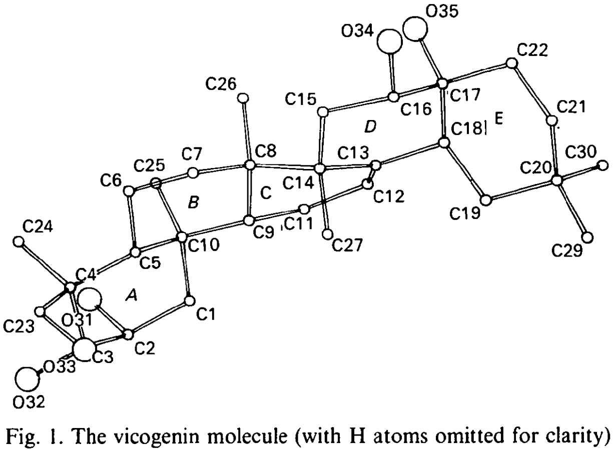


Fig. 1. The vicogenin molecule (with H atoms omitted for clarity) showing the atomic numbering scheme and ring identifiers.

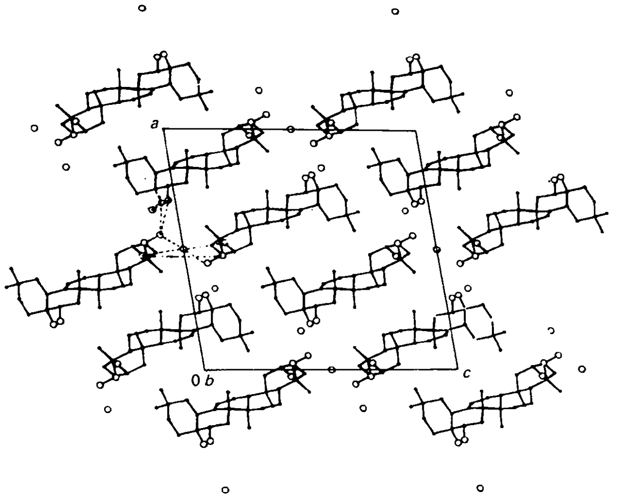


Fig. 2. Molecular packing viewed along the *b* axis.

Experimental

Crystal data

$C_{29}H_{48}O_5.2H_2O$
$M_r = 512$
Monoclinic
$I2$
$a = 19.14\ (3)$ Å
$b = 7.635\ (3)$ Å
$c = 19.838\ (1)$ Å
$\beta = 99.84\ (4)°$
$V = 2856.36$ Å^3
$Z = 4$
$D_x = 1.192$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 20 reflections
$\theta = 20$-$30°$
$\mu = 0.634$ mm^{-1}
$T = 293$ K
Needle
0.5 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.960$, $T_{\max} = 1.021$
2220 measured reflections
2220 independent reflections
2025 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.03$
$\theta_{\max} = 60°$
$h = -21 \rightarrow 21$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 22$
2 standard reflections monitored every 100 reflections
intensity variation: <2%

Refinement

Refinement on F by full-matrix least squares	$w = 7.4521/[\sigma^2(F) + 0.000092F^2]$
Final $R = 0.056$	$(\Delta/\sigma)_{\max} = -0.156$
$wR = 0.060$	$\Delta\rho_{\max} = 0.4765$ e Å^{-3}
$S = 2.3$	$\Delta\rho_{\min} = -0.398$ e Å^{-3}
2025 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
526 parameters	
All H-atom parameters refined; positions found from difference Fourier syntheses	

Cell refinement: *SDP* (Frenz, 1978). Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
C(1)	0.5236 (4)	−0.1923 (10)	0.2686 (3)	0.39 (3)
C(2)	0.4881 (4)	−0.2416 (11)	0.1971 (4)	0.43 (2)
C(3)	0.4760 (4)	−0.0811 (11)	0.1509 (4)	4.15 (2)
C(4)	0.5417 (4)	0.0321 (11)	0.1481 (3)	3.65 (2)

Ring *B*

C(8)—C(9)—C(10)—C(5)	−54.9 (7)	C(5)—C(6)—C(7)—C(8)	53.7 (9)
C(7)—C(8)—C(9)—C(10)	45.1 (8)	C(10)—C(5)—C(6)—C(7)	−64.3 (8)
C(6)—C(7)—C(8)—C(9)	−42.4 (9)		

Ring *C*

C(8)—C(9)—C(11)—C(12)	38.9 (8)	C(12)—C(13)—C(14)—C(8)	−25.0 (10)
C(9)—C(11)—C(12)—C(13)	−7.6 (12)	C(9)—C(8)—C(14)—C(13)	54.7 (7)
C(11)—C(12)—C(13)—C(14)	0.8 (13)		

Ring *D*

C(14)—C(13)—C(18)—C(17)	−43.4 (9)	C(14)—C(15)—C(16)—C(17)	54.3 (9)
C(16)—C(17)—C(18)—C(13)	52.7 (9)	C(13)—C(14)—C(15)—C(16)	−42.0 (9)
C(15)—C(16)—C(17)—C(18)	−58.1 (8)		

Ring *E*

C(17)—C(18)—C(19)—C(20)	−54.8 (9)	C(20)—C(21)—C(22)—C(17)	54.3 (11)
C(18)—C(19)—C(20)—C(21)	55.3 (9)	C(18)—C(17)—C(22)—C(21)	−51.4 (10)
C(19)—C(20)—C(21)—C(22)	−54.4 (10)		

Table 3. *Hydrogen-bond distances* (Å)

D—H···*A*	*D*···*A*	Symmetry location of acceptor
O(31)—H(31)···O(33)*	2.688 (8)	$x, y-1, z$
O(31)—H(31)···O(36)*	3.246 (6)	$x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$
O(32)—H(32)···O(34)	3.142 (6)	$x-\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$
O(34)—H(34)···O(32)	3.142 (6)	$x+\frac{1}{2}, y+\frac{1}{2}, z+\frac{1}{2}$
O(35)—H(35)···O(32)	2.834 (5)	$x+\frac{1}{2}, y+\frac{1}{2}, z+\frac{1}{2}$
O(36)—H2(36)···O(32)	3.021 (3)	$x-\frac{1}{2}, y+\frac{1}{2}, z+\frac{1}{2}$
O(37)—H1(37)···O(34)	2.678 (13)	$x-\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$
O(37)—H2(37)···O(35)	2.714 (13)	$x-\frac{1}{2}, y+\frac{1}{2}, z-\frac{1}{2}$

* Angle O(33)···H(31)···O(36) = 80°; sum of angles at H(31) = 354°.