

bicyclic aza sugar, 8-epicastanospermine, without loss of stereochemical integrity. In the solid state, molecules dimerize in a 'head-to-tail' manner through O—H...N hydrogen bonds (O5—N1 2.74 Å), as shown in the section of the unit cell depicted in Fig. 2.

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## Structure of (3β,7α)-3,7-Dihydroxykaur-16-en-18-yl Acetate (Linearol)

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**Abstract.** C<sub>22</sub>H<sub>34</sub>O<sub>4</sub>, *M<sub>r</sub>* = 362.51, monoclinic, *P*2<sub>1</sub>, *a* = 7.785 (9), *b* = 11.688 (6), *c* = 11.792 (7) Å, β = 107.18 (6)°, *V* = 1025 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.145 (4) (by flotation), *D<sub>x</sub>* = 1.174 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.74 cm<sup>-1</sup>, *F*(000) = 396, *T* = 296 K, *R* = 0.042 for 1240 observed independent reflections. The skeleton of the molecule consists of contacting bicyclo[3.2.1]octane and bicyclo[4.4.0]decane (*trans*-decalin) systems. All of the cyclohexanes have an approximate chair conformation while the cyclopentane presents an envelope conformation. The CH<sub>3</sub> groups occupy axial positions. The COCOCH<sub>3</sub> group and its neighbouring OH group are in equatorial positions. The other OH group is in an axial position.

**Introduction.** *Sideritis* species (Lamiaceae) are used in traditional medicine for their anti-inflammatory (Villar, Jimenez & Alcaraz, 1986; Yeşilada & Ezer, 1989), antimicrobial and cytostatic activity (Diaz, Garcia-Granados, Moreno, Parra, Quevedo-Sarmiento & Saenz de Buruaga, 1987; Darias, Bravo, Rabanal, Sanchez-Mateo & Martin-Herrera, 1990). *Sideritis congesta* Davis et Huber-Morath, which is endemic to Turkey, is the most widely used species of the genus in Turkish folk medicine as herbal tea (Sezik & Ezer, 1983). From the aerial parts of this plant, we isolated flavonoids and a major diterpenoid, linearol, which was determined on the basis of spectroscopic data (IR, MS, NMR) (Ezer, 1980).

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In this study, the crystal and molecular structure of linearol has been determined by X-ray diffraction.

**Experimental.** A prismatic colourless crystal, 0.20 × 0.25 × 0.30 mm, was used for data collection on a Rigaku AFC-5 diffractometer with graphite-monochromated Mo *K*α radiation. Lattice parameters were determined from a least-squares fit of 25 centred reflections in the 2θ range 20.2–32.2°. Intensity data were measured by ω–2θ-scan technique with a scan rate of 4° min<sup>-1</sup> in 2θ, and for 6.0 ≤ 2θ ≤ 50.0° and *hkl* ranges 0, 0, –14 to 9, 13, 12. Of a total of 2053 reflections measured, 1907 were independent (*R<sub>int</sub>* = 0.024), of which 1240 reflections with *I* > 2.00σ(*I*) were used for structure solution and refinement. Three standard reflections (131̄, 113̄, 032) monitored at intervals of 150 showed no significant variation. No absorption correction, but Lorentz, polarization and secondary-extinction (coefficient = 0.31645 × 10<sup>-5</sup>) corrections were applied. The structure was solved by direct methods (Gilmore, 1984; Beurskens, 1984), and refinement, based on |*F*| values, was carried out by full-matrix least squares, with non-H atoms refined anisotropically. 337 parameters were refined. Final *R* = 0.042, *wR* = 0.042 with the weighting scheme *w* = 4*F<sub>o</sub>*<sup>2</sup>/σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>); *S* = 1.40. Maximum final shift to e.s.d. ratio was 0.09, and maximum and minimum peak heights in the final difference Fourier map were 0.13 and –0.13 e Å<sup>-3</sup>. All calculations were performed with

TEXSAN (Molecular Structure Corporation, 1985) and PLUTO (Motherwell & Clegg, 1978). Neutral-atom scattering factors and  $f'$  and  $f''$  values for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

**Discussion.** Fig. 1 shows the molecular structure with the numbering scheme and Fig. 2 presents a unit-cell packing diagram. Atomic coordinates with the equivalent isotropic temperature factors and intramolecular bond lengths are given in Tables 1\* and 2, respectively. The skeleton of the molecule consists of a bicyclo[3.2.1]octane system and a

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond angles, torsion angles and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55359 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

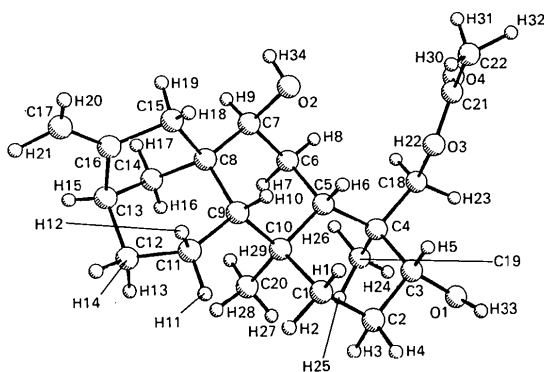


Fig. 1. Molecular structure with the atom-numbering scheme.

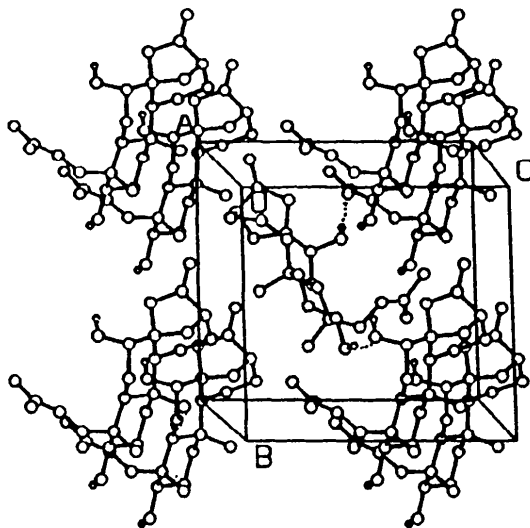


Fig. 2. Unit-cell packing diagram.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2$ ) of non-H atoms

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	y	z	$B_{eq}$
O(1)	0.5088 (5)	0.7234 (5)	0.4489 (3)	5.5 (2)
O(2)	0.1459 (5)	0.2359 (5)	0.3891 (3)	4.6 (2)
O(3)	0.2677 (5)	0.5045 (5)	0.5519 (3)	4.7 (2)
O(4)	0.0254 (8)	0.5236 (6)	0.6078 (5)	10.4 (3)
C(1)	0.5906 (7)	0.4534 (6)	0.2976 (6)	4.7 (3)
C(2)	0.6074 (9)	0.5779 (7)	0.3344 (6)	5.1 (3)
C(3)	0.4955 (7)	0.6048 (6)	0.4165 (5)	4.1 (2)
C(4)	0.2967 (6)	0.5750 (6)	0.3632 (4)	3.5 (2)
C(5)	0.2820 (6)	0.4491 (5)	0.3196 (4)	3.1 (2)
C(6)	0.0909 (7)	0.4045 (6)	0.2704 (5)	4.2 (3)
C(7)	0.0885 (7)	0.2743 (6)	0.2673 (5)	4.1 (3)
C(8)	0.2042 (6)	0.2245 (6)	0.1949 (4)	3.5 (2)
C(9)	0.3927 (6)	0.2817 (5)	0.2283 (4)	3.3 (2)
C(10)	0.3964 (6)	0.4147 (5)	0.2349 (4)	3.4 (2)
C(11)	0.5059 (7)	0.2287 (7)	0.1532 (5)	4.4 (3)
C(12)	0.408 (1)	0.2008 (7)	0.0243 (5)	5.5 (3)
C(13)	0.2253 (9)	0.1431 (6)	0.0142 (5)	5.0 (3)
C(14)	0.1110 (8)	0.2271 (7)	0.0599 (5)	4.8 (3)
C(15)	0.2279 (8)	0.0947 (6)	0.2119 (6)	4.5 (3)
C(16)	0.2458 (7)	0.0467 (6)	0.0994 (6)	4.9 (3)
C(17)	0.275 (1)	-0.0646 (7)	0.0756 (8)	6.5 (4)
C(18)	0.2019 (8)	0.5895 (6)	0.4596 (5)	4.3 (2)
C(19)	0.201 (1)	0.6641 (7)	0.2678 (6)	5.3 (3)
C(20)	0.333 (1)	0.4725 (7)	0.1140 (6)	5.3 (3)
C(21)	0.1663 (9)	0.4797 (6)	0.6198 (5)	5.2 (3)
C(22)	0.248 (1)	0.3902 (8)	0.7083 (8)	7.9 (5)

Table 2. Bond lengths ( $\text{\AA}$ ) between non-H atoms

O(1)—C(3)	1.434 (7)	C(7)—C(8)	1.528 (7)
O(2)—C(7)	1.444 (7)	C(8)—C(9)	1.554 (7)
O(3)—C(18)	1.450 (6)	C(8)—C(14)	1.542 (7)
O(3)—C(21)	1.312 (6)	C(8)—C(15)	1.534 (8)
O(4)—C(21)	1.181 (7)	C(9)—C(10)	1.557 (7)
C(1)—C(2)	1.513 (8)	C(9)—C(11)	1.551 (7)
C(1)—C(10)	1.542 (7)	C(10)—C(20)	1.522 (8)
C(2)—C(3)	1.515 (8)	C(11)—C(12)	1.521 (8)
C(3)—C(4)	1.528 (7)	C(12)—C(13)	1.547 (9)
C(4)—C(5)	1.553 (7)	C(13)—C(14)	1.527 (9)
C(4)—C(18)	1.536 (7)	C(13)—C(16)	1.487 (9)
C(4)—C(19)	1.553 (8)	C(15)—C(16)	1.484 (8)
C(5)—C(6)	1.521 (7)	C(16)—C(17)	1.363 (9)
C(5)—C(10)	1.574 (7)	C(21)—C(22)	1.48 (1)
C(6)—C(7)	1.522 (8)		

bicyclo[4.4.0]decane (*trans*-decalin) system which are in contact through C(9) and C(8) atoms. It can be seen from the torsion angles that the cyclohexane fragment and the cyclopentane fragment of the bicyclooctane have a distorted chair conformation and an envelope conformation with C(14) as a 'flap' atom, respectively. From the *trans*-fused cyclohexanes of bicyclodecane, the cyclohexane which includes both of the  $\text{CH}_3$  substituents has a chair conformation, whereas the other cyclohexane has a slightly deformed chair conformation. The contacting cyclohexanes of the bicyclic systems are joined together as equatorial and axial substituents. The torsion angles also show that the OH group at the C(3) atom and the  $\text{COCOCH}_3$  group are in equatorial positions, while both of the  $\text{CH}_3$  groups are bonded to the same side of the average plane of their common ring as axial substituents. The  $\text{CH}_2$  group is

almost in the plane formed by C(8)—C(15)—C(16)—C(13). There are two intermolecular hydrogen bonds between the neighbouring molecules. Bond distances O(1<sup>i</sup>)...O(2<sup>ii</sup>), O(2<sup>i</sup>)...O(4<sup>iii</sup>) and bond angles O(1<sup>i</sup>)—H(33<sup>i</sup>)...O(2<sup>ii</sup>), O(2<sup>i</sup>)—H(34<sup>i</sup>)...O(4<sup>iii</sup>) are found as 2.804 (6), and 2.823 (7) Å, and 178 (7) and 168 (6)°, respectively. The symmetry codes are: (i)  $x, y, z$ ; (ii)  $-x + 1, y + \frac{1}{2}, -z + 1$ ; (iii)  $-x, y - \frac{1}{2}, -z + 1$ .

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## Structure of Oxmetidine Dihydrochloride (SKF 92994)

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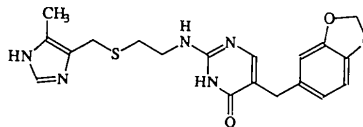
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**Abstract.** 5-(1,3-Benzodioxol-5-ylmethyl)-2-[2-(5-methyl-1*H*-imidazol-4-ylmethylthio)ethylamino]-4(1*H*)-pyrimidone dihydrochloride, C<sub>19</sub>H<sub>23</sub>N<sub>5</sub>O<sub>3</sub>·S<sup>2+</sup>·2Cl<sup>-</sup>,  $M_r = 472.4$ , triclinic,  $P\bar{1}$ ,  $a = 8.291$  (2),  $b = 10.759$  (3),  $c = 13.169$  (3) Å,  $\alpha = 108.42$  (2),  $\beta = 93.28$  (2),  $\gamma = 100.3$  (3)°,  $V = 1088.6$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.44$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.42$  mm<sup>-1</sup>,  $F(000) = 492$ ,  $T = 294$  K, final  $R = 0.038$  for 3184 unique observed reflexions. All bonded distances and interbond angles lie within the range of values for compounds of a similar nature. The planar imidazole and isocytosine groups are connected by a four-atom folded methylthioethyl chain with *gauche* linkages at the S atom. Intermolecular bonding includes N—H...Cl hydrogen bonds and van der Waals forces.

**Introduction.** Oxmetidine (1) was identified as a highly effective histamine H<sub>2</sub> antagonist inhibiting gastric acidity in man and effective in the treatment of duodenal ulcers. The compound was withdrawn in the later stages of clinical development owing to a

low incidence of side effects (Brown, Blakemore, Durant, Emmett, Ganellin, Parsons, Rawlings & Walker, 1988).



(1)

**Experimental.** The compound was prepared at Smith Kline and French Research Ltd, and was characterized by elemental analysis, proton NMR, and infrared and mass spectrometry (Brown *et al.*, 1988). The crystals were prepared in Oxford by several recrystallizations from a mixture of *n*-propanol and water. After preliminary X-ray precession and Weissenberg photography the X-ray data were collected with an Enraf-Nonius CAD-4 diffractometer from a crystal of size 0.5 × 0.5 × 0.3 mm, following the manufacturer's recommended procedures to optimize the data collection parameters. Lattice parameters were obtained by the least-squares best fit of 25

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