

surprising that atoms C(3), C(4) and C(8) to C(10) have relatively high thermal parameters. Indeed, the further an atom is from N or C(1) the larger is its thermal parameter.  $U_{eq}$  for N or C(1) is 0.031 Å<sup>2</sup>, while the average of the  $U_{eq}$ 's of C(2), C(6) and C(7) increases to 0.043 Å<sup>2</sup> and reaches 0.068 Å<sup>2</sup> for C(3), C(4), C(5), C(8), C(9) and C(10). The H-atom  $U_{iso}$ 's follow the same trend (except for those on N which are H-bonded). This observation is also consistent with a slight oscillation or disorder about the C<sub>3</sub> axis of the molecule. These results are in agreement with those of the vibrational spectroscopic and spin-relaxation time studies, in that the hydrogen bonding of the NH<sub>3</sub> group holds this part of the molecule in place and the hydrocarbon portion undergoes hindered rotation about the molecular threefold axis (Harvey *et al.*, 1986*b*), *i.e.* the symmetry is maintained and the high-temperature phase is ordered. Since the entropy of transition is quite small, it seems probable that only a minor change in crystal structure occurs at the transition, perhaps a doubling of one of the unit-cell dimensions.

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### Triterpenoids. III. Structure of 3β-Acetoxy-11-oxo-18α-olean-12-en-28-oic Acid Methyl Ester

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**Abstract.** C<sub>33</sub>H<sub>50</sub>O<sub>5</sub>,  $M_r = 526.40$ , monoclinic,  $P2_1$ ,  $a = 7.748$  (1),  $b = 16.370$  (2),  $c = 12.099$  (2) Å,  $\beta = 102.50$  (1)°,  $V = 1498.1$  (4) Å<sup>3</sup>,  $D_m = 1.17$  (1),  $D_x = 1.17$  Mg m<sup>-3</sup>,  $Z = 2$ ,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,

$\mu(\text{Cu } K\alpha) = 0.529$  mm<sup>-1</sup>,  $F(000) = 576$ , room temperature, final  $R = 0.054$  for 1893 observed reflections. The molecule consists of five *trans* fused six-membered rings. Ring C has a sofa conformation distorted towards a half chair. The H atom at the C(18) asymmetric centre takes an axial position relative to the D and E rings.

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**Introduction.** In a previous paper (Gzella, Zaprutko, Wrzeciono & Gdaniec, 1986) we reported the structure of the 18-diastereomeric derivative of the present compound. This paper reports the first X-ray structure of a triterpenoid with the 18 $\alpha$ -olean-12-ene skeleton. This structure investigation has been undertaken to facilitate interpretation of <sup>1</sup>H NMR, <sup>13</sup>C NMR and MS data of 11-oxo-18 $\alpha$ -oleanolic acid derivatives.

**Experimental.** The title compound was prepared by a modified version of the procedure of Corey & Ursprung (1956). The modifications consisted of not performing the reaction in a nitrogen atmosphere and purifying the product by column chromatography. M.p. 586–590 K ( $\alpha$ )<sub>D</sub><sup>22°C</sup> = +68° (CHCl<sub>3</sub>); [m.p. 579–580 K (Corey & Ursprung, 1956), m.p. 581–582 K, ( $\alpha$ )<sub>D</sub><sup>22°C</sup> = +84° (Barton & Holnes, 1952)]; needle crystals recrystallized from methanol,  $D_m$  by flotation, crystal 0.4 × 0.3 × 0.1 mm, Syntex P2<sub>1</sub> diffractometer, graphite-monochromated Cu K $\alpha$  radiation, cell parameters from least-squares treatment of setting angles of 15 reflections,  $\theta$ -2 $\theta$  scan technique, intensity variation for two standard reflections monitored every 100 reflections less than 2%, profiles measured for 2189 reflections with  $2\theta \leq 115^\circ$ , 2097 unique reflections,  $R_{\text{int}} = 0.005$ , range of  $hkl$ :  $h$  -8→8,  $k$  0→17,  $l$  0→13, profile analysis according to Lehmann & Larsen (1974), no absorption correction, 1893 observed reflections with  $I \geq 1.96\sigma(I)$ , structure solved by direct methods using MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refinement on  $F$  by full-matrix least-squares method, H-atoms generated from assumed geometries and included as fixed isotropic contributions to  $F_c$ ,  $w = \sigma^{-2}(F)$ , empirical isotropic extinction parameter  $x$  used to correct  $F_c$  according to  $F_c' = F_c(1 - xF_c/\sin\theta)$ ,  $x$  converged at 0.022 (1), final  $R = 0.054$  and  $wR = 0.057$  for 343 refined parameters,  $(\Delta/\sigma)_{\text{max}} = 0.002$ , max. and min. height in final  $\Delta F$  map 0.16 and -0.15 e Å<sup>-3</sup>; computer programs: MULTAN80 (Main *et al.*, 1980), SHELX76 (Sheldrick, 1976) and local programs (Jaskólski, 1982); molecular illustrations drawn using PLUTO (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** Table 1 lists the final atomic coordinates and isotropic temperature factors. The bond lengths and angles are listed in Table 2. The numbering scheme and the endocyclic torsion angles are given in Fig. 1.\* A stereoview of the molecule is given in Fig. 2.

\* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43530 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O(1)	1.2374 (5)	0.6096	0.8268 (3)	0.065 (2)
O(2)	1.4028 (6)	0.6040 (4)	0.6949 (4)	0.091 (2)
O(3)	1.2009 (5)	0.2067 (3)	0.9021 (4)	0.064 (2)
O(4)	0.6606 (6)	0.0190 (4)	0.8484 (3)	0.071 (2)
O(5)	0.3934 (5)	-0.0230 (4)	0.7628 (3)	0.068 (2)
C(1)	1.2218 (7)	0.3792 (4)	0.8353 (4)	0.048 (2)
C(2)	1.2875 (7)	0.4667 (4)	0.8664 (5)	0.051 (2)
C(3)	1.1685 (7)	0.5277 (4)	0.7951 (5)	0.053 (2)
C(4)	0.9759 (7)	0.5251 (4)	0.8081 (5)	0.051 (2)
C(5)	0.9133 (7)	0.4351 (4)	0.7852 (5)	0.046 (2)
C(6)	0.7194 (7)	0.4214 (4)	0.7896 (5)	0.054 (2)
C(7)	0.6519 (7)	0.3431 (4)	0.7242 (5)	0.052 (2)
C(8)	0.7582 (7)	0.2660 (4)	0.7659 (4)	0.045 (2)
C(9)	0.9623 (7)	0.2848 (4)	0.7881 (4)	0.042 (2)
C(10)	1.0308 (7)	0.3658 (4)	0.8502 (4)	0.042 (2)
C(11)	1.0626 (7)	0.2076 (4)	0.8304 (4)	0.047 (2)
C(12)	0.9895 (7)	0.1309 (4)	0.7765 (5)	0.053 (2)
C(13)	0.8328 (7)	0.1226 (4)	0.7058 (4)	0.046 (2)
C(14)	0.7136 (7)	0.1960 (4)	0.6724 (4)	0.045 (2)
C(15)	0.5150 (8)	0.1725 (5)	0.6560 (5)	0.055 (2)
C(16)	0.4657 (8)	0.0927 (5)	0.5903 (5)	0.057 (2)
C(17)	0.5817 (7)	0.0210 (4)	0.6437 (4)	0.049 (2)
C(18)	0.7744 (8)	0.0428 (4)	0.6460 (5)	0.050 (2)
C(19)	0.9020 (8)	-0.0284 (4)	0.6828 (5)	0.057 (2)
C(20)	0.8477 (9)	-0.1085 (5)	0.6168 (5)	0.060 (2)
C(21)	0.6566 (9)	-0.1279 (5)	0.6156 (5)	0.067 (3)
C(22)	0.5311 (9)	-0.0566 (5)	0.5709 (6)	0.070 (3)
C(23)	0.8678 (8)	0.5805 (5)	0.7136 (6)	0.067 (3)
C(24)	0.9540 (8)	0.5592 (5)	0.9231 (6)	0.068 (3)
C(25)	1.0356 (7)	0.3627 (4)	0.9789 (5)	0.055 (2)
C(26)	0.7098 (8)	0.2369 (5)	0.8770 (5)	0.058 (2)
C(27)	0.7450 (8)	0.2241 (5)	0.5549 (4)	0.056 (2)
C(28)	0.5548 (8)	0.0061 (4)	0.7620 (5)	0.055 (2)
C(29)	0.878 (1)	-0.1025 (5)	0.4940 (6)	0.082 (3)
C(30)	0.966 (1)	-0.1782 (5)	0.6755 (6)	0.077 (3)
C(31)	1.3604 (9)	0.6378 (5)	0.7722 (6)	0.069 (3)
C(32)	1.437 (1)	0.7171 (6)	0.8243 (8)	0.097 (4)
C(33)	0.3605 (9)	-0.0384 (6)	0.8754 (6)	0.083 (3)

The C(sp<sup>3</sup>)-C(sp<sup>3</sup>) bond lengths range from 1.499 (8) to 1.594 (7) Å, with a mean of 1.544 (8) Å. The significant deviations from this mean may be a consequence of the degree of substitution at different C atoms and of the presence of steric strain in the molecule. The only single bond between C(sp<sup>2</sup>) atoms, C(11)-C(12), equals 1.470 (8) Å and indicates some degree of conjugation between C(11)=O(3) and C(12)=C(13). The endocyclic bond angles for tetrahedral atoms in rings *A*, *B*, *C*, *D* and *E* vary from 106.0 (4) to 118.3 (5)° with a mean of 111.3 (5)°. The large values of the C(4)-C(5)-C(10) [118.3 (5)°] and C(8)-C(9)-C(10) [118.2 (4)°] angles are probably caused by repulsions between neighbouring bulky axial methyl groups.

The molecule is bent progressively from ring *E* to ring *A* as shown by the angles between the mean planes of these rings [*A*-*B* 17.4, *B*-*C* 14.5, *C*-*D* 15.1, *D*-*E* 4.6°]. From Fig. 1 it is clear that rings *A*, *B*, *D* and *E* take a chair conformation with different degree of distortion. The *C*-ring conformation is intermediate between a half-chair and a sofa, but more nearly a sofa as shown by asymmetry parameters (Duax & Norton, 1975)  $\Delta C_s^8 = 10.9$ ,  $\Delta C_2^{8,9} = 14.2^\circ$ ,  $|\phi|_m = 187.0^\circ$ . All ring junctions are *trans*. The H atom at the C(18) asymmetric centre takes an axial position relative to the

*D* and *E* rings. In our previous study of the 18-diastereomeric derivative of the title compound (Gzella *et al.*, 1986) we reported the *D* and *E* rings as *cis*-fused and the H atom at the C(18) asymmetric centre as equatorial to the *D* ring.

The non-bonded distances between C atoms of diaxial methyl groups C(24)...C(25) and C(25)...C(26) amount to 3.319 (10) and 3.284 (9) Å, and are larger than the values expected for non-distorted rings. In a regular chair-form six-membered ring, the non-bonded distances between 1,3 diaxial groups would be 2.52 Å (Gzella *et al.*, 1986).

The C(18)–C(17)–C(28)–O(4) torsion angle is 8.9 (8)°. The same torsion angle in the 18β-diastereomeric derivative amounts to –28.0 (9)° (Gzella *et al.*, 1986).

Table 2. Bond distances (Å) and angles (°) and their standard deviations

C(1)–C(2)	1.539 (8)	C(13)–C(14)	1.516 (8)
C(1)–C(10)	1.545 (7)	C(13)–C(18)	1.514 (8)
C(2)–C(3)	1.499 (8)	C(14)–C(15)	1.557 (8)
C(3)–C(4)	1.535 (7)	C(14)–C(27)	1.563 (7)
C(3)–O(1)	1.462 (7)	C(15)–C(16)	1.534 (8)
C(4)–C(5)	1.556 (8)	C(16)–C(17)	1.532 (8)
C(4)–C(23)	1.555 (8)	C(17)–C(18)	1.530 (8)
C(4)–C(24)	1.543 (8)	C(17)–C(22)	1.548 (8)
C(5)–C(6)	1.531 (7)	C(17)–C(28)	1.511 (8)
C(5)–C(10)	1.558 (7)	C(18)–C(19)	1.531 (8)
C(6)–C(7)	1.537 (8)	C(19)–C(20)	1.545 (8)
C(7)–C(8)	1.532 (8)	C(20)–C(21)	1.511 (9)
C(8)–C(9)	1.575 (7)	C(20)–C(29)	1.557 (9)
C(8)–C(14)	1.594 (7)	C(20)–C(30)	1.537 (9)
C(8)–C(26)	1.546 (8)	C(21)–C(22)	1.539 (10)
C(9)–C(10)	1.560 (8)	C(28)–O(4)	1.199 (7)
C(9)–C(11)	1.513 (8)	C(28)–O(5)	1.340 (7)
C(10)–C(25)	1.551 (7)	C(31)–O(1)	1.353 (7)
C(11)–C(12)	1.470 (8)	C(31)–O(2)	1.192 (8)
C(11)–O(3)	1.224 (6)	C(31)–C(32)	1.508 (10)
C(12)–C(13)	1.332 (7)	C(33)–O(5)	1.460 (7)
C(3)–O(1)–C(31)	116.1 (5)	C(11)–C(12)–C(13)	125.6 (6)
C(28)–O(5)–C(33)	114.8 (5)	C(12)–C(13)–C(14)	120.5 (5)
C(2)–C(1)–C(10)	112.2 (4)	C(12)–C(13)–C(18)	122.1 (6)
C(1)–C(2)–C(3)	110.5 (5)	C(14)–C(13)–C(18)	117.1 (4)
C(2)–C(3)–C(4)	114.3 (5)	C(8)–C(14)–C(13)	110.8 (4)
C(2)–C(3)–O(1)	108.4 (4)	C(8)–C(14)–C(15)	108.9 (4)
C(4)–C(3)–O(1)	107.9 (4)	C(8)–C(14)–C(27)	112.4 (4)
C(3)–C(4)–C(5)	106.3 (4)	C(13)–C(14)–C(15)	111.5 (5)
C(3)–C(4)–C(23)	106.8 (5)	C(13)–C(14)–C(27)	105.6 (4)
C(3)–C(4)–C(24)	112.4 (5)	C(15)–C(14)–C(27)	107.5 (4)
C(5)–C(4)–C(23)	108.9 (5)	C(14)–C(15)–C(16)	113.9 (5)
C(5)–C(4)–C(24)	114.4 (5)	C(15)–C(16)–C(17)	112.1 (4)
C(23)–C(4)–C(24)	107.8 (5)	C(16)–C(17)–C(18)	108.1 (5)
C(4)–C(5)–C(6)	113.8 (5)	C(16)–C(17)–C(22)	109.7 (4)
C(4)–C(5)–C(10)	118.3 (5)	C(16)–C(17)–C(28)	109.5 (5)
C(6)–C(5)–C(10)	110.2 (5)	C(18)–C(17)–C(22)	109.1 (5)
C(5)–C(6)–C(7)	109.3 (5)	C(18)–C(17)–C(28)	110.8 (4)
C(6)–C(7)–C(8)	114.8 (4)	C(22)–C(17)–C(28)	109.6 (5)
C(7)–C(8)–C(9)	110.1 (5)	C(13)–C(18)–C(17)	113.5 (5)
C(7)–C(8)–C(14)	109.9 (4)	C(13)–C(18)–C(19)	114.3 (5)
C(7)–C(8)–C(26)	109.0 (5)	C(17)–C(18)–C(19)	113.2 (5)
C(9)–C(8)–C(14)	108.4 (4)	C(18)–C(19)–C(20)	114.2 (5)
C(9)–C(8)–C(26)	109.6 (4)	C(19)–C(20)–C(21)	109.9 (5)
C(14)–C(8)–C(26)	109.8 (5)	C(19)–C(20)–C(29)	111.0 (5)
C(8)–C(9)–C(10)	118.2 (4)	C(19)–C(20)–C(30)	108.8 (5)
C(8)–C(9)–C(11)	108.5 (5)	C(21)–C(20)–C(29)	110.7 (5)
C(10)–C(9)–C(11)	116.8 (4)	C(21)–C(20)–C(30)	109.5 (5)
C(1)–C(10)–C(5)	107.9 (4)	C(29)–C(20)–C(30)	106.9 (6)
C(1)–C(10)–C(9)	107.4 (4)	C(20)–C(21)–C(22)	112.8 (6)
C(1)–C(10)–C(25)	107.9 (4)	C(17)–C(22)–C(21)	111.1 (5)
C(5)–C(10)–C(9)	106.0 (4)	C(17)–C(22)–O(4)	126.1 (6)
C(5)–C(10)–C(25)	114.6 (4)	C(17)–C(28)–O(5)	112.6 (5)
C(9)–C(10)–C(25)	112.8 (5)	O(4)–C(28)–O(5)	121.3 (6)
C(9)–C(11)–C(12)	116.3 (4)	O(1)–C(31)–C(32)	110.2 (6)
O(3)–C(11)–C(9)	123.8 (5)	O(2)–C(31)–C(32)	125.2 (7)
O(3)–C(11)–C(12)	119.9 (5)	O(1)–C(31)–O(2)	124.6 (7)

The atoms of the 3β-acetate group are coplanar. The torsion angle C(2)–C(3)–O(1)–C(31) equals 87.2 (5)° and indicates a synclinal orientation of this group relative to the C(2)–C(3) bond. The C(31)=O(2) carbonyl group is synperiplanar to the O(1)–C(3) bond, the torsion angle C(3)–O(1)–C(31)–O(2) being 7.0 (7)°.

A projection of the crystal structure is shown in Fig. 3. There are no intermolecular separations less than the sum of the respective C–C, C–O or O–O van der Waals radii.

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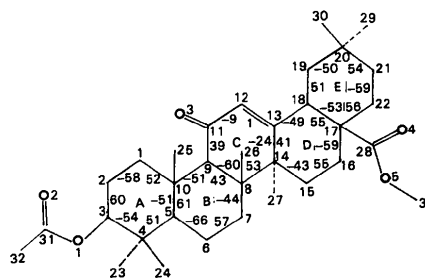


Fig. 1. Numbering of atoms and endocyclic torsion angles (°).

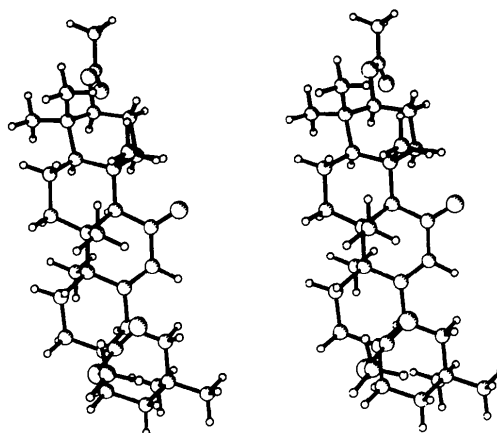


Fig. 2. Stereodrawing of the molecule.

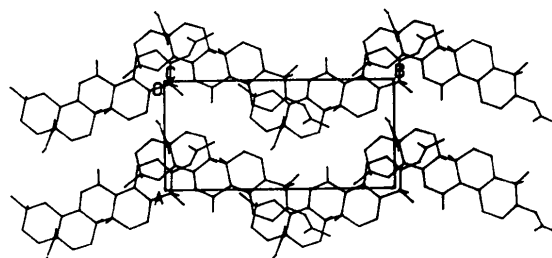


Fig. 3. Projection of the crystal structure along the *c* axis.

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## The Structure of 20-Epiheyneanine Acetone Solvate

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**Abstract.** Methyl 1,2,3,4,4a,6,7,12b-octahydro-4-(1-hydroxyethyl)-2,5-methano-5*H*,12*H*-[1]benzazepino-[4,5-*b*]indol-12*b*-acetate acetone solvate. C<sub>21</sub>H<sub>26</sub>N<sub>2</sub>O<sub>3</sub>·C<sub>3</sub>H<sub>6</sub>O, *M<sub>r</sub>* = 412.6, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 8.626 (1), *b* = 13.490 (4), *c* = 19.181 (3) Å, *V* = 2232 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 1.16 (floatation), *D<sub>x</sub>* = 1.22 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.048 mm<sup>-1</sup>, *F*(000) = 888, room temperature, final *R* = 0.056 for 907 observed reflections. An indole ring system is fused with a seven-membered nitrogen-containing ring with two sides fused to a bicyclic isoquinuclidine ring system. The bond lengths and angles are normal. The indole rings are planar. The seven-membered tetrahydro-1*H*-azepine ring and the isoquinuclidine rings have distorted boat conformations. The isoquinuclidine cage is twisted.

**Introduction.** The indole alkaloid 20-epiheyneanine (Fig. 1) is the major alkaloid (Fonteles, Jerram, Matos & Alquist, 1974) isolated from the crude extract of the root bark of *Peschiera affinis* (Muell Arg.) Miers, a characteristic shrub of the sandy plateaus around Fortaleza, Brazil. The molecular formula was determined by high-resolution mass spectrometry. The structure was elucidated by UV, IR and <sup>1</sup>H NMR spectra

(Matos, Braz F<sup>o</sup>, Gottlieb, Machado & Madruga, 1976) and defined as an epimer of heyneanine (Govindachari, Joshi, Saksema, Sathe & Viswanathan, 1965; Kupchan, Cassady & Telang, 1966). The <sup>13</sup>C NMR spectra of these two compounds confirmed the different stereochemistry of the C(20) atom and furnished its relative configuration (Wenkert *et al.*, 1976). An intramolecular hydrogen bond between the hydroxyl group and N(4) was also inferred from the invariance of the IR 3470 cm<sup>-1</sup> band upon dilution in CHCl<sub>3</sub> and by <sup>13</sup>C NMR, measured in CDCl<sub>3</sub>. The crystallographic analysis was undertaken to clarify the stereochemistry of the hydroxyl group and the relative position of H(C20) in the crystal.

**Experimental.** Transparent single crystal, obtained by slow evaporation of an acetone solution, with dimensions 0.20 × 0.43 × 0.48 mm; all data collected using an Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo *K*α radiation; 18 reflections used for a least-squares determination of cell constants in the range 10 < θ < 19°; systematic absences (*h* = 2*n* + 1 for *h*00, *k* = 2*n* + 1 for 0*k*0, *l* = 2*n* + 1 for 00*l*) consistent with space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>; 1664 unique measured reflections, 1 ≤ θ ≤ 22°, 0 ≤