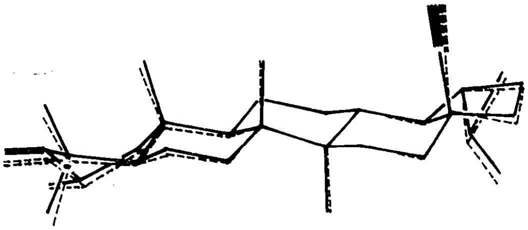


Fig. 3. Relative conformations of molecules 1 and 2 (dashed lines) illustrated by superimposing the *C* ring as a reference element.

differ further by a twist about the length of the triterpenoid skeleton. This effect is probably best measured by the pseudo torsion angle C(25)—C(10)—C(14)—C(27) which has values of −170·2 (4) and −174·3 (4)° for molecules 1 and 2 respectively.

The structure is characterized by a relatively dense packing with numerous intermolecular contacts in the usual van der Waals range. On close inspection it appears that dissimilarities in the surroundings of molecules 1 and 2 can be clearly related to the differences in their intramolecular geometry. Crystal-packing effects can therefore easily be the factor responsible for the slightly different conformations of the two independent molecules in the present structure.

References

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* A**30**, 129–147.

Duax, W. L., Strong, P. D. & Rohrer, D. C. (1980). *Acta Cryst.* B**36**, 824–827.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271–383.

Fowell, D. T., Melsom, B. G. & Smith, G. W. (1978). *Acta Cryst.* B**34**, 2244–2250.

Klinot, J., Buděšínský, M., Klinotová, E. & Vystrčil, A. (1986). *Collect. Czech. Chem. Commun,* **51**, 593–610.

Klinot, J., Světlý, J., Klinotová, E. & Vystrčil, A. (1985). *Collect. Czech. Chem. Commun.* **50**, 2918–2924.

Langs, D. A., Duax, W. L., Carrell, H. L., Berman, H. & Caspi, E. (1977). *J. Org. Chem.* **42**, 2134–2137.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sklenář, I. & Petříček, V. (1973). *TLS.* Institute of Physics, Czechoslovak Academy of Sciences, Prague, Czechoslovakia.

Acta Cryst. (1987). C**43**, 2214–2216

Structure of 2α-Methyl-3-oxolupane-28-nitrile

By J. Podlahová and J. Podlaha

Department of Chemistry, Charles University, 128 40 *Prague, Czechoslovakia*

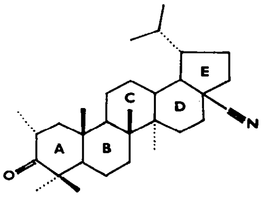
and K. Malý and V. Petříček

Institute of Physics, Czechoslovak Academy of Sciences, 180 40 *Prague, Czechoslovakia*

(*Received* 23 *March* 1987; *accepted* 2 *July* 1987)

Abstract. $C_{31}H_{49}NO$, $M_r = 451·75$, orthorhombic, $P2_12_12_1$, $a = 14·414$ (4), $b = 17·631$ (7), $c = 10·631$ (2) Å, $V = 2702$ (1) Å^3, $Z = 4$, $D_m = 1·094$ (8), $D_x = 1·110$ (1) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å $\mu = 0·061$ mm^{-1}, $F(000) = 1000$, $T = 296$ K, $R = 0·0757$ for 1759 unique observed reflections. The most important feature of the structure is the nearly ideal chair conformation of the triterpenoid *A* ring. The *B, C, D* and *E* rings are in the usual chair, chair, chair and distorted envelope conformations, respectively, with ring junctions all *trans*. The triterpenoid skeleton is bent into a shallow V shape.

Introduction. The study of the title compound was carried out as a continuation of a comparative study of *A*-ring conformation in triterpenoid ketones both in solution (Klinot, Buděšínský, Klinotová & Vystrčil, 1986) and in the solid state (Podlahová, Podlaha, Malý & Petříček, 1987).



Experimental. The sample was kindly provided by Professor J. Klinot. Single crystals were grown by slow evaporation of a 1% solution in benzene/octane (1:5 v/v) at room temperature. Under these conditions, two modifications co-crystallize as tiny needles and well formed bipyramids which can be easily separated by

0108-2701/87/112214-03$01.50

flotation. The modifications yield different powder patterns but behave identically in solution (NMR). Rapid cooling of a hot saturated solution in chloroform gives exclusively the former modification; extensive attempts to obtain it in a habit suitable for crystal structure determination were, however, unsuccessful. The latter modification was therefore used for data collection. The density was determined by flotation in an aqueous $ZnBr_2$ solution. The measurement and refinement data are summarized in Table 1.

Table 1. *Measurement and refinement data*

Crystal dimensions (mm)	0·25 × 0·27 × 0·30
Number of reflections for lattice-parameter determination	25 ($10 < 2\theta < 19°$)
Diffractometer	Syntex $P2_1$
Scan mode	θ–2θ
Maximum value of $(\sin\theta)/\lambda$ (Å^{-1})	0·66
Standard reflections (variation)	3 after every 47 (<3%)
Absorption correction	None
$h\in$, $k\in$, $l\in$	⟨0,19⟩, ⟨0,23⟩, ⟨0,14⟩
Number of reflections measured	3668
Number of reflections used $[I \geq 1{\cdot}96\sigma(I)]$	1759
Residual electron density (e Å^{-3})	0·26, −0·26
$(\Delta/\sigma)_{\max}$	0·342
Function minimized	$w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weight	$\sigma^{-2}(F_o)$, derived from $\sigma(F_o)/F_o = \frac{1}{2}\,\sigma_2(I)/I$ and $\sigma_2(I) = [\sigma_1^2(I) + (0{\cdot}06I)^2]^{1/2}$
R, wR	0·0757, 0·0678

Table 2. *Final coordinates* (×10^4) *of non*-H *atoms with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq} (Å^2 × 10^3)
C(1)	3437 (4)	−637 (3)	2515 (5)	37 (2)
C(2)	4213 (4)	−1219 (3)	2468 (6)	39 (2)
C(3)	5004 (4)	−949 (3)	1626 (7)	49 (2)
C(4)	5382 (4)	−149 (3)	1889 (6)	43 (2)
C(5)	4537 (4)	412 (3)	1984 (5)	32 (2)
C(6)	4814 (4)	1236 (3)	2177 (6)	39 (2)
C(7)	3999 (4)	1760 (3)	1857 (6)	38 (2)
C(8)	3112 (4)	1591 (3)	2619 (5)	33 (2)
C(9)	2910 (4)	726 (3)	2608 (5)	29 (2)
C(10)	3736 (4)	180 (3)	2890 (5)	33 (2)
C(11)	2027 (4)	549 (3)	3376 (6)	44 (2)
C(12)	1188 (4)	975 (3)	2907 (6)	48 (2)
C(13)	1351 (4)	1837 (3)	2784 (5)	33 (2)
C(14)	2244 (4)	2024 (3)	2014 (5)	34 (2)
C(15)	2405 (4)	2900 (3)	2009 (6)	44 (2)
C(16)	1568 (4)	3353 (3)	1565 (6)	50 (2)
C(17)	706 (4)	3152 (3)	2354 (6)	46 (2)
C(18)	515 (4)	2296 (3)	2280 (5)	40 (2)
C(19)	−468 (4)	2208 (3)	2898 (6)	42 (2)
C(20)	−1018 (5)	1508 (4)	2529 (6)	58 (3)
C(21)	−952 (5)	2968 (4)	2543 (7)	62 (3)
C(22)	−227 (4)	3488 (3)	1927 (6)	55 (2)
C(23)	5995 (5)	85 (4)	759 (7)	62 (3)
C(24)	6011 (4)	−177 (4)	3053 (7)	56 (2)
C(25)	4021 (4)	177 (3)	4306 (5)	38 (2)
C(26)	3287 (5)	1887 (3)	3982 (5)	48 (2)
C(27)	2105 (4)	1766 (4)	614 (5)	45 (2)
C(28)	865 (5)	3380 (4)	3675 (7)	50 (3)
C(29)	−1191 (6)	1428 (6)	1110 (8)	116 (5)
C(30)	−1938 (4)	1483 (4)	3253 (8)	74 (3)
C(31)	3844 (4)	−1997 (3)	2046 (7)	57 (3)
N	1004 (5)	3583 (4)	4690 (6)	74 (3)
O	5318 (4)	−1340 (3)	815 (5)	91 (2)

Structure solution and refinement: direct methods; F magnitudes used in full-matrix least-squares refinement; scale factor, positions and anisotropic temperature factors for non-H atoms refined simultaneously in two blocks; H atoms fixed in calculated positions; programs used: *MULTAN*80 (Main *et al.*, 1980), *SHELX*76 (Sheldrick, 1976); source of atomic scattering factors: Cromer & Mann (1968). Obviously, the relatively poor diffraction by the crystals available (less than 50% observed reflections from the total measured) is responsible for the high value of the final R factor.*

Discussion. The final coordinates are listed in Table 2 and bond distances and angles are summarized in Table

Table 3. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·519 (7)	C(11)–C(12)	1·508 (8)
C(1)–C(10)	1·555 (7)	C(12)–C(13)	1·542 (8)
C(2)–C(3)	1·526 (9)	C(13)–C(14)	1·560 (8)
C(2)–C(31)	1·538 (8)	C(13)–C(18)	1·549 (8)
C(3)–O	1·193 (9)	C(14)–C(15)	1·562 (7)
C(3)–C(4)	1·537 (8)	C(14)–C(27)	1·570 (8)
C(4)–C(5)	1·572 (8)	C(15)–C(16)	1·521 (8)
C(4)–C(23)	1·547 (9)	C(16)–C(17)	1·539 (9)
C(4)–C(24)	1·534 (9)	C(17)–C(18)	1·536 (8)
C(5)–C(6)	1·521 (7)	C(17)–C(22)	1·537 (9)
C(5)–C(10)	1·559 (7)	C(17)–C(28)	1·479 (10)
C(6)–C(7)	1·531 (7)	C(18)–C(19)	1·570 (8)
C(7)–C(8)	1·542 (8)	C(19)–C(20)	1·518 (9)
C(8)–C(9)	1·553 (7)	C(19)–C(21)	1·558 (9)
C(8)–C(14)	1·601 (8)	C(20)–C(29)	1·535 (11)
C(8)–C(26)	1·560 (8)	C(20)–C(30)	1·535 (9)
C(9)–C(10)	1·559 (7)	C(21)–C(22)	1·536 (9)
C(9)–C(11)	1·545 (8)	C(28)–N	1·154 (10)
C(10)–C(25)	1·560 (8)		

C(2)–C(1)–C(10)	115·5 (5)	C(1)–C(2)–C(3)	110·0 (4)
C(1)–C(2)–C(31)	110·0 (5)	C(3)–C(2)–C(31)	111·4 (5)
C(2)–C(3)–C(4)	116·4 (4)	C(2)–C(3)–O	121·8 (5)
C(4)–C(3)–O	121·8 (6)	C(3)–C(4)–C(5)	108·3 (5)
C(3)–C(4)–C(23)	107·8 (5)	C(3)–C(4)–C(24)	109·1 (5)
C(5)–C(4)–C(23)	108·9 (5)	C(5)–C(4)–C(24)	115·2 (5)
C(23)–C(4)–C(24)	107·3 (5)	C(4)–C(5)–C(6)	114·0 (4)
C(4)–C(5)–C(10)	116·7 (4)	C(6)–C(5)–C(10)	111·2 (4)
C(5)–C(6)–C(7)	110·2 (4)	C(6)–C(7)–C(8)	113·8 (4)
C(7)–C(8)–C(9)	109·9 (4)	C(7)–C(8)–C(14)	110·2 (4)
C(7)–C(8)–C(26)	106·8 (4)	C(9)–C(8)–C(14)	108·6 (4)
C(9)–C(8)–C(26)	111·4 (4)	C(14)–C(8)–C(26)	109·9 (4)
C(8)–C(9)–C(10)	117·5 (4)	C(8)–C(9)–C(11)	110·4 (4)
C(10)–C(9)–C(11)	113·8 (4)	C(1)–C(10)–C(5)	106·8 (4)
C(1)–C(10)–C(9)	108·1 (4)	C(1)–C(10)–C(25)	108·5 (4)
C(5)–C(10)–C(9)	106·5 (4)	C(5)–C(10)–C(25)	113·7 (4)
C(9)–C(10)–C(25)	112·9 (4)	C(9)–C(11)–C(12)	112·7 (5)
C(11)–C(12)–C(13)	113·4 (5)	C(12)–C(13)–C(14)	112·3 (4)
C(12)–C(13)–C(18)	115·2 (4)	C(14)–C(13)–C(18)	110·4 (4)
C(8)–C(14)–C(13)	109·5 (4)	C(8)–C(14)–C(15)	110·9 (4)
C(8)–C(14)–C(27)	110·0 (4)	C(13)–C(14)–C(15)	109·5 (4)
C(13)–C(14)–C(27)	109·4 (4)	C(15)–C(14)–C(27)	107·6 (5)
C(14)–C(15)–C(16)	113·7 (5)	C(15)–C(16)–C(17)	110·5 (5)
C(16)–C(17)–C(18)	110·0 (5)	C(16)–C(17)–C(22)	117·2 (5)
C(16)–C(17)–C(28)	109·3 (5)	C(18)–C(17)–C(22)	101·8 (5)
C(18)–C(17)–C(28)	110·1 (5)	C(22)–C(17)–C(28)	108·1 (5)
C(13)–C(18)–C(17)	110·9 (5)	C(13)–C(18)–C(19)	120·4 (5)
C(17)–C(18)–C(19)	103·8 (4)	C(18)–C(19)–C(20)	116·3 (5)
C(18)–C(19)–C(21)	102·5 (5)	C(20)–C(19)–C(21)	113·8 (5)
C(19)–C(20)–C(29)	114·5 (6)	C(19)–C(20)–C(30)	110·1 (6)
C(29)–C(20)–C(30)	110·4 (6)	C(19)–C(21)–C(22)	108·1 (5)
C(17)–C(22)–C(21)	103·9 (5)	C(17)–C(28)–N	177·4 (7)

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44211 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

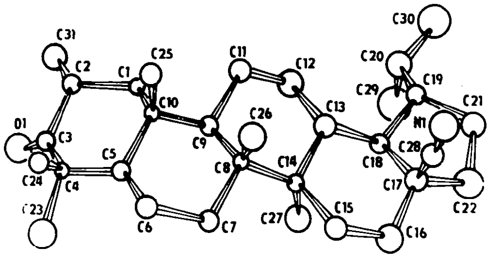


Fig. 1. Perspective view of the molecule with atom numbering. H atoms (omitted for clarity) are given the numbers of their bonding partners.

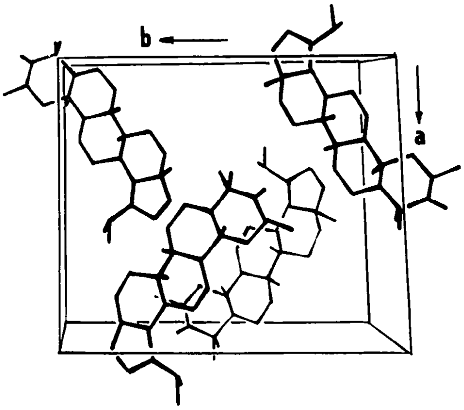


Fig. 2. The unit-cell contents viewed down the *c* axis.

3. Fig. 1 depicts a perspective view of the molecule together with the atom numbering. The unit-cell contents are shown in Fig. 2.

The general features of the structure are closely related to similar triterpenoid and steroid compounds (Cambridge Structural Database, 1986; Duax, Weeks & Rohrer, 1976). The C—C bond lengths average 1·543 (22) Å with only two typical bonds, C(8)—C(14) and C(17)—C(28), outside the 2σ limit of the mean. Similarly, the mean C—C—C angle of 110·8 (3·7)° is exceeded by more than 2σ in three cases, all involving C(18). In accordance with the proposed solution structure (Klinot, Buděšínský, Klinotová & Vystrčil, 1986), the *A*-ring conformation is very close to an ideal chair as reflected in the mean endocyclic torsion angle of ±52 (2)°. The arrangement of the *B, C, D* and *E* rings is normal and corresponds to chair, chair, chair and distorted envelope, respectively, with all *trans* ring junctions.

A remarkable feature of the structure is a distinct bending of the triterpenoid skeleton which is apparent, for example, from the dihedral angle of 156·5° between the mean C(1)C(2)C(4)C(5) and C(13)C(15)C(16)-C(18) planes. It seems likely that this effect is caused by intramolecular constraints, since the crystal packing is very loose (with only four C···C contacts in the usual van der Waals range of 3·7 to 3·9 Å) and hence cannot be assumed to influence significantly the molecular geometry.

References

Cambridge Structural Database (1986). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271–383.

Klinot, J., Buděšínský, M., Klinotová, E. & Vystrčil, A. (1986). *Collect. Czech. Chem. Commun.* **51**, 593–610.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Podlahová, J., Podlaha, J., Malý, K. & Petříček, V. (1987). *Acta Cryst.* C**43**, 2211–2214.

Sheldrick (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1987). C**43**, 2216–2218

X-ray Structure of 6-Deoxymikanokryptin, a New Guaianolide from *Vernonia scorpioides* Pers: 3-Oxo-1α,7α,8β,10β(H)-guaia-4(5),11(13)-dien-8,12-olide

By Dora Gómez C., A. Valentina Rivera, Eldrys Rodulfo de Gil,* Brigita Vogler de Valeri and Jorge Triana

Departamento de Química, Facultad de Ciencias, Universidad de Los Andes, Mérida, Venezuela

(*Received* 12 *December* 1986; *accepted* 6 *July* 1987)

Abstract. $C_{15}H_{18}O_3$, $M_r = 246{\cdot}31$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}800\,(3)$, $b = 9{\cdot}685\,(3)$, $c = 12{\cdot}704\,(4)$ Å, $V = 1329$ Å^3, $Z = 4$, $D_m = 1{\cdot}24$, $D_x = 1{\cdot}232$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}049$ mm^{-1}, $F(000) = 528$, $T = 295$ K, $R = 0{\cdot}055$ for 952 unique reflections with $I > 3\sigma(I)$. The crystal

---

* To whom correspondence should be addressed.