in Fig. 2, molecules forming the respective layers have not the same height above the projection plane *ac* but rather are displaced against each other relative to the *b* axis. The relatively loose packing of the molecules in the layer structure is probably the origin of the slightly different conformations of the two crystallographically independent molecules and also for the high proportion of unobserved data. Apart from the layer structure no particular orientation phenomena or exceptionally short intermolecular distances are observed. On the basis of the room-temperature crystal structure analysis alone no definite conclusions can be drawn regarding a mechanism for the solid-state disproportionation reaction observed upon heating of the title compound.

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

BY A. GZELLA, L. ZAPRUTKO AND U. WRZECIONO*

Department of Organic Chemistry, Faculty of Pharmacy, K. Marcinkowski Medical Academy, ul. Grunwaldzka 6, 60–780 Poznań, Poland

AND M. GDANIEC

Department of Crystallography, Faculty of Chemistry, A. Mickiewicz University, ul. Grunwaldzka 6, 60–780 Poznań, Poland

Abstract. $C_{33}H_{50}O_5$, $M_r = 526 \cdot 40$, monoclinic, $P2_1$, $a = 6 \cdot 824$ (1), $b = 12 \cdot 658$ (2), $c = 17 \cdot 848$ (3) Å, $\beta = 97 \cdot 80$ (1)°, $V = 1527 \cdot 4$ (4) Å³, $D_m = 1 \cdot 15$ (1), $D_x = 1 \cdot 14$ Mg m⁻³, $Z = 2$, $\lambda(Cu\,K\alpha) = 1 \cdot 54178$ Å, $\mu(Cu\,K\alpha) = 0 \cdot 519$ mm⁻¹, $F(000) = 576$, room temperature, final $R = 0 \cdot 070$ for 1762 observed reflections. The molecule consists of five six-membered rings, *D* and *E* rings *cis* fused. Ring *C* has a slightly distorted sofa conformation. The H atom at the C(18) asymmetric centre takes an equatorial position with respect to the *D* ring.

Introduction. The structure investigation of the title compound has been undertaken to facilitate the interpretation of ¹H NMR, ¹³C NMR and MS data of pentacyclic triterpenoids with the 11-oxo-18β-olean-12-ene skeleton.

Experimental. The title compound, m.p. 522–524 K, $[\alpha]_D^{22°C} = +79°$ ($CHCl_3$) was prepared by the method

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* To whom correspondence should be addressed.

described by Corey & Ursprung (1956) [m.p. 516·7–518·2 K (Corey & Ursprung, 1956), m.p. 515–517 K, $[\alpha]_D = +83°$ (Barton & Holnes, 1952), m.p. 525–526 K (Ruzicka, Cohen, Furter & Sluys-Veer, 1938)], needle crystals recrystallized from methanol, D_m by flotation, crystal dimensions 0·4 × 0·3 × 0·1 mm, Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation, lattice parameters from setting angles of 15 reflections, θ–2θ scan mode, $2\theta \le 115°$, two standard reflections monitored every 100 reflections, variation < 2%, 2478 reflections measured, 2093 unique reflections, $R_{int} = 0·024$, range of hkl: h −7→7, k 0→13, l 0→19, profile analysis according to Lehmann & Larsen (1974), no absorption correction, 1762 reflections with $I \ge 1·96\ \sigma(I)$ considered observed and used in the refinement, structure solved by direct methods using MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refinement by full-matrix least-squares method, refined $N = 343$ parameters, H atoms generated from assumed geometries and included as fixed isotropic contribution to F_c, final refinement: anisotropic non-H atoms, $w^{-1} = \sigma^2(F)$ and empirical isotropic extinction parameter x used to correct F_c according to $F'_c = F_c\ (1 - xF/\sin\theta)$, x converged at 0·010 (1), final R factors minimized to $R = 0·070$ and $wR = 0·065$, $(\Delta/\sigma)_{max} = 0·02$, max. and min. height in the final ΔF map 0·23 and −0·22 e Å^{-3}, y coordinate of O(5) held fixed to define the origin.

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 were taken from International Tables for X-ray Crystallography (1974).

Discussion. Atomic coordinates, bond lengths and angles are given in Tables 1 and 2, respectively. The numbering system and the values of torsion angles are given in Fig. 1.* A stereoview of the molecule is given in Fig. 2.

Bonds between formally sp^3-hybridized carbon atoms are in the range 1·501 (11)–1·586 (9) Å, with a mean value 1·545 (10) Å. The deviations from their average length are probably due both to the presence of electron-withdrawing substituents and to the ring closure. It should be noticed that the longest values of the $C(sp^3)$–$C(sp^3)$ bond lengths are between fully substituted carbon atoms. The only single bond between sp^2-hybridized C atoms equals 1·456 (10) Å and indicates some degree of resonance between the C(11)–O(3) and C(12)–C(13) double bonds. The

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* 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 43249 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

internal bond angles in the rings A, B, C, D and E for tetrahedral C–C–C bonds vary from 105·1 (6) to 118·6 (6)° with an average of 111·2 (6)°. These interatomic angles are not unusual when compared with similar compounds (Roques, Declercq & Germain, 1978; Campsteyn, Dupont, Lamotte, Dideberg & Vermeire, 1977). The large values of the C(4)–C(5)–C(10) [117·5 (6)°] and C(8)–C(9)–C(10) [118·6 (6)°] angles in rings A and B reflect the ring strain due to the large amount of steric overcrowding of the bulky axial methyl groups. The latter bond angle seems to be influenced additionally by the strain arising from the junction to ring C. The presence of two C(11)–O(3) and C(12)–C(13) double bonds in ring C causes the C(11)–C(12)–C(13)–C(14) fragment to be flat ($\chi^2 = 0·70$).

The molecule is bent progressively from ring D to ring A as shown by the angles between the mean planes of these rings: A–B 17·2 (2), B–C 166·4 (2), C–D 166·1 (3)°. Ring E is at an angle of 120·2 (2)° to ring D.

The non-bonded distances between carbon atoms of diaxial methyl groups C(24)–C(25) and C(25)–C(26) amount to 3·219 (12) Å and 3·318 (11) Å, respectively. In a six-membered ring, the non-bonded distances between 1,3-diaxial methyl groups would have been

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters (Å^2)*

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

	x	y	z	U_{eq}
O(1)	−0·0211 (8)	0·3273 (7)	0·7443 (3)	0·069 (2)
O(2)	−0·336 (1)	0·2746 (8)	0·7459 (4)	0·104 (4)
O(3)	0·523 (1)	0·2011 (7)	1·0719 (3)	0·092 (3)
O(4)	0·6935 (9)	0·4157 (7)	1·3358 (4)	0·088 (3)
O(5)	0·5348 (8)	0·5648	1·3524 (3)	0·067 (2)
C(1)	0·230 (1)	0·2414 (7)	0·9357 (4)	0·055 (3)
C(2)	0·187 (1)	0·2500 (8)	0·8495 (4)	0·062 (3)
C(3)	0·018 (1)	0·3248 (8)	0·8271 (4)	0·057 (3)
C(4)	0·063 (1)	0·4385 (8)	0·8564 (4)	0·052 (3)
C(5)	0·113 (1)	0·4272 (7)	0·9441 (4)	0·044 (3)
C(6)	0·143 (1)	0·5343 (7)	0·9857 (4)	0·054 (3)
C(7)	0·118 (1)	0·5192 (7)	1·0696 (4)	0·048 (3)
C(8)	0·2586 (9)	0·4353 (7)	1·1104 (4)	0·041 (2)
C(9)	0·268 (1)	0·3350 (7)	1·0607 (4)	0·043 (3)
C(10)	0·279 (1)	0·3491 (7)	0·9746 (4)	0·048 (3)
C(11)	0·413 (1)	0·2586 (8)	1·1029 (4)	0·058 (3)
C(12)	0·424 (1)	0·2545 (8)	1·1849 (4)	0·054 (3)
C(13)	0·325 (1)	0·3180 (8)	1·2259 (4)	0·049 (3)
C(14)	0·183 (1)	0·4015 (8)	1·1871 (4)	0·047 (3)
C(15)	0·171 (1)	0·4995 (7)	1·2378 (4)	0·048 (3)
C(16)	0·173 (1)	0·4739 (8)	1·3226 (4)	0·055 (3)
C(17)	0·350 (1)	0·4058 (8)	1·3542 (4)	0·054 (3)
C(18)	0·341 (1)	0·3016 (8)	1·3111 (4)	0·050 (3)
C(19)	0·176 (1)	0·2279 (8)	1·3319 (4)	0·063 (3)
C(20)	0·187 (1)	0·2047 (9)	1·4170 (5)	0·076 (4)
C(21)	0·190 (1)	0·3130 (9)	1·4587 (4)	0·074 (4)
C(22)	0·357 (1)	0·3861 (9)	1·4409 (4)	0·066 (3)
C(23)	0·128 (1)	0·5037 (9)	0·8376 (4)	0·070 (3)
C(24)	0·224 (1)	0·4925 (8)	0·8162 (4)	0·067 (3)
C(25)	0·490 (1)	0·3834 (8)	0·9581 (4)	0·057 (3)
C(26)	0·464 (1)	0·4889 (8)	1·1267 (4)	0·058 (3)
C(27)	−0·029 (1)	0·3491 (8)	1·1735 (4)	0·054 (3)
C(28)	0·545 (1)	0·4599 (8)	1·3456 (4)	0·058 (3)
C(29)	0·000 (2)	0·143 (1)	1·4297 (5)	0·105 (5)
C(30)	0·373 (2)	0·138 (1)	1·4462 (5)	0·087 (4)
C(31)	−0·202 (2)	0·3014 (9)	0·7121 (5)	0·079 (4)
C(32)	−0·221 (2)	0·306 (1)	0·6276 (5)	0·092 (4)
C(33)	0·709 (1)	0·6246 (9)	1·3393 (5)	0·077 (4)

2·52 Å if the ring took a regular chair form (Spirlet, Dupont, Dideberg & Kapundu, 1980).

Fig. 1 reports the torsion angles characterizing the molecular conformation. An analysis of these data shows that rings A and E have a chair conformation. The B and D rings form a slightly flattened chair partially because of the connection with ring C. The C ring has a slightly distorted sofa form with the corresponding asymmetry parameter (Duax & Norton, 1975) $\Delta C_s^8 = 4 \cdot 6°$, $|\varphi_m| = 187 \cdot 7°$.

The E ring is cis-fused at the C(17)—C(18) junction. The hydrogen atom at the C(18) asymmetric centre takes an equatorial position with respect to the D ring.

In the 3β-acetate group the atoms O(1), C(31), O(2) and C(32) are coplanar. The torsion angle C(2)—

C(3)—O(1)—C(31) equals 122·1 (9)° and indicates an anticlinal conformation of this group with respect to the C(2)—C(3) bond. The carbonyl group C(31)—O(2) is synperiplanar to C(3); the torsion angle C(3)—O(1)—C(31)—O(2) equals −1·0 (12)°.

Fig. 3 shows molecular packing in the unit cell. The following intermolecular contacts are shorter than the sum of van der Waals radii of corresponding atoms: C(6)—H···O(3^i) = 3·369 (11) Å, H(62)···O(3^i) = 2·43 Å, C(6)—H(62)···O(3^i) = 144° and C(16)—H···O(4^{ii}) = 3·392 (10) Å, H(161)···O(4^{ii}) = 2·38 Å,

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

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

C(1)—C(2)	1·529 (10)	C(13)—C(14)	1·533 (10)
C(1)—C(10)	1·546 (10)	C(13)—C(18)	1·525 (10)
C(2)—C(3)	1·501 (11)	C(14)—C(15)	1·545 (10)
C(3)—C(4)	1·547 (11)	C(14)—C(27)	1·584 (10)
C(3)—O(1)	1·465 (8)	C(15)—C(16)	1·545 (9)
C(4)—C(5)	1·564 (9)	C(16)—C(17)	1·527 (11)
C(4)—C(23)	1·544 (11)	C(17)—C(18)	1·524 (11)
C(4)—C(24)	1·549 (10)	C(17)—C(22)	1·560 (10)
C(5)—C(6)	1·545 (10)	C(17)—C(28)	1·527 (11)
C(5)—C(10)	1·545 (10)	C(18)—C(19)	1·545 (11)
C(6)—C(7)	1·542 (9)	C(19)—C(20)	1·539 (10)
C(7)—C(8)	1·547 (9)	C(20)—C(21)	1·559 (13)
C(8)—C(9)	1·554 (10)	C(20)—C(29)	1·538 (13)
C(8)—C(14)	1·586 (9)	C(20)—C(30)	1·550 (13)
C(8)—C(26)	1·550 (10)	C(21)—C(22)	1·537 (12)
C(9)—C(10)	1·560 (9)	C(28)—O(4)	1·190 (10)
C(9)—C(11)	1·510 (10)	C(28)—O(5)	1·336 (10)
C(10)—C(25)	1·566 (10)	C(31)—O(1)	1·331 (11)
C(11)—C(12)	1·456 (10)	C(31)—O(2)	1·212 (11)
C(11)—O(3)	1·230 (9)	C(31)—C(32)	1·497 (12)
C(12)—C(13)	1·333 (10)	C(33)—O(5)	1·455 (10)

C(3)—O(1)—C(31)	117·6 (7)	C(11)—C(12)—C(13)	124·6 (7)
C(28)—O(5)—C(33)	116·6 (7)	C(12)—C(13)—C(14)	120·4 (6)
C(2)—C(1)—C(10)	112·9 (6)	C(12)—C(13)—C(18)	119·4 (7)
C(1)—C(2)—C(3)	110·4 (6)	C(14)—C(13)—C(18)	120·0 (7)
C(2)—C(3)—C(4)	112·8 (6)	C(8)—C(14)—C(13)	108·5 (5)
C(2)—C(3)—O(1)	108·2 (7)	C(8)—C(14)—C(15)	109·8 (6)
C(4)—C(3)—O(1)	108·8 (6)	C(8)—C(14)—C(27)	112·2 (5)
C(3)—C(4)—C(5)	105·1 (6)	C(13)—C(14)—C(15)	111·7 (6)
C(3)—C(4)—C(23)	107·5 (7)	C(13)—C(14)—C(27)	106·8 (6)
C(3)—C(4)—C(24)	111·9 (6)	C(15)—C(14)—C(27)	107·8 (6)
C(5)—C(4)—C(23)	109·3 (6)	C(14)—C(15)—C(16)	114·3 (7)
C(5)—C(4)—C(24)	115·5 (6)	C(15)—C(16)—C(17)	112·5 (6)
C(23)—C(4)—C(24)	107·2 (6)	C(16)—C(17)—C(18)	109·2 (6)
C(4)—C(5)—C(6)	113·4 (6)	C(16)—C(17)—C(22)	112·0 (6)
C(4)—C(5)—C(10)	117·5 (6)	C(16)—C(17)—C(28)	111·5 (7)
C(6)—C(5)—C(10)	110·5 (6)	C(18)—C(17)—C(22)	110·9 (7)
C(5)—C(6)—C(7)	109·4 (6)	C(18)—C(17)—C(28)	108·1 (7)
C(6)—C(7)—C(8)	113·4 (6)	C(22)—C(17)—C(28)	105·1 (6)
C(7)—C(8)—C(9)	111·3 (5)	C(13)—C(18)—C(17)	112·2 (7)
C(7)—C(8)—C(14)	109·7 (5)	C(13)—C(18)—C(19)	111·4 (6)
C(7)—C(8)—C(26)	106·1 (6)	C(17)—C(18)—C(19)	112·3 (6)
C(9)—C(8)—C(14)	108·3 (6)	C(18)—C(19)—C(20)	114·4 (7)
C(9)—C(8)—C(26)	111·1 (6)	C(19)—C(20)—C(21)	107·5 (7)
C(14)—C(8)—C(26)	110·3 (5)	C(19)—C(20)—C(29)	108·2 (8)
C(8)—C(9)—C(10)	118·6 (6)	C(19)—C(20)—C(30)	111·2 (7)
C(8)—C(9)—C(11)	108·3 (6)	C(21)—C(20)—C(29)	109·5 (8)
C(10)—C(9)—C(11)	116·6 (6)	C(21)—C(20)—C(30)	111·1 (8)
C(1)—C(10)—C(5)	107·6 (6)	C(29)—C(20)—C(30)	109·3 (8)
C(1)—C(10)—C(9)	107·8 (6)	C(20)—C(21)—C(22)	113·0 (7)
C(1)—C(10)—C(25)	108·0 (6)	C(17)—C(22)—C(21)	112·1 (7)
C(5)—C(10)—C(9)	106·7 (5)	C(17)—C(28)—O(4)	125·2 (9)
C(5)—C(10)—C(25)	113·8 (6)	C(17)—C(28)—O(5)	112·3 (8)
C(9)—C(10)—C(25)	112·7 (6)	O(4)—C(28)—O(5)	122·5 (9)
C(9)—C(11)—C(12)	117·8 (7)	O(1)—C(31)—C(32)	112·0 (10)
O(3)—C(11)—C(9)	123·6 (7)	O(2)—C(31)—C(32)	123·0 (10)
O(3)—C(11)—C(12)	118·6 (8)	O(1)—C(31)—O(2)	125·0 (9)
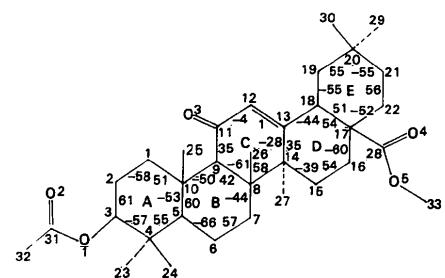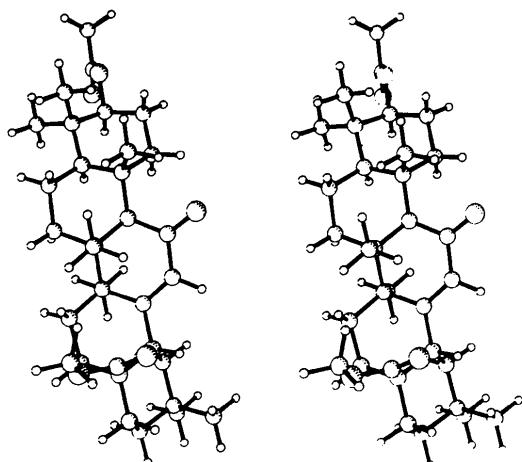
Fig. 2. Stereodrawing of the molecule.
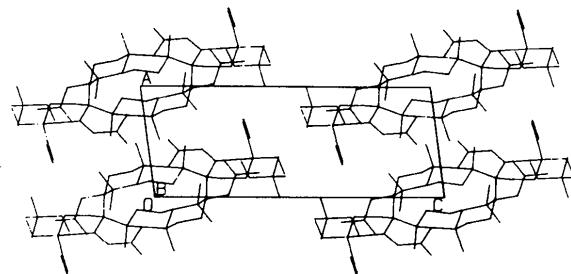
Fig. 3. Projection of the crystal structure along the b axis.

$C(16)-H(161)\cdots O(4^{ii}) = 155°$; with symmetry codes (i) $1-x$, $\frac{1}{2}+y$, $2-z$; (ii) $x-1$, y, z. However, the lack of electron-withdrawing groups in the close neighbourhood of C(6) and C(16) does not allow us to consider this contact as a hydrogen bond (Taylor & Kennard, 1982).

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Structures of 4,5,15,16-Tetraacetoxy[2.2]paracyclophane and Two Bis(crown-6)[2.2]paracyclophanes*

BY CAROLYN B. KNOBLER, EMILY F. MAVERICK, KEITH M. PARKER, KENNETH N. TRUEBLOOD,† ROBERT L. WEISS, DONALD J. CRAM AND ROGER C. HELGESON

J. D. McCullough Laboratory for X-ray Crystallography, Department of Chemistry and Biochemistry, University of California, Los Angeles, California 90024, USA

Abstract. (1): 4,5,15,16-Tetraacetoxy[2.2]paracyclophane, $C_{24}H_{24}O_8$, $M_r = 440.4$, orthorhombic, *Pbca*, $a = 13.543$ (2), $b = 9.411$ (1), $c = 16.997$ (1) Å, $V = 2166.3$ (4) Å^3, $Z = 4$, $D_x = 1.350$, $D_m = 1.322$ (4) g cm^{-3} (flotation), $\lambda(Cu\,K\alpha) = 1.5418$ Å, $\mu = 8.09$ cm^{-1}, $F(000) = 928$, $T = 295$ K, $R = 0.0363$ for 1765 reflections with $I > 3\sigma(I)$. (2): 4,7,13,16-Bis(22-crown-6)[2.2]paracyclophane, $C_{36}H_{52}O_{12}$, $M_r = 676.8$, monoclinic, $P2_1/c$, $T = 115$ K, $a = 9.049$ (3), $b = 24.760$ (11), $c = 15.617$ (4) Å, $\beta = 104.14$ (2)°, $V = 3393$ Å^3, $Z = 4$, $D_x = 1.325$ (at 115 K), $D_m = 1.286$ (5) (at 296 K) g cm^{-3}, $\lambda(Mo\,K\alpha) = 0.7107$ Å, $\mu = 0.60$ cm^{-1}, $F(000) = 1456$, $R = 0.070$ for 3782 reflections with $|F| > 3\sigma(F)$. (3): 4,5,15,16-Bis(18-crown-6)[2.2]paracyclophane, $C_{36}H_{52}O_{12}$, $M_r = 676.8$, orthorhombic, *Pbca*, $T = 296$ K, $a = 8.347$ (4), $b = 16.939$ (9), $c = 24.330$ (20) Å, $V = 3440$ Å^3, $Z = 4$, $D_x = 1.306$, $D_m = 1.305$ (4) g cm^{-3} (flotation), $\lambda(Cu\,K\alpha) = 1.5418$ Å, $\mu = 7.15$ cm^{-1}, $F(000) = 1456$, $R = 0.074$ for 2336 reflections with $I > 2.5\sigma(I)$. The molecules of (1) and (3), unlike those of (2), are centrosymmetric in the crystal. The geometry of the cyclophane moieties is typical of [2.2]paracyclophane units in other structures; the aromatic rings are characteristically bent into a boat shape. The O atoms attached to these rings are, in each structure, displaced from the planes of the four approximately coplanar aromatic C atoms. In (1) and (3), in which O atoms are bonded *ortho* to one another, these displacements average more than 0.1 Å and are uniformly in the direction of the bending of the aromatic rings, toward the bridging methylene groups. In (2), where the polyether rings join the two aromatic rings in pseudogeminal positions, the oxygen atoms are displaced about 0.06 Å in the opposite direction. The polyether (crown-6) rings in (3) are disordered in two regions; the disorder persists at low temperature. In (2), on the other

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* [2.2]Paracyclophane = tricyclo[8.2.2.2^{4,7}]hexadeca-4,6,10,12,13,15-hexaene. 18-Crown-6 = 1,4,7,10,13,16-hexaoxacyclooctadecane.

† To whom correspondence should be addressed.