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Structure of 2,3-Di-*O*-acetyl-2-*C*-methylerythrono-1,4-lactone

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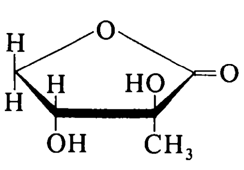
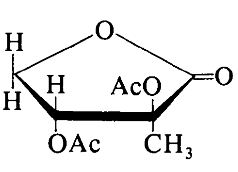
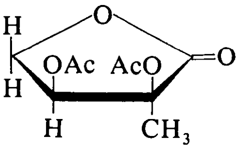
Abstract. $C_9H_{12}O_6$, monoclinic, $P2_1$ (from statistics), $a = 11{\cdot}021\,(3)$, $b = 6{\cdot}440\,(2)$, $c = 7{\cdot}407\,(2)$ Å, $\beta = 92{\cdot}88\,(2)^\circ$, $V = 526{\cdot}7\,(3)$ Å^3, $Z = 2$, $D_m = 1{\cdot}37\,(1)$, $D_x = 1{\cdot}363$ Mg m^{-3}, $\mu = 0{\cdot}95$ mm^{-1} (for Cu $K\alpha$), m.p. 359 (1) K. Refinement with 982 observed reflexions ($\theta_{\max} = 65^\circ$) gave $R_w(F) = 0{\cdot}051$. The *erythro* form has been established. The lactone ring displays an approximate T configuration with the twist axis through the C(2) atom. Packing of the molecules is due to van der Waals interactions only.

Introduction. *Anthyllis tetraphylla* is a variety of the endemic flora of south-west Spain (West Andalucia and Extremadura regions). In the course of a phytochemical study of this plant a compound with the IR characteristics of a γ-lactone group was isolated and chemically identified as 2-*C*-methylthreono-1,4-lactone (I). Recently, the isolation of this compound in tobacco smoke has been reported (Shumache, Green, Best & Newell, 1977). As it was not possible to obtain compound (I) in crystalline form, its acetylated derivative was prepared. Of the two possible isomers [threono (II) and erythrono (III)] the threono form (II) was assigned from IR and PMR spectroscopic data because of the Overhauser effect (Montero de Espinosa, 1978). A detailed molecular conformation was required and an X-ray study of the crystals was suggested in order to confirm the PMR assignment.


(I)


(II)


(III)

Colourless prismatic crystals of the title compound were kindly supplied by Professor J. M. Viguera of the Organic Chemistry Department of this University. Preliminary unit-cell dimensions and space-group information were obtained from oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation. The systematic absences ($0k0$ with k odd) suggest the possible space groups $P2_1$ (C_2^2) and $P2_1/m$ (C_{2h}^2). As $Z = 2$, $P2_1$ was chosen. Accurate cell dimensions were obtained by least-squares refinement from the θ values of 59 reflexions measured on a Philips PW 1100 automated four-circle diffractometer. Intensity data were also collected on this instrument using graphite-monochromated Cu $K\alpha$ radiation and the ω–2θ step scanning mode. Two standard reflexions were monitored after each group of 54 measurements; these did not show any noticeable change in intensity. Diffraction data were collected over the range $2^\circ < \theta < 65^\circ$ to give a total of 982 independent reflexions, of which 873 were classified as observed by the criterion $I > 2\sigma(I)$.

The structure was solved by multisolution weighted tangent-formula refinement (*MULTAN*, Main, Lessinger, Woolfson, Germain & Declercq, 1977) of 135 reflexions with $|E| > 1{\cdot}40$ (approximately ten reflexions per non-hydrogen atom in the asymmetric unit). Scale and overall temperature factors ($B = 4{\cdot}5$ Å^2) were evaluated by the Wilson method. The values of the statistical averages of calculated normalized structure factors were 0·868 and 0·792 for $\langle |E| \rangle$ and $\langle |E^2 - 1| \rangle$ respectively; these indicated the noncentrosymmetric space-group symmetry $P2_1$.

An E map computed with the phase set of the highest combined figure of merit revealed clearly the positions of all the non-hydrogen atoms of the molecule. Refinement of the positional parameters, isotropic temperature factors and a scale factor was carried out by full-matrix least-squares methods [program *CRYLSQ* of the XRAY system (Stewart, Kundell & Baldwin, 1970)] and the R index reduced from 0·22 to 0·12. Anisotropic refinement of temperature factors further reduced R to 0·075. A difference map revealed the positions of most of the H atoms as peaks of maximum density 0·35 e Å^{-3} and the positional parameters for the other H atoms were

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calculated (C—H bond length 1·00 Å, sp^3 geometry in methyl groups). A further refinement including H atoms with the isotropic temperature factor $B = 5$ Å^2 reduced R to 0·051. The weighting scheme was $w = 1/\sigma^2(F)$. The positional parameters and the equivalent isotropic temperature coefficients are given in Table 1.*

Discussion. The atomic labelling is indicated in Fig. 1 which shows the crystal structure as viewed down the *b* axis. Bond lengths and angles for non-hydrogen atoms are given in Table 2. The values obtained agree with those reported for other compounds with a lactone ring (Usher & English, 1978; Sikirica, Vicković & Viterbo, 1979; Fortier & DeTitta, 1979; DeTitta & Fortier, 1979). The lactone ring deviates significantly from planarity. In terms of ring-puckering parameters (Cremer & Pople, 1975), the amplitude of puckering is $q_2 = 0{\cdot}13\ (1)$ Å and the phase angle $\varphi_2 = 337\ (1)°$. The φ_2 value is close to that (342°) appropriate to the T form with a twist axis through C(2). This is also shown by the torsion angles (Table 4) through the ring bonds. O(3) and O(5) are clearly on the same side of the ring, as can be seen from the least-squares planes (Table 3). This is consistent with the *erythro* form, in dis-

Table 1. *Fractional atomic coordinates* (×10^4, *for* H ×10^3) *and isotropic temperature factors*

For H $B = 5$ Å^2.

	x	y	z	B (Å^2)
C(1)	2057 (4)	1354 (10)	1087 (7)	4·9
C(2)	1778 (3)	3235 (10)	2279 (5)	3·8
C(3)	1688 (3)	2325 (11)	4176 (6)	4·2
C(4)	2034 (6)	40 (12)	3947 (9)	6·3
C(5)	623 (3)	4246 (11)	1535 (6)	4·7
C(6)	3871 (3)	4230 (11)	2388 (6)	4·7
C(7)	4685 (5)	6073 (13)	2350 (8)	3·9
C(8)	2047 (3)	4878 (11)	6433 (5)	4·6
C(9)	2977 (5)	5855 (12)	7653 (6)	5·9
O(1)	2041 (3)	−366 (10)	2052 (6)	6·4
O(2)	2198 (4)	1373 (11)	−511 (6)	7·3
O(3)	2697 (2)	4815 (9)	2193 (3)	3·9
O(4)	4165 (3)	2459 (11)	2569 (6)	6·4
O(5)	2488 (2)	3287 (10)	5484 (4)	4·7
O(6)	1016 (3)	5425 (11)	6235 (7)	7·2
H(3)	81 (6)	238 (12)	459 (9)	
H(41)	284 (6)	−27 (12)	465 (9)	
H(42)	151 (7)	−94 (12)	465 (10)	
H(51)	66 (6)	477 (12)	27 (9)	
H(52)	38 (6)	549 (12)	232 (9)	
H(53)	−5 (6)	319 (11)	160 (8)	
H(71)	450 (6)	705 (12)	340 (8)	
H(72)	456 (6)	687 (12)	121 (9)	
H(73)	557 (7)	567 (12)	252 (9)	
H(91)	367 (6)	640 (11)	697 (8)	
H(92)	331 (6)	479 (12)	857 (9)	
H(93)	266 (6)	705 (12)	839 (10)	

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·541 (8)	C(4)—O(1)	1·429 (8)
C(1)—O(1)	1·320 (9)	C(6)—C(7)	1·491 (10)
C(1)—O(2)	1·203 (7)	C(6)—O(3)	1·350 (5)
C(2)—C(3)	1·532 (6)	C(6)—O(4)	1·193 (10)
C(2)—C(5)	1·511 (6)	C(8)—C(9)	1·474 (7)
C(2)—O(3)	1·440 (7)	C(8)—O(5)	1·349 (8)
C(3)—C(4)	1·534 (10)	C(8)—O(6)	1·193 (6)
C(3)—O(5)	1·421 (6)		
O(1)—C(1)—O(2)	123·2 (6)	C(4)—C(3)—O(5)	110·2 (4)
C(2)—C(1)—O(2)	126·6 (5)	C(3)—C(4)—O(1)	107·3 (5)
C(2)—C(1)—O(1)	110·1 (5)	O(3)—C(6)—O(4)	122·2 (5)
C(1)—C(2)—O(3)	111·8 (4)	C(7)—C(6)—O(4)	127·2 (6)
C(1)—C(2)—C(5)	108·6 (4)	C(7)—C(6)—O(3)	110·5 (5)
C(1)—C(2)—C(3)	104·4 (4)	O(5)—C(8)—O(6)	121·9 (5)
C(5)—C(2)—O(3)	105·1 (4)	C(9)—C(8)—O(6)	125·4 (6)
C(3)—C(2)—O(3)	112·9 (4)	C(9)—C(8)—O(5)	112·7 (5)
C(3)—C(2)—C(5)	114·1 (4)	C(1)—O(1)—C(4)	112·3 (5)
C(2)—C(3)—O(5)	113·1 (4)	C(2)—O(3)—C(6)	118·1 (4)
C(2)—C(3)—C(4)	103·8 (4)	C(3)—O(5)—C(8)	117·4 (4)

Table 3. *Least-squares planes through the lactone ring*

(*a*) Atomic deviations (Å ×10^3)

	Plane (1)	Plane (2)
O(1)	55 (4)*	15 (4)*
C(1)	−41 (5)*	−29 (5)*
C(2)	−3 (4)*	16 (4)*
C(3)	29 (4)*	−11 (4)*
C(4)	−122 (6)*	−197 (6)
O(2)	−71 (5)	−23 (5)
O(3)	−1129 (3)	−1079 (3)
O(5)	−1024 (3)	−1074 (3)
C(5)	1211 (4)	1262 (4)

(*b*) Equations of the planes

Plane (1): $-0{\cdot}9781x - 0{\cdot}1393y - 0{\cdot}1545z = -2{\cdot}3846$

Plane (2): $-0{\cdot}9765x - 0{\cdot}1116y - 0{\cdot}1842z = -2{\cdot}3925$

* Atoms included in the least-squares-plane calculation.

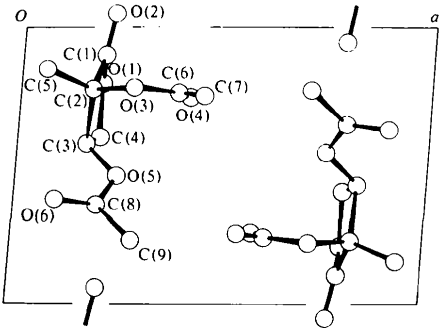

Fig. 1. The structure as viewed down the *b* axis.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35190 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Torsion angles* (°)

O(1)–C(1)–C(2)–C(3)	4·1 (6)	C(5)–C(2)–C(1)–O(1)	117·9 (5)	C(2)–O(3)–C(6)–O(4)	−2·4 (7)
C(1)–C(2)–C(3)–C(4)	4·9 (5)	C(5)–C(2)–C(3)–C(4)	123·3 (5)	O(3)–C(2)–C(3)–O(5)	2·7 (6)
C(2)–C(3)–C(4)–O(1)	−11·9 (6)	C(5)–C(2)–C(3)–O(5)	−117·3 (5)	C(1)–C(2)–C(3)–O(5)	124·4 (4)
C(3)–C(4)–O(1)–C(1)	15·7 (7)	C(5)–C(2)–O)3)–O(6)	−165·4 (4)	O(1)–C(4)–C(3)–O(5)	133·3 (5)
C(4)–O(1)–C(1)–C(2)	12·5 (7)	C(1)–C(2)–O(3)–C(6)	−47·7 (6)	C(2)–C(3)–O(5)–C(8)	93·4 (5)
O(2)–C(1)–O(1)–C(4)	171·6 (6)	C(3)–C(2)–O(3)–C(6)	69·6 (5)	C(4)–C(3)–O(5)–C(8)	−150·9 (5)
O(2)–C(1)–C(2)–O(3)	−57·7 (7)	O(1)–C(1)–C(2)–O(3)	126·5 (5)	C(3)–O(5)–C(8)–O(6)	−2·3 (7)
O(2)–C(1)–C(2)–C(3)	179·9 (5)	C(4)–C(3)–C(2)–O(3)	116·7 (5)	C(3)–O(5)–C(8)–C(9)	175·8 (4)
O(2)–C(1)–C(2)–C(5)	57·8 (7)	C(2)–O(3)–C(6)–C(7)	177·3 (4)		

agreement with the PMR spectrum interpretation. The *cis* configuration is consistent with the generally accepted criterion that the *trans* isomers are less stable than the *cis*.

The packing of the molecules in the crystal is due solely to van der Waals interactions. Intermolecular contacts do not show evidence for hydrogen bonds or distances significantly shorter than the sum of the van der Waals radii.

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References

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

DETITTA, G. T. & FORTIER, S. (1979). *Acta Cryst.* B**35**, 1903–1905.

FORTIER, S. & DETITTA, G. T. (1979). *Acta Cryst.* B**35**, 1742–1744.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN* 77. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MONTERO DE ESPINOSA, V. L. (1978). PhD Thesis, Univ. of Seville.

SHUMACHE, J. N., GREEN, C. R., BEST, F. W. & NEWELL, M. P. (1977). *J. Agric. Food Chem.* **25**, 310.

SIKIRICA, M., VICKOVIĆ, J. & VITERBO, D. (1979). *Acta Cryst.* B**35**, 1735–1737.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

USHER, J. J. & ENGLISH, R. B. (1978). *Acta Cryst.* B**34**, 2012–2014.

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Chlorhydrate de Chloro-4 Acétophénone-*O*-(*tert*-butylamino-3 hydroxy-2 propyl)oxime (IPS 345)

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Abstract. $C_{15}H_{24}ClN_2O_2^+.Cl^-$, monoclinic, $P2_1/n$, $Z = 4$, $a = 15{\cdot}051$ (2), $b = 9{\cdot}020$ (1), $c = 15{\cdot}305$ (2) Å, $\beta = 115{\cdot}52$ (1)°, $V = 1869{\cdot}74$ Å^3. The structure was solved by direct methods from three-dimensional X-ray data and refined by a least-squares procedure to $R = 0{\cdot}052$ for 3669 structure amplitudes. Hydrogen bonds and van der Waals contacts are responsible for crystalline cohesion.

Introduction. L'IPS 345 appartient à une nouvelle série de drogues à activité β bloquante (Imbs, Miesch, Schwartz, Velly, Leclerc, Mann & Wermuth, 1977;

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