translatée d'une unité paramétrique suivant les trois axes de la maille cristalline. Il n'existe pas d'autres distances courtes entre molécules, les contacts atomiques étant du type van der Waals.

Des études sont en cours actuellement sur d'autres composés d'activité thérapeutique comparable. Elles devraient permettre de proposer une description des caractéristiques communes liées à la stéréochimie du récepteur.

Nous tenons à exprimer notre gratitude aux Laboratoires Delagrange qui ont mis à notre disposition les échantillons de sulpiride nécessaires à cette étude.

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8-Hydroxy-8-methoxy-19-methoxycarbonyl-(13 → 17)-pentanorlabd-9(11)-eno-8,12-lactone

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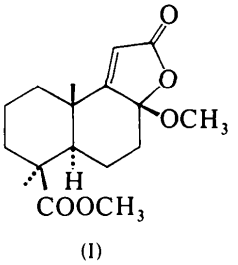
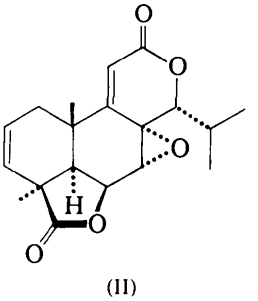
Abstract. $C_{17}H_{24}O_5$, M_r = 308·37, orthorhombic, $P2_12_12_1$, a = 23·364 (8), b = 9·569 (3), c = 7·380 (2) Å, U = 1649·9 Å^3, Z = 4, D_x = 1·241 Mg m^{-3}, μ(Cu $K\alpha$) = 0·71 mm^{-1}. R = 0·056 for 1082 unique diffractometer data. The two fused C_6 rings adopt chair conformations, with axial methoxy and acetyl groups.

Introduction. The title compound (I) was prepared by Jennings-White (1978) as a potential intermediate in the synthesis of podolide (II; Kupchan, Baxter, Ziegler, Smith & Bryan, 1975), an antileukaemic norditerpene dilactone, from podocarpic acid (III).



(I)



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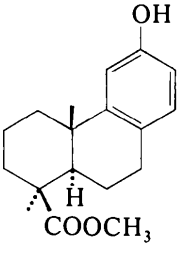

(III)

Intensities of 1123 reflexions ($5 \le 2\theta \le 140°$) were measured for a crystal 0·16 × 0·13 × 0·13 mm (grown in pentane) with a Philips PW1100 diffractometer and graphite-monochromated Cu $K\alpha$ radiation. Equivalents were merged to give 1082 unique data with $|F| > 3\sigma(F)$ which were employed for structure determination and refinement. No corrections were applied for absorption. Cell dimensions were obtained by least squares from the diffractometer angles of 25 strong reflexions. The structure was solved by multisolution tangent refinement after separate renormalization of the parity groups $h + k = 2n$ and $h + k = 2n + 1$. Since a relatively small number of data could be measured, only O and other peripheral atoms were refined anisotropically. H atoms were refined according to a riding model with idealized geometry [C–H 1·08 Å;

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Table 1. *Atom coordinates* ($\times 10^4$) *and isotropic temperature factors* ($Å^2 \times 10^3$)

	x	*y*	*z*	*U*
C(1)	1009 (3)	−631 (7)	3190 (10)	54 (2)
C(2)	1537 (3)	361 (8)	3334 (11)	68 (2)
C(3)	1900 (3)	185 (7)	4986 (11)	65 (2)
C(4)	2099 (3)	−1335 (6)	5203 (10)	56 (2)
C(5)	1592 (2)	−2363 (6)	5210 (9)	42 (1)
C(6)	1239 (3)	−2158 (6)	3452 (9)	48 (2)
C(7)	793 (3)	−3332 (7)	3195 (11)	59 (2)
C(8)	1090 (3)	−4770 (7)	3082 (10)	57 (2)
C(9)	1413 (2)	−5053 (6)	4754 (9)	50 (1)
C(10)	1815 (2)	−3842 (6)	5208 (9)	45 (1)
C(11)	2343 (2)	−4329 (6)	5351 (9)	48 (1)
C(12)	2348 (2)	−5847 (6)	4995 (9)	54 (2)
C(13)	1236 (3)	−5784 (9)	7832 (12)	79 (3)*
C(14)	1238 (3)	−2180 (8)	6971 (9)	54 (3)*
C(15)	533 (2)	−254 (7)	4540 (10)	53 (3)*
C(16)	171 (3)	1375 (9)	6605 (12)	78 (3)*
C(17)	729 (4)	−447 (10)	1338 (10)	82 (4)*
O(1)	1797 (2)	−6237 (4)	4561 (7)	59 (2)*
O(2)	2730 (2)	−6678 (4)	5024 (8)	69 (2)*
O(3)	1009 (2)	−5369 (5)	6118 (7)	63 (2)*
O(4)	619 (2)	937 (5)	5398 (8)	68 (2)*
O(5)	102 (2)	−957 (5)	4751 (8)	71 (2)*

* U_{eq} = one third of the trace of the orthogonalized U_{ij} tensor.

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

C(2)–C(1)	1·560 (11)	C(3)–C(2)	1·494 (11)
C(4)–C(3)	1·536 (10)	C(5)–C(4)	1·540 (9)
C(6)–C(5)	1·550 (10)	C(6)–C(1)	1·569 (11)
C(7)–C(6)	1·544 (10)	C(8)–C(7)	1·544 (11)
C(9)–C(8)	1·471 (10)	C(10)–C(9)	1·529 (10)
C(10)–C(5)	1·508 (10)	C(11)–C(10)	1·323 (8)
C(12)–C(11)	1·476 (9)	C(15)–C(1)	1·536 (11)
C(17)–C(1)	1·525 (12)	C(14)–C(5)	1·550 (11)
O(1)–C(9)	1·452 (8)	O(1)–C(12)	1·377 (8)
O(2)–C(12)	1·196 (7)	O(3)–C(9)	1·413 (8)
O(3)–C(13)	1·428 (10)	O(4)–C(15)	1·319 (9)
O(4)–C(16)	1·437 (9)	O(5)–C(15)	1·220 (8)
C(6)–C(1)–C(2)	106·7 (6)	C(15)–C(1)–C(2)	112·7 (7)
C(15)–C(1)–C(6)	112·8 (6)	C(17)–C(1)–C(2)	109·2 (7)
C(17)–C(1)–C(6)	111·4 (7)	C(17)–C(1)–C(15)	104·1 (6)
C(3)–C(2)–C(1)	115·8 (7)	C(4)–C(3)–C(2)	111·3 (7)
C(5)–C(4)–C(3)	111·9 (6)	C(6)–C(5)–C(4)	109·0 (6)
C(10)–C(5)–C(4)	109·4 (5)	C(10)–C(5)–C(6)	107·6 (6)
C(14)–C(5)–C(4)	109·9 (6)	C(14)–C(5)–C(6)	113·8 (5)
C(14)–C(5)–C(10)	107·0 (6)	C(5)–C(6)–C(1)	113·8 (6)
C(7)–C(6)–C(1)	115·6 (6)	C(7)–C(6)–C(5)	111·7 (6)
C(8)–C(7) C(6)	110·5 (6)	C(9)–C(8)–C(7)	110·5 (7)
C(10)–C(9)–C(8)	111·1 (6)	O(1)–C(9)–C(8)	112·2 (6)
O(1)–C(9)–C(10)	103·5 (5)	O(3)–C(9)–C(8)	107·1 (6)
O(3)–C(9)–C(10)	114·6 (6)	O(3)–C(9)–O(1)	108·4 (6)
C(9)–C(10)–C(5)	119·9 (6)	C(11)–C(10)–C(5)	130·8 (5)
C(11)–C(10)–C(9)	108·8 (6)	C(12)–C(11)–C(10)	109·8 (6)
O(1)–C(12)–C(11)	107·6 (6)	O(2)–C(12)–C(11)	131·0 (5)
O(2)–C(12)–O(1)	121·4 (6)	O(4)–C(15)–C(1)	113·8 (6)
O(5)–C(15)–C(1)	123·4 (7)	O(5)–C(15)–O(4)	122·8 (7)
C(12)–O(1)–C(9)	110·0 (5)	C(13)–O(3)–C(9)	116·3 (6)
C(16)–O(4)–C(15)	116·0 (6)		

common U(H) 0·085 (7) Å^2], except for the methyl H atoms which were refined as rigid CH_3 groups [common U(H) 0·108 (8) Å^2]. The chemically probable absolute configuration was assumed. The structure was refined to $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0{\cdot}066$ and a corresponding unweighted $R = 0{\cdot}056$, where $w = [\sigma^2(F) + 0{\cdot}003F_o^2]^{-1}$. The results are presented in Tables 1 and 2.*

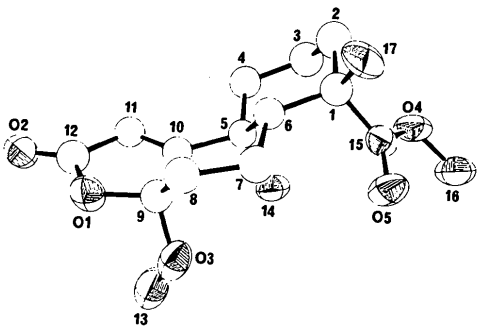

Fig. 1. *ORTEP* plot of (I), showing atom numbering and 50% thermal ellipsoids for the anisotropic atoms. H atoms have been omitted for clarity.

Discussion. Fig. 1 shows that the two fused six-membered rings, which are saturated except for C(10), adopt chair conformations. The acetyl, methoxy and one methyl group [C(14)] are axial, whereas the other methyl [C(17)] is equatorial. The lactone ring is attached to the sp^2 ring C(10) and equatorially to C(9), and is thus almost exactly planar; the maximum deviation of the six atoms from their least-squares plane is 0·04 (2) Å. The acetate group is orientated to minimize transannular repulsions between its O atoms and the methyl group C(14) [C(14)···O(5) 3·33 (2), C(14)···O(4) 3·51 (2) Å], which accounts for its relatively rigid conformation; usually such a substituent would have appreciably larger thermal parameters than the fused ring system. There are no particularly short intermolecular interactions.

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* Tables of structure factors, anisotropic temperature factors and hydrogen atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35864 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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