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## (9*S*,11*S*)-9-Acetoxydihydrocostunolide (Herbolide A)

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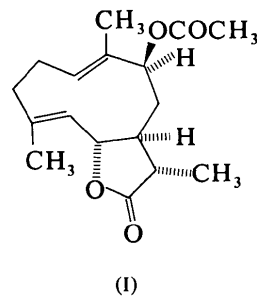
**Abstract.** C<sub>17</sub>H<sub>24</sub>O<sub>4</sub>, *M<sub>r</sub>* = 292.38, monoclinic, *P*2<sub>1</sub>, *a* = 11.279 (4), *b* = 7.703 (2), *c* = 9.499 (3) Å, β = 93.61 (2)°, *U* = 823.7 Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.179 Mg m<sup>-3</sup>, μ(Mo *K*α) = 0.05 mm<sup>-1</sup>. The structure was refined to *R* = 0.059 for 1468 unique reflexions; it provides confirmation of the structure deduced by chemical and spectroscopic methods.

**Introduction.** The title compound (I; trivial name herbolide A) is one of three sesquiterpene lactones isolated from the composite plant *Artemisia herba alba* (Segal, Sokoloff, Haran, Zaitschek & Lichtenberg, 1977). We have already reported the structure of herbolide B (Hull & Kennard, 1978). The crystal structure determination of (I) was undertaken (*a*) to confirm the structure earlier deduced from chemical and spectroscopic results (Segal *et al.*, 1977), in particular the position of the two double bonds; and (*b*) to allow calculation of the sign of the circular-dichroism couplet from exciton theory.

Large colourless crystals in the form of flat blocks were obtained from ethanol. Intensities were measured on a Syntex *P*2<sub>1</sub> diffractometer with monochromated Mo *K*α radiation and a crystal 0.75 × 0.75 × 0.25 mm. Cell dimensions were obtained by least-squares methods from 15 strong reflexions. Systematic absences 0*kl*0, *k* odd, indicated space group *P*2<sub>1</sub>. 2138

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reflexions were measured in the range 3° < 2θ < 55°; after application of *L<sub>p</sub>* corrections, averaging equivalent reflexions gave 1468 unique reflexions with *F* > 4σ(*F*).

The structure was solved by multiresolution tangent refinement with *SHELX*. A starting set of seven reflexions (chosen from a convergence map) gave 2<sup>7</sup> phase permutations. The best *E* map showed all non-hydrogen atoms. Least-squares refinement proceeded to *R* = 0.14 (isotropic), 0.09 (anisotropic); a difference synthesis then revealed all non-methyl H atoms. In the final stages of refinement C–H distances and H–C–H angles were fixed at 1.08 Å and 109.5°, and common isotropic temperature factors were assigned to methyl and non-methyl H atoms. High H-atom temperature factors and slow convergence of the methyl groups probably indicate some rotational disorder, as would be expected for methyl groups attached to *sp*<sup>2</sup> C atoms. The final *R*' = ∑ *w*<sup>1/2</sup> Δ / ∑ *w*<sup>1/2</sup> |*F*<sub>o</sub>| was 0.063, with a corresponding *R* of 0.059; the weighting scheme was

$w = 1/[\sigma^2(F) + 0.0006F^2]$ , which gave mean values of  $w\Delta^2$  varying only slightly with  $\sin \theta$  or  $|F_o|$ . A final difference map showed no peaks  $>0.24 e \text{ \AA}^{-3}$ . Final atomic coordinates are given in Table 1, bond lengths and angles and torsion angles in Tables 2 and 3. Diagrams are given in Figs. 1 and 2.\*

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34225 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atom coordinates ( $\times 10^4$ )

Overall isotropic temperature factors for H atoms: non-methyl H, 0.071 (3); methyl H, 0.27 (1)  $\text{\AA}^2$ .

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	185 (3)	5013 (5)	8303 (4)
C(2)	-850 (4)	3858 (6)	8595 (5)
C(3)	-1197 (4)	2715 (6)	7266 (4)
C(4)	-105 (3)	1855 (5)	6793 (4)
C(5)	559 (3)	2686 (5)	5900 (4)
C(6)	1835 (3)	2394 (5)	5680 (4)
C(7)	2583 (3)	4055 (5)	5706 (3)
C(8)	3092 (3)	4720 (6)	7145 (4)
C(9)	2258 (3)	5797 (5)	8015 (4)
C(10)	1324 (3)	4755 (5)	8713 (4)
C(11)	3585 (3)	3544 (6)	4773 (4)
C(12)	2994 (5)	2261 (6)	3758 (5)
C(13)	4179 (4)	5042 (10)	4036 (6)
C(14)	249 (4)	174 (6)	7511 (4)
C(15)	1771 (4)	3388 (7)	9753 (4)
C(16)	3474 (3)	8166 (6)	8898 (5)
C(17)	4256 (4)	8763 (8)	10158 (5)
O(1)	3336 (3)	1778 (6)	2654 (3)
O(2)	1966 (3)	1695 (4)	4250 (3)
O(3)	3007 (2)	6602 (4)	9156 (3)
O(4)	3276 (3)	8955 (5)	7817 (4)
H(11)	-17	6172	7695
H(21)	-604	3024	9480
H(22)	-1599	4650	8842
H(31)	-1582	3525	6429
H(32)	-1834	1740	7534
H(51)	120	3686	5263
H(61)	2136	1540	6530
H(71)	2034	5143	5362
H(81)	3855	5516	6961
H(82)	3370	3603	7769
H(91)	1795	6694	7301
H(111)	4334	3018	5398
H(131)	4585	5914	4815
H(132)	3532	5740	3369
H(133)	4855	4516	3400
H(141)	-430	-251	8186
H(142)	1065	404	8139
H(143)	393	-816	6735
H(151)	2718	3547	9958
H(152)	1583	2075	9407
H(153)	1327	3644	10706
H(171)	4656	10004	9951
H(172)	4944	7797	10331
H(173)	3754	8862	11086

**Discussion.** The structure confirms that deduced earlier (Segal *et al.*, 1977). However, formal application of simple exciton theory leads to the wrong sign of the circular-dichroism couplet, as in some other cases with related compounds (G. Snatzke, private communication). The coordinates quoted are for the naturally occurring enantiomer. The absolute configuration was not determined in this study.

Although bond lengths are normal, many bond angles and torsion angles differ appreciably from ideal values; *e.g.* C(4)–C(5)–C(6) 127.3, C(2)–C(1)–

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

C(1)–C(2)	1.508 (7)	C(1)–C(10)	1.333 (6)
C(2)–C(3)	1.569 (8)	C(3)–C(4)	1.493 (7)
C(4)–C(5)	1.331 (6)	C(4)–C(14)	1.505 (8)
C(5)–C(6)	1.484 (6)	C(6)–C(7)	1.532 (7)
C(6)–O(2)	1.477 (5)	C(7)–C(8)	1.537 (7)
C(7)–C(11)	1.530 (7)	C(8)–C(9)	1.534 (7)
C(9)–C(10)	1.510 (7)	C(9)–O(3)	1.468 (5)
C(10)–C(15)	1.509 (8)	C(12)–O(1)	1.198 (7)
C(12)–O(2)	1.350 (7)	C(11)–C(12)	1.507 (8)
C(11)–C(13)	1.526 (10)	C(16)–O(3)	1.344 (6)
C(16)–O(4)	1.201 (7)	C(16)–C(17)	1.514 (8)
C(2)–C(1)–C(10)	127.0 (5)	C(1)–C(2)–C(3)	109.9 (4)
C(2)–C(3)–C(4)	108.8 (4)	C(3)–C(4)–C(5)	119.1 (5)
C(3)–C(4)–C(14)	116.5 (4)	C(5)–C(4)–C(14)	124.0 (4)
C(4)–C(5)–C(6)	127.3 (4)	C(5)–C(6)–C(7)	114.1 (4)
C(5)–C(6)–O(2)	109.8 (4)	C(7)–C(6)–O(2)	103.4 (4)
C(6)–C(7)–C(8)	117.8 (4)	C(6)–C(7)–C(11)	101.7 (4)
C(8)–C(7)–C(11)	110.6 (4)	C(7)–C(8)–C(9)	116.9 (4)
C(8)–C(9)–C(10)	114.7 (4)	C(8)–C(9)–O(3)	106.4 (4)
C(10)–C(9)–O(3)	106.6 (4)	C(1)–C(10)–C(9)	118.6 (4)
C(1)–C(10)–C(15)	124.8 (5)	C(9)–C(10)–C(15)	116.4 (4)
C(6)–O(2)–C(12)	109.8 (4)	O(2)–C(12)–O(1)	122.2 (5)
O(2)–C(12)–C(11)	110.2 (5)	O(2)–C(12)–C(11)	110.2 (5)
O(1)–C(12)–C(11)	127.6 (6)	C(7)–C(11)–C(12)	103.1 (4)
C(7)–C(11)–C(13)	115.7 (5)	C(12)–C(11)–C(13)	113.0 (5)
C(9)–O(3)–C(16)	117.3 (4)	O(3)–C(16)–O(4)	123.7 (5)
O(3)–C(16)–C(17)	110.1 (5)	O(4)–C(16)–C(17)	126.1 (5)

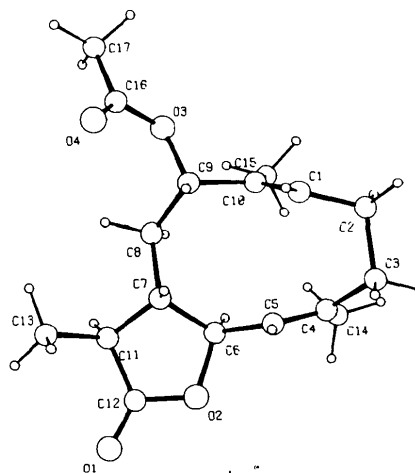
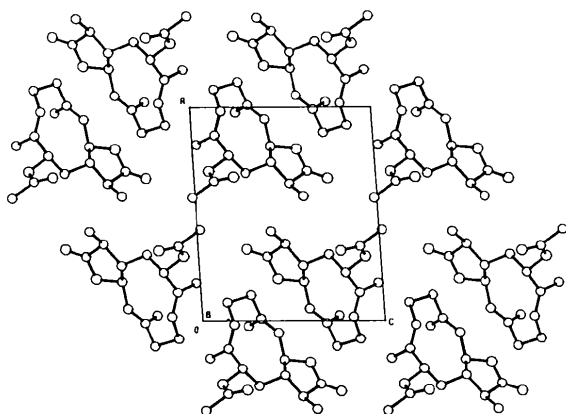


Fig. 1. The title compound, showing the labelling scheme for non-H atoms.

Table 3. *Torsion angles* (°)

Sign convention as defined by Klyne &amp; Prelog (1960).

C(10)–C(1)–C(2)–C(3)	–100.3 (6)	O(2)–C(6)–C(7)–C(11)	–33.7 (4)	O(3)–C(9)–C(10)–C(1)	127.7 (4)
C(2)–C(1)–C(10)–C(9)	164.6 (5)	C(5)–C(6)–O(2)–C(12)	147.5 (4)	O(3)–C(9)–C(10)–C(15)	–57.0 (5)
C(2)–C(1)–C(10)–C(15)	–10.3 (7)	C(7)–C(6)–O(2)–C(12)	25.3 (5)	C(8)–C(9)–O(3)–C(16)	89.7 (5)
C(1)–C(2)–C(3)–C(4)	50.5 (5)	C(6)–C(7)–C(8)–C(9)	–82.2 (5)	C(10)–C(9)–O(3)–C(16)	–147.4 (4)
C(2)–C(3)–C(4)–C(5)	–87.5 (5)	C(11)–C(7)–C(8)–C(9)	161.5 (4)	C(6)–O(2)–C(12)–O(1)	175.1 (4)
C(2)–C(3)–C(4)–C(14)	85.2 (5)	C(6)–C(7)–C(11)–C(12)	30.2 (5)	C(6)–O(2)–C(12)–C(11)	–5.7 (6)
C(3)–C(4)–C(5)–C(6)	157.2 (5)	C(6)–C(7)–C(11)–C(13)	154.0 (4)	O(2)–C(12)–C(11)–C(7)	–16.3 (6)
C(14)–C(4)–C(5)–C(6)	–14.9 (7)	C(8)–C(7)–C(11)–C(12)	156.1 (4)	O(2)–C(12)–C(11)–C(13)	–141.9 (5)
C(4)–C(5)–C(6)–C(7)	–130.9 (5)	C(8)–C(7)–C(11)–C(13)	–80.0 (5)	O(1)–C(12)–C(11)–C(7)	162.8 (6)
C(4)–C(5)–C(6)–O(2)	113.5 (5)	C(7)–C(8)–C(9)–C(10)	75.2 (5)	O(1)–C(12)–C(11)–C(13)	37.3 (8)
C(5)–C(6)–C(7)–C(8)	86.0 (5)	C(7)–C(8)–C(9)–O(3)	–167.3 (4)	C(9)–O(3)–C(16)–O(4)	2.9 (7)
C(5)–C(6)–C(7)–C(11)	–152.9 (4)	C(8)–C(9)–C(10)–C(1)	–114.8 (5)	C(9)–O(3)–C(16)–C(17)	–177.6 (4)
O(2)–C(6)–C(7)–C(8)	–154.7 (4)	C(8)–C(9)–C(10)–C(15)	60.5 (5)		

Fig. 2. Packing diagram projected down *b*; H atoms are omitted for clarity.

C(10) 127.0, C(6)–C(7)–C(8) 117.8, C(3)–C(4)–C(5)–C(6) 157.2°. This is probably due to ring strain, there being two *trans* double bonds in the ten-membered ring. Similar angles are observed in her-

bolide B (Hull & Kennard, 1978), in which there is a C(1)–C(10) epoxide ring.

There are no non-bonded contacts between non-H atoms <3.4 Å.

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### Structure of 3-(7-Adeniny)propionic Acid Lactam

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**Abstract.** C<sub>8</sub>H<sub>7</sub>N<sub>5</sub>O, FW = 189, orthorhombic, *Pbca*, *a* = 14.006 (2), *b* = 6.751 (1), *c* = 16.442 (2) Å, *D<sub>m</sub>* (floatation) = 1.61<sub>6</sub>, *D<sub>x</sub>* = 1.616 Mg m<sup>–3</sup>, *Z* = 8, *R* = 0.053 for 1499 reflexions (Mo *K*α). As compared with the adenine derivatives alkylated at N(9), the N(7)–C(8) bond is lengthened, the C(8)–N(9) bond is

shortened, the C(5)–N(7)–C(8) and C(5)–C(4)–N(9) angles are expanded, and the C(4)–N(9)–C(8) and C(4)–C(5)–N(7) angles are contracted.

**Introduction.** In the preparation of 3-(9-adeniny)propionic acid (I), we have found that the reaction

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