dihedral angle between these two rings is 1·5 (1)°. The dimensions of the phthalimido group are consistent with those in other phthalimido derivatives (Iwasaki & Mazuko, 1986; Bats & Teuber, 1985; Voliotis, Arrieta & Germain, 1984).

A view of the molecular packing is given in Fig. 2.

The authors are indebted to Professor A. Deljac for supplying the crystals and for helpful discussion.

References

BATS, W. J. & TEUBER, H. J. (1985). *Acta Cryst.* C**41**, 298–300.
BELLETI, D., UGOZZOLI, F., CANTONI, A. & PASQUINELLI, G. (1979). *Gestione on line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo* 220. Internal Report 1.3/79. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.
BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1981). *Acta Cryst.* B**37**, 2106–2108.
BOCELLI, G. & GRENIER-LOUSTALOT, M. F. (1982). *J. Mol. Struct.* **81**, 113–119; 295–299.
BRUVO, M., SIKIRICA, M. & VICKOVIĆ, I. (1981). *Acta Cryst.* B**37**, 700–701.
CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
ELIEL, E. L. (1962). *Stereochemistry of Carbon Compounds*. New York: McGraw-Hill.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
HARLOW, R. L. & SIMONSEN, S. H. (1976). *Acta Cryst.* B**32**, 2137–2144.
IWASAKI, F. & MAZUKO, Y. (1986). *Acta Cryst.* C**42**, 124–127.
LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–589.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SIKIRICA, M., VICKOVIĆ, I. & VITERBO, D. (1979). Proc. III Italian–Yugoslav Crystallogr. Congr. Parma, Italy, June 1979, 124–127.
VOLIOTIS, S., ARRIETA, J. M. & GERMAIN, G. (1984). *Acta Cryst.* C**40**, 1946–1948.

Acta Cryst. (1988). C**44**, 161–163

Structure of *O*-Methylbaccharocephol

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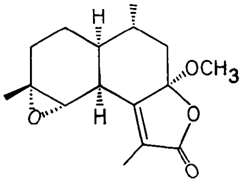
Abstract. $C_{16}H_{22}O_4$, $M_r = 278{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}078\,(7)$, $b = 10{\cdot}476\,(9)$, $c = 17{\cdot}639\,(7)$ Å, $V = 1493$ Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}8$ cm^{-1}, $F(000) = 600$, $T = 293$ K, final $R = 0{\cdot}057$ for 883 observed reflections. The title compound, isolated from *Baccharis sphaerocephala*, corresponds to a new amorphane sesquiterpene γ-lactol derivative, a very rare type of carbon skeleton, with two *cis*-fused six-membered rings, a five-membered-ring γ-lactone and an epoxide.

Introduction. *O*-Methylbaccharocephol was isolated from *B. sphaerocephala* from southern Chile. Its spectroscopic data pointed to a sesquiterpene lactone without a hydrogen at the lactone-ring closure. A tertiary methoxyl group, an epoxide, and secondary, tertiary and vinylic methyl groups were also shown to be present (Rivera, González, Castillo & Rodríguez, 1987).

The structure (1) (without stereochemical assignments) was tentatively arrived at among other less likely alternatives. This X-ray crystallographic study was undertaken to verify the structure and relative configuration of *O*-methylbaccharocephol.



(1)

Experimental. Colourless crystal, 0·6 × 0·1 × 0·1 mm. Diffraction maxima with $2\theta < 100°$ were collected on a computer-controlled four-circle Siemens AED-4 diffractometer, using graphite-monochromated Cu $K\alpha$

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radiation and ω-scan mode; cell parameters by least squares from 12 reflections with $20 < \theta < 40°$; of 904 independent reflections, 883 were treated as observed $[I > 3\sigma(I)]$ and corrected for Lorentz and polarization effects; no absorption correction; range of hkl: h 0→8, k 0→10, l 0→17. The structure was solved by direct methods (Main *et al.*, 1980). Most of the H atoms were located on a difference synthesis map and the remainder placed on calculated positions (Fayos & Martínez-Ripoll, 1980). Full-matrix least-squares refinement (Stewart, 1976) with anisotropic non-H atoms and fixed isotropic H atoms converged to a final $R = 0{\cdot}057$ for 883 reflections and 180 parameters; function minimized $\sum w(|F_o| - |F_c|)^2$ (unit weights); maximum shift to e.s.d. in final cycle $0{\cdot}08\sigma$; maximum electron density in final difference Fourier synthesis 0·24 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Refined atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$) *for non*-H *atoms, with e.s.d.'s in parentheses*

	x	y	z	U_{eq}
O(1)	5807 (7)	11173 (5)	2681 (3)	54 (2)
O(2)	1323 (6)	9596 (6)	444 (3)	55 (2)
O(3)	943 (7)	7906 (5)	862 (3)	58 (2)
O(4)	−144 (7)	6704 (6)	1802 (4)	75 (3)
C(1)	5441 (8)	9822 (7)	1279 (4)	36 (2)
C(2)	6254 (9)	8686 (7)	1664 (4)	45 (3)
C(3)	6914 (9)	9002 (8)	2445 (4)	52 (3)
C(4)	5773 (10)	9843 (8)	2908 (4)	50 (3)
C(5)	4309 (9)	10440 (7)	2561 (4)	45 (3)
C(6)	3882 (9)	10225 (6)	1745 (4)	33 (2)
C(7)	2564 (8)	9232 (7)	1632 (4)	33 (2)
C(8)	2093 (9)	8964 (8)	813 (4)	44 (3)
C(9)	3609 (9)	8588 (8)	348 (4)	53 (3)
C(10)	5015 (9)	9540 (8)	437 (4)	51 (3)
C(11)	1732 (9)	8458 (7)	2091 (4)	38 (2)
C(12)	704 (10)	7558 (9)	1606 (5)	55 (3)
C(13)	1698 (10)	8259 (8)	2935 (4)	54 (3)
C(14)	4807 (12)	10782 (10)	−17 (5)	76 (4)
C(15)	5797 (12)	9653 (10)	3770 (4)	79 (4)
C(16)	−67 (11)	10545 (9)	823 (5)	69 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(4)	1·449 (9)	C(4)—C(5)	1·471 (11)
O(1)—C(5)	1·448 (9)	C(4)—C(15)	1·533 (10)
O(2)—C(8)	1·407 (9)	C(5)—C(6)	1·496 (9)
O(2)—C(16)	1·427 (10)	C(6)—C(7)	1·501 (9)
O(3)—C(8)	1·448 (9)	C(7)—C(8)	1·520 (9)
O(3)—C(12)	1·374 (11)	C(7)—C(11)	1·327 (10)
O(4)—C(12)	1·178 (10)	C(8)—C(9)	1·524 (10)
C(1)—C(2)	1·519 (10)	C(9)—C(10)	1·519 (11)
C(1)—C(6)	1·561 (9)	C(10)—C(14)	1·537 (13)
C(1)—C(10)	1·553 (10)	C(11)—C(12)	1·520 (11)
C(2)—C(3)	1·513 (10)	C(11)—C(13)	1·503 (10)
C(3)—C(4)	1·514 (11)		
O(4)—O(1)—O(5)	61·0 (4)	C(6)—C(7)—C(11)	134·5 (6)
C(8)—O(2)—C(16)	116·1 (5)	C(6)—C(7)—C(8)	115·5 (5)
C(8)—O(3)—C(12)	110·5 (6)	C(8)—C(7)—C(11)	109·8 (6)
C(6)—C(1)—C(10)	112·0 (5)	O(3)—C(8)—C(7)	104·1 (5)
C(2)—C(1)—C(10)	112·0 (6)	O(2)—C(8)—C(7)	114·0 (6)
C(2)—C(1)—C(6)	109·0 (5)	O(2)—C(8)—O(3)	109·4 (5)
C(1)—C(2)—C(3)	112·7 (6)	C(7)—C(8)—C(9)	110·9 (6)
C(2)—C(3)—C(4)	113·8 (6)	O(3)—C(8)—C(9)	110·4 (6)
O(1)—C(4)—C(3)	113·5 (6)	O(2)—C(8)—C(9)	107·6 (6)
C(3)—C(4)—C(15)	116·8 (7)	C(8)—C(9)—C(10)	112·0 (6)
C(3)—C(4)—C(5)	120·7 (6)	C(1)—C(10)—C(9)	112·8 (6)
O(1)—C(4)—C(15)	113·4 (6)	C(9)—C(10)—C(14)	114·8 (6)
O(1)—C(4)—C(5)	59·4 (4)	C(1)—C(10)—C(14)	111·2 (6)
C(5)—C(4)—C(15)	118·5 (7)	C(7)—C(11)—C(13)	134·1 (6)
O(1)—C(5)—C(4)	59·5 (4)	C(7)—C(11)—C(12)	108·2 (6)
C(4)—C(5)—C(6)	121·4 (6)	C(12)—C(11)—C(13)	117·4 (6)
O(1)—C(5)—C(6)	114·4 (5)	O(4)—C(12)—C(11)	128·5 (8)
C(1)—C(6)—C(5)	111·1 (5)	O(3)—C(12)—C(11)	107·1 (6)
C(5)—C(6)—C(7)	113·2 (5)	O(3)—C(12)—O(4)	124·2 (8)
C(1)—C(6)—C(7)	108·3 (5)		

Discussion. The refined positional and isotropic thermal parameters for all non-H atoms are given in Table 1.* A perspective molecular drawing (Johnson, 1971) with the atomic labelling is displayed in Fig. 1 (the absolute configuration was not established). Bond distances and bond angles are listed in Table 2.

The molecular structure of *O*-methylbaccharocephol consists of two *cis*-fused six-membered rings, a five-membered-ring γ-lactone and an epoxide across C(4)—C(5).

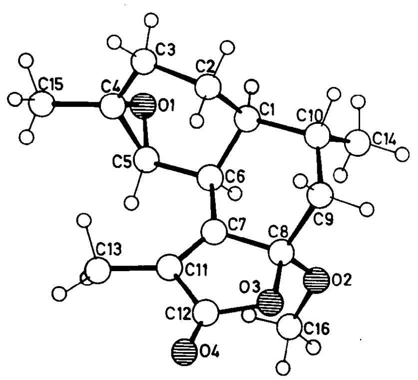

Fig. 1. A perspective drawing of the molecule of *O*-methylbaccharocephol showing the atom labelling.

The epoxide-bearing cyclohexane ring *A* is distorted: C(2), C(3), C(5) and C(6) have a maximum deviation of 0·058 (7) Å from their mean plane, whereas C(1) and C(4) deviate from this plane by 0·697 (7) and −0·054 (8) Å, respectively; O(1) is *cis* to C(1), 1·147 (5) Å out of the same plane. The C(4) methyl group is quasiaxial. The cyclohexane ring *B* is in a chair conformation with C(10) methyl and C(8) methoxyl groups, both substituents occupying axial orientations. The lactone ring is essentially planar. The torsional angles of rings *A* and *B* about the common C(1)—C(6)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44381 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

bond are 53·1 (7) and 52·6 (7)°, respectively, whereas those of rings *B* and *C* are 55·7 (8) and −2·2 (8)°.

Thus, *O*-methylbaccharocephol is a new member of a rare group of sesquiterpene *γ*-lactols with an amorphane-type of carbon skeleton (Fischer, Olivier & Fischer, 1979). The true natural product corresponds to baccharocephol (an OH group instead of OCH_3); the isolation procedure and spectroscopic data of baccharocephol, of its *O*-methyl derivative and of other diterpenoids also isolated from *B. sphaerocephala* will be published elsewhere.

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References

FAYOS, J. & MARTÍNEZ-RIPOLL, M. (1980). *HSEARCH*. Instituto Rocasolano, CSIC, Madrid, Spain.

FISCHER, N. H., OLIVIER, E. J. & FISCHER, H. D. (1979). *Fortschr. Chem. Org. Naturst.* **38**, 48–390.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

RIVERA, P., GONZÁLEZ, C., CASTILLO, M. & RODRÍGUEZ, M. L. (1987). In preparation.

STEWART, J. M. (1976). The *XRAY*76 system. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

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Structure of *cis*-4-Acetyl-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one*

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Abstract. $C_9H_{12}O_3$, $M_r = 168{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}814\ (1)$, $b = 10{\cdot}632\ (1)$, $c = 12{\cdot}183\ (1)$ Å, $V = 882{\cdot}6\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}28$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}102$ mm^{-1}, $F(000) = 360$, $T = 293$ K, $R = 0{\cdot}033$ for 657 reflections. The bicyclohexane moiety adopts a boat conformation with the five-membered lactone ring having an envelope conformation. The cyclopropane ring is inclined to the five-membered ring at an angle of 69·7 (2)°. The phase angle of pseudorotation for the five-membered ring is 38·4° and the degree of puckering is 7·5°.

Introduction. (1*R*)-*cis*-Deltamethrin molecules are used as agricultural insecticides because of their high potency, low mammalian toxicity and biodegradability (Mandal, Borude, Armugasamy, Soni, Jawalkar, Mahajan, Ratnam & Goghare, 1986). In an attempt to synthesize acid moieties of deltamethrins, the title compound was prepared and is characterized by X-ray structure analysis.

Experimental. Crystals of approximate dimensions 0·62 × 0·35 × 0·25 mm; density by flotation in KI solution; lattice parameters from 24 reflections ($23 < 2\theta < 41°$); intensity data collected on Enraf–Nonius CAD-4F-11M single-crystal X-ray diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}; $\theta < 23{\cdot}5°$; of 851 reflections collected ($h = 0$ to 7, $k = 0$ to 11 and $l = 0$ to 13), 657 were judged significant ($|F_o| \geq 3\sigma|F_o|$). Four standard reflections (440, 314, 106 and $\bar{1}5\bar{2}$) measured every 3600 s, 3% variation in intensity; no correction for absorption; structure solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Dec-

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters for non*-H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}(B_{11} + B_{22} + B_{33}).$$

	x	y	z	B_{eq}(Å^2)
O(1)	2487 (3)	1614 (2)	1776 (17)	5·53
O(2)	1272 (3)	2122 (2)	5540 (2)	5·33
C(1)	1406 (4)	−273 (2)	2718 (2)	3·59
C(2)	1694 (4)	1092 (3)	2530 (2)	4·01
O(3)	876 (3)	1771 (1)	3361 (1)	4·17
C(4)	−256 (3)	974 (2)	4081 (2)	3·35
C(5)	249 (3)	−373 (2)	3776 (2)	3·21
C(6)	2353 (4)	−780 (2)	3770 (2)	3·17
C(7)	90 (4)	1328 (2)	5277 (2)	3·70
C(8)	−1138 (4)	645 (2)	6088 (2)	4·78
C(9)	2655 (5)	−2199 (2)	3778 (2)	4·56
C(10)	3991 (4)	−83 (3)	4325 (2)	4·14

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