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Structure of a *β*-Caryophyllene Derivative

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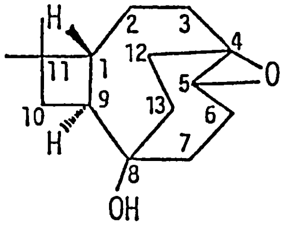
Abstract. $C_{15}H_{24}O_2$, $M_r = 236{\cdot}35$, trigonal, $P3_2$, $a = 13{\cdot}104\,(1)$, $c = 6{\cdot}756\,(1)$ Å, $V = 1004{\cdot}6\,(1)$ Å^3, $D_x = 1{\cdot}172$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 5{\cdot}982$ cm^{-1}, $F(000) = 390$, $T = 295$ K, $R = 0{\cdot}029$ for 1313 observed reflections. A novel sesquiterpenoid isolated from the oxidation of *β*-caryophyllene with lead tetraacetate is shown to be 5,5-dimethyl-12-oxatetracyclo[6.4.2.$0^{1,11}$.$0^{4,7}$]tetradecan-8-ol. The molecule consists of nine-, eight-, seven- and four-membered rings and the epoxide ring.

Experimental. Title compound (I): Crystal size $0{\cdot}35 \times 0{\cdot}30 \times 0{\cdot}25$ mm. Data were collected on the Rigaku AFC-5R four-circle diffractometer, monocromated Cu *Kα* radiation. *θ*–2*θ* scans. Cell constants refined from 2*θ* values of 25 reflections in the range 15–25°.



(I)

1418 reflections measured, $(2\theta)_{\max} = 140°$, range of indices $h = 0$ to 16, $k = -16$ to 16, $l = -8$ to 0, three check reflections monitored periodically showed no significant variation in intensity. 1330 unique reflections, of which 1313 with $F_o > 3\sigma(F_o)$ were used for all calculations. No absorption correction. The structure was solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located by difference Fourier synthesis. Final full-matrix least-squares refinement was performed with all non-H atoms having anisotropic and H atoms having isotropic thermal parameters, with minimization of $\sum w(|F_o| - |F_c|)^2$. Final convergence at $R = 0{\cdot}029$, unit weights, $wR = 0{\cdot}029$, $S = 0{\cdot}33$ for 250 parameters, $(\Delta/\sigma)_{\max} = 0{\cdot}02$. Final $(\Delta\rho)_{\max} = 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Final atomic parameters are given in Table 1, bond distances and angles in Table 2.† Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	0·6850 (2)	1·0259 (2)	0·86184	38 (1)
C(2)	0·7033 (3)	1·0925 (3)	1·0556 (7)	53 (1)
C(3)	0·6454 (3)	1·1697 (3)	1·0582 (7)	53 (1)
C(4)	0·5276 (2)	1·1099 (2)	0·9523 (6)	40 (1)
C(5)	0·5273 (2)	1·1364 (2)	0·7428 (6)	40 (1)
C(6)	0·4385 (3)	1·0471 (2)	0·6032 (6)	41 (1)
C(7)	0·4625 (2)	0·9450 (2)	0·5690 (6)	36 (1)
C(8)	0·4557 (2)	0·8740 (2)	0·7559 (6)	28·9 (9)
C(9)	0·5759 (2)	0·9006 (2)	0·8357 (6)	31 (1)
C(10)	0·6557 (3)	0·8709 (2)	0·7074 (7)	46 (1)
C(11)	0·7650 (2)	0·9737 (2)	0·8066 (7)	50 (1)
C(12)	0·4347 (2)	0·9951 (3)	1·0439 (6)	46 (1)
C(13)	0·3785 (2)	0·8827 (2)	0·9178 (6)	35 (1)
C(14)	0·8070 (3)	0·9330 (4)	0·9832 (9)	71 (2)
C(15)	0·8681 (3)	1·0515 (4)	0·673 (1)	78 (2)
O(16)	0·3963 (2)	0·7524 (1)	0·6879 (5)	36·9 (7)
O(17)	0·4867 (2)	1·1893 (2)	0·8879 (6)	54 (1)

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† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52652 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)—C(2)	1·525 (5)	C(1)—C(9)	1·558 (3)
C(1)—C(11)	1·556 (5)	C(2)—C(3)	1·538 (6)
C(3)—C(4)	1·516 (5)	C(4)—C(5)	1·458 (6)
C(4)—C(12)	1·515 (4)	C(4)—O(17)	1·456 (5)
C(5)—C(6)	1·500 (5)	C(5)—O(17)	1·446 (5)
C(6)—C(7)	1·536 (5)	C(7)—C(8)	1·545 (5)
C(8)—C(9)	1·531 (4)	C(8)—C(13)	1·533 (5)
C(8)—O(16)	1·454 (3)	C(9)—C(10)	1·551 (5)
C(10)—C(11)	1·544 (4)	C(11)—C(14)	1·518 (7)
C(11)—C(15)	1·518 (6)	C(12)—C(13)	1·534 (5)
C(2)—C(1)—C(9)	120·0 (2)	C(2)—C(1)—C(11)	120·7 (3)
C(9)—C(1)—C(11)	88·5 (2)	C(1)—C(2)—C(3)	113·4 (3)
C(2)—C(3)—C(4)	111·4 (3)	C(3)—C(4)—C(5)	117·3 (3)
C(3)—C(4)—C(12)	115·2 (3)	C(3)—C(4)—O(17)	114·5 (2)
C(5)—C(4)—C(12)	122·4 (3)	C(5)—C(4)—O(17)	59·5 (3)
C(12)—C(4)—O(17)	115·5 (3)	C(4)—C(5)—C(6)	121·2 (2)
C(4)—C(5)—O(17)	60·2 (3)	C(6)—C(5)—O(17)	118·2 (3)
C(5)—C(6)—C(7)	111·0 (3)	C(6)—C(7)—C(8)	115·3 (3)
C(7)—C(8)—C(9)	114·1 (2)	C(7)—C(8)—C(13)	112·3 (3)
C(7)—C(8)—O(16)	103·9 (3)	C(9)—C(8)—C(13)	112·0 (3)
C(9)—C(8)—O(16)	106·7 (2)	C(13)—C(8)—O(16)	107·2 (2)
C(1)—C(9)—C(8)	125·3 (2)	C(1)—C(9)—C(10)	87·1 (2)
C(8)—C(9)—C(10)	119·5 (3)	C(9)—C(10)—C(11)	89·2 (3)
C(1)—C(11)—C(10)	87·3 (2)	C(1)—C(11)—C(14)	114·1 (4)
C(1)—C(11)—C(15)	114·6 (3)	C(10)—C(11)—C(14)	112·0 (3)
C(10)—C(11)—C(15)	117·0 (4)	C(14)—C(11)—C(15)	110·3 (3)
C(4)—C(12)—C(13)	119·2 (3)	C(8)—C(13)—C(12)	116·7 (2)
C(4)—O)17)—C(5)	60·3 (2)		

Related literature. The title compound was prepared in connection with our studies on the spectroscopic irradiations of the β-caryophyllene (Damodaran & Sukh Dev, 1968) derivatives. Its chemical structure was formulated from spectroscopic evidence (Matsubara, Uchida & Ono, 1985).

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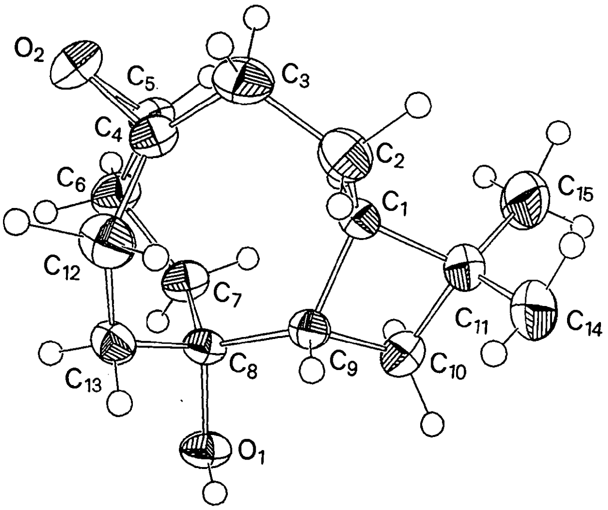

Fig. 1. View of the title compound showing the atomic numbering scheme.

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Structure of (3*SR*,4*SR*,S*SR*)-Methyl 4-(*tert*-Butylsulfinyl)-3-methyl-5-phenylvalerate

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Abstract. $C_{17}H_{26}O_3S$, $M_r = 310{\cdot}4$, triclinic, $P\bar{1}$, $a = 9{\cdot}022$ (3), $b = 9{\cdot}088$ (3), $c = 12{\cdot}265$ (4) Å, $\alpha = 106{\cdot}49$ (2), $\beta = 106{\cdot}12$ (2), $\gamma = 100{\cdot}71$ (2)°, $V = 887{\cdot}1$ (5) Å^3, $Z = 2$, $D_x = 1{\cdot}162$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}181$ mm^{-1}, $F(000) = 336$, $T = 293$ K, $R = 0{\cdot}051$ for 2150 observed reflections. The orientation of the O(1)—S—C(2)—C(4) fragment is eclipsed [torsion angle 4·8 (2)°] whereas in studies of *tert*-butyl phenethyl sulfoxides the corresponding system is staggered (torsion angles 55, 72°) [Kodama, Nishihata, Nishio & Iitaka (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 1490–1495]. This difference is presumably the result of unacceptable steric interactions between a methyl group of the *tert*-butyl func-

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