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

C(1)–C(2)	1·353 (3)	C(1)–C(9)	1·484 (2)
C(1)–C(11)	1·480 (3)	C(2)–C(3)	1·504 (2)
C(2)–C(21)	1·468 (2)	C(3)–O	1·216 (3)
C(3)–C(4)	1·482 (3)	C(4)–C(5)	1·377 (2)
C(4)–C(9)	1·391 (3)	C(5)–C(6)	1·385 (3)
C(6)–C(7)	1·374 (4)	C(7)–C(8)	1·399 (3)
C(8)–C(9)	1·373 (3)	C(11)–C(12)	1·387 (2)
C(11)–C(16)	1·386 (3)	C(12)–C(13)	1·384 (3)
C(13)–C(14)	1·373 (4)	C(14)–C(15)	1·367 (4)
C(15)–C(16)	1·386 (4)	C(21)–C(22)	1·389 (3)
C(21)–C(26)	1·398 (2)	C(22)–C(23)	1·379 (3)
C(23)–C(24)	1·376 (3)	C(24)–C(25)	1·379 (4)
C(25)–C(26)	1·371 (3)		

C(2)–C(1)–C(9)	110·0 (2)	C(2)–C(1)–C(11)	127·9 (1)
C(9)–C(1)–C(11)	122·1 (2)	C(1)–C(2)–C(3)	107·7 (1)
C(1)–C(2)–C(21)	129·9 (2)	C(3)–C(2)–C(21)	122·4 (2)
C(2)–C(3)–O	126·6 (2)	C(2)–C(3)–C(4)	106·2 (2)
O–C(3)–C(4)	127·2 (2)	C(3)–C(4)–C(5)	130·3 (2)
C(3)–C(4)–C(9)	107·6 (1)	C(5)–C(4)–C(9)	122·1 (2)
C(4)–C(5)–C(6)	117·5 (2)	C(5)–C(6)–C(7)	121·1 (2)
C(6)–C(7)–C(8)	121·0 (2)	C(7)–C(8)–C(9)	118·2 (2)
C(1)–C(9)–C(4)	108·4 (2)	C(1)–C(9)–C(8)	131·5 (2)
C(4)–C(9)–C(8)	120·0 (2)	C(1)–C(11)–C(12)	120·7 (2)
C(1)–C(11)–C(16)	120·2 (2)	C(12)–C(11)–C(16)	119·1 (2)
C(11)–C(12)–C(13)	120·2 (2)	C(12)–C(13)–C(14)	120·0 (2)
C(13)–C(14)–C(15)	120·4 (3)	C(14)–C(15)–C(16)	120·0 (2)
C(11)–C(16)–C(15)	120·2 (2)	C(2)–C(21)–C(22)	120·0 (2)
C(2)–C(21)–C(26)	121·9 (2)	C(22)–C(21)–C(26)	118·1 (2)
C(21)–C(22)–C(23)	121·2 (2)	C(22)–C(23)–C(24)	120·0 (2)
C(23)–C(24)–C(25)	119·5 (2)	C(24)–C(25)–C(26)	120·9 (2)
C(21)–C(26)–C(25)	120·3 (2)		

tion $F^* = F_c/[1{\cdot}0 + 0{\cdot}002gF_c^2/(\sin 2\theta)]^{0{\cdot}25}$ applied, g refined to 0·0033 (2). All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/C configurations; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters, while Table 2 gives interatomic distances and valence angles.* The atom numbering is shown in Fig. 1.

Related literature. This study is part of an investigation of factors affecting the planarity of π-electron systems (Watson, 1983). The structure of dimethyl 3-phenylindene-1,2-dicarboxylate has been reported (Hanson, 1977).

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44245 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Hanson, A. W. (1977). *Acta Cryst.* B33, 891–893.

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

Nicolet Instrument Corporation (1986). *SHELXTL* for Desktop 30 (Microeclipse), PN269-1040340, April.

Watson, W. H. (1983). In *Stereochemistry and Reactivity of Systems Containing π-Electrons.* Deerfield Beach, Florida: Verlag Chemie International.

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Structure of (5*E*)-1α-Hydroxy-10β,14-epoxy-4(15),5-germacradiene-9-one*

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Abstract. $C_{15}H_{22}O_3$, $M_r = 250{\cdot}3$, monoclinic, $P2_1/c$, $a = 9{\cdot}936\ (2)$, $b = 5{\cdot}187\ (1)$, $c = 28{\cdot}852\ (5)$ Å, $\beta = 110{\cdot}77\ (1)°$, $V = 1390{\cdot}3\ (4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}20\ (1)$, $D_x = 1{\cdot}20$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}076$ mm^{-1}, $F(000) = 544{\cdot}2$, $T = 296\ (1)$ K, $R = 0{\cdot}049$ for 1586 unique observed reflections. To our disappointment, the relative configurations of this synthesized compound differ from those of (±)-periplanone B. However, the new synthetic compound will be used for biomimetic model reactions of a number of sesquiterpenes. The torsion angle of the C=C–C=C moiety is −53·2 (8)°. There is an intermolecular hydrogen bond, O–H···O, with O···O distance 2·836 (3) Å.

Experimental. The title compound was prepared from germacrene D isolated from the leaves of *Solidago*

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* This numbering is slightly different to that used within the paper.

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altissima L. (Shizuri, Yamaguchi, Yamamura, Ishihara, Ohba, Saito, Niwa, Terada & Miyasaki, 1987). Colorless needle-like crystals grown from a mixed solvent of *n*-hexane and ethyl acetate. D_m measured by flotation in KI aqueous solution. Crystal size 0·35 × 0·4 × 0·5 mm, Rigaku AFC-5 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$, Laue group $2/m$, cell parameters from least-squares refinement of 20 reflections ($20 < 2\theta < 30°$). Intensity measurement performed to $2\theta = 50°$ (h 0→11, k 0→6, l −34→34), θ–2θ scan with speed 6° min^{-1} in θ. Five standard reflections showed no significant decay, $0{\cdot}981 < |F_o|/|F_o|_{\rm initial} < 1{\cdot}012$. Space group $P2_1/c$ determined from systematic absences ($h0l$, l odd; $0k0$, k odd). Although c is nearly 30 Å and Mo $K\alpha$ radiation was used, $00l$ reflections were properly measured owing to the systematic absences. 2655 reflections measured, 59 weak reflections classified as unobserved $[|F_o| < 3\sigma(|F_o|)]$, 925 zero and 1671 observed reflections. Absorption correction ($0{\cdot}974 < A < 0{\cdot}978$). After averaging equivalent reflections 1586 unique ones used for structure determination ($R_{\rm int} = 0{\cdot}014$). Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); all positions of non-H atoms determined from E map and refined by block-diagonal least squares with anisotropic thermal parameters ($R = 0{\cdot}10$) using *UNICS*III computation program system (Sakurai & Kobayashi, 1979); all H atoms found from difference synthesis and refined with isotropic thermal parameters. $\sum w||F_o| - |F_c||^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0{\cdot}015|F_o|)^2$. Final $R = 0{\cdot}049$, $wR = 0{\cdot}065$, $S = 2{\cdot}65$.* Reflection/parameter (refined) ratio 6·3, $\Delta/\sigma < 0{\cdot}052$, $-0{\cdot}22 < \Delta\rho < 0{\cdot}57$ e Å^{-3}. Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters in Table 1. Bond lengths and bond angles in Table 2. Molecular structure in Fig. 1.

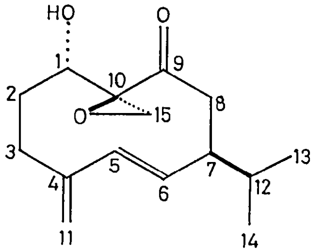

Table 1. *Positional parameters* (×10^4) *and equivalent isotropic thermal parameters* (*Hamilton*, 1959)

	x	y	z	$B_{\rm eq}$(Å^2)
O(1)	5402 (2)	2604 (4)	549 (1)	3·7
O(2)	1221 (2)	6981 (4)	392 (1)	3·8
O(3)	2759 (2)	4722 (4)	−153 (1)	3·6
C(1)	4421 (3)	3960 (6)	717 (1)	2·7
C(2)	4883 (3)	6762 (6)	856 (1)	3·1
C(3)	5909 (3)	7146 (6)	1390 (1)	4·0
C(4)	5361 (3)	6048 (7)	1774 (1)	4·0
C(5)	3790 (3)	6332 (6)	1643 (1)	3·6
C(6)	2926 (3)	4345 (6)	1601 (1)	3·5
C(7)	1338 (3)	4255 (6)	1328 (1)	3·2
C(8)	1136 (3)	3080 (6)	816 (1)	3·0
C(9)	1704 (3)	4842 (6)	505 (1)	2·7
C(10)	2891 (3)	3856 (6)	333 (1)	2·6
C(11)	6201 (4)	4783 (9)	2169 (1)	6·3
C(12)	602 (4)	2711 (8)	1628 (1)	4·6
C(13)	−944 (4)	2042 (11)	1336 (2)	7·4
C(14)	720 (5)	4207 (9)	2093 (1)	6·4
C(15)	2491 (3)	2017 (6)	−74 (1)	3·7

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

O(1)—C(1)	1·420 (4)	C(5)—C(6)	1·319 (5)
O(2)—C(9)	1·207 (3)	C(6)—C(7)	1·493 (4)
O(3)—C(10)	1·434 (4)	C(7)—C(8)	1·544 (4)
O(3)—C(15)	1·462 (4)	C(7)—C(12)	1·541 (5)
C(1)—C(2)	1·534 (4)	C(8)—C(9)	1·524 (5)
C(1)—C(10)	1·532 (3)	C(9)—C(10)	1·521 (5)
C(2)—C(3)	1·530 (4)	C(10)—C(15)	1·454 (4)
C(3)—C(4)	1·510 (5)	C(12)—C(13)	1·507 (5)
C(4)—C(5)	1·477 (5)	C(12)—C(14)	1·518 (6)
C(4)—C(11)	1·322 (5)	O(1)···O(3$^{\rm i}$)	2·836 (3)
C(10)—O(3)—C(15)	60·3 (2)	C(7)—C(8)—C(9)	112·2 (2)
O(1)—C(1)—C(2)	112·4 (2)	O(2)—C(9)—C(8)	121·0 (3)
O(1)—C(1)—C(10)	111·1 (2)	O(2)—C(9)—C(10)	120·2 (3)
C(2)—C(1)—C(10)	110·5 (2)	C(8)—C(9)—C(10)	118·9 (3)
C(1)—C(2)—C(3)	114·6 (2)	O(3)—C(10)—C(1)	114·8 (2)
C(2)—C(3)—C(4)	114·0 (3)	O(3)—C(10)—C(9)	113·6 (2)
C(3)—C(4)—C(5)	113·4 (3)	O(3)—C(10)—C(15)	60·8 (2)
C(3)—C(4)—C(11)	122·4 (3)	C(1)—C(10)—C(9)	115·9 (2)
C(5)—C(4)—C(11)	124·0 (4)	C(1)—C(10)—C(15)	121·6 (3)
C(4)—C(5)—C(6)	122·7 (3)	C(9)—C(10)—C(15)	117·5 (3)
C(5)—C(6)—C(7)	127·2 (3)	C(7)—C(12)—C(13)	113·5 (3)
C(6)—C(7)—C(8)	105·7 (3)	C(7)—C(12)—C(14)	109·1 (3)
C(6)—C(7)—C(12)	110·1 (3)	C(13)—C(12)—C(14)	110·9 (3)
C(8)—C(7)—C(12)	113·9 (3)	O(3)—C(15)—C(10)	58·9 (2)

Symmetry code: (i) $1-x$, $1-y$, $-z$.

Related literature. Periplanone A and B are pheromones isolated from *Periplaneta americana*. The relative configuration of periplanone A is unsettled because only 20 µg have been isolated from 75 000–100 000 virgin female cockroaches (Persoons, Verwiel, Ritter & Nooyen, 1982) and the total synthesis has not yet been performed. The structures of synthesized periplanone B (Hauptmann, Mühlbauer & Walker, 1986) and an intermediate alcohol, periplanol B (Adams, Nakanishi, Still, Arnold & Persoons, 1979), have been determined. The dihedral angles of the C=C—C=C moiety are −158 and −154° for periplanone B and periplanol B, respectively, while it is −53·2 (8)° in this compound. The difference may be attributable to the presence of the non-epoxide O(1)—

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* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44249 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

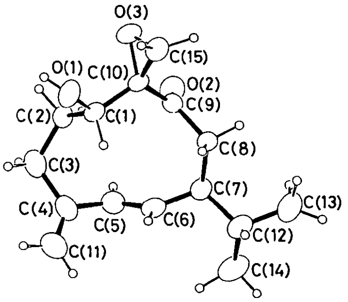

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the molecule with 50% probability ellipsoids. H atoms are represented by circles of radius 0·08 Å.

C(1)—C(2) group in the present compound, the torsion angle C(10)—C(1)—C(2)—C(3) being 148·1 (4)°. The corresponding values in periplanone B and periplanol B, which involve an epoxide moiety at the C(1)—C(2) bond, are −1·7 (5) and 0·7 (6)°, respectively.

References

ADAMS, M. A., NAKANISHI, K., STILL, W. C., ARNOLD, E. V. & PERSOONS, C. J. (1979). *J. Am. Chem. Soc.* **101**, 2493–2498.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HAUPTMANN, H., MÜHLBAUER, G. & WALKER, N. P. C. (1986). *Tetrahedron Lett.* **27**, 1315–1318.

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

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

PERSOONS, C. J., VERWIEL, P. E. J., RITTER, F. J. & NOOYEN, W. J. (1982). *J. Chem. Ecol.* **8**, 439–451.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

SHIZURI, Y., YAMAGUCHI, S., YAMAMURA, S., ISHIHARA, M., OHBA, S., SAITO, Y., NIWA, M., TERADA, Y. & MIYAZAKI, M. (1987). *Tetrahedron Lett.* **28**, 3831–3834.

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Structure of 6-Ethyl-3,3-dimethyl-1,2,3,4-tetrahydro-1,5-benzodiazocin-4-one

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Abstract. $C_{14}H_{18}N_2O$, $M_r = 230·31$, orthorhombic, $Pca2_1$, $a = 8·405$ (1), $b = 8·960$ (1), $c = 17·020$ (1) Å, $V = 1281·8$ (2) Å^3, $Z = 4$, $D_x = 1·194$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1·54184$ Å, $\mu = 5·66$ cm^{-1}, $F(000) = 496$, $T = 291$ K, $R = 0·0372$, $wR = 0·0358$ for 1049 unique observed reflections. The diazocine ring is boat-shaped. The dihedral angles between the least-squares planes defining the boat conformation are: plane 1 (N1,C2,C6,C6a,C10a) and plane 2 (C2,C3,N5,C6) 54·9°, plane 2 and plane 3 (C3,C4,N5) 123·7 (2)°. The benzene ring is essentially planar with a maximum deviation from the least-squares plane of 0·008 (4) Å (C8,C10a). Bond lengths and angular properties of the diazocine ring are in fair agreement with previously studied benzodiazocines. Subsequent molecules are linked together into a chain by hydrogen bonds running in the direction of the a axis.

Experimental. Crystals from CH_3CN, thin yellow transparent plates, size of specimen 0·50 × 0·40 × 0·05 mm. Stoe four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan, scan width 1·05–1·35°, scan speed 1·21–3·64° min^{-1}. 1161 reflections measured, $2·60 \leq \theta \leq 63·94°$, 1073 unique and 1049 with $I \geq 2\sigma(I)$ considered observed, $0 \leq h \leq 9$, $0 \leq k \leq 10$, $0 \leq l \leq 19$, three reflections checked every 60 min, variation in intensity below ±1%. No absorption correction was applied. Lattice parameters from least-squares refinement of 17 reflections, $5·58 \leq \theta \leq 20·13°$. Structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement with *SHELX*76 (Sheldrick, 1976) minimizing $\sum w(\Delta F)^2$. Non-H atoms anisotropic, H1 isotropically and individually refined, remaining H atoms in calculated positions with isotropic group temperature factors. $R = 0·0372$, $wR = 0·0358$, $w = 1/[\sigma^2(F) + 0·0001F^2]$, for 1049 reflections with $F \geq 4\sigma(F)$ and 186 parameters. Max. $\Delta/\sigma = 0·002$ for any parameter, max. and min. peaks in final $\Delta\rho$ map 0·12 and −0·19 e Å^{-3}. The inverse structure slightly worsened the R factors, although this difference was not statistically significant enough to identify the correct enantiomer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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