We thank the SERC for provision of equipment, and ICI Plant Protection Division and St John's College, Cambridge, for financial assistance to MJD.

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*Acta Cryst.* (1989). C**45**, 678–680

# 8-α-Hydroxyachillin, a Guaianolide Sesquiterpene Lactone from *Dendroseris neriifolia*

BY VERONICA CAMPOS AND MARIO SILVA

*Laboratorio de Quimica de Productos Naturales, Departamento de Botanica, Universidad de Concepcion, Concepcion, Chile*

AND WILLIAM H. WATSON* AND ANTE NAGL†

*Department of Chemistry, Texas Christian University, Fort Worth, Texas* 76129, *USA*

(*Received* 6 *July* 1988; *accepted* 31 *October* 1988)

**Abstract.** *rel*-(5*S*,6*S*,7*R*,8*S*,11*S*)-8-Hydroxy-2-oxoguaia-1(10),3-dien-6,12-olide methanol solvate, $C_{15}H_{18}O_4.CH_3OH$, $M_r = 294{\cdot}35$, monoclinic, $P2_1$, $a = 10{\cdot}047$ (1), $b = 7{\cdot}717$ (1), $c = 10{\cdot}193$ (1) Å, $\beta = 112{\cdot}72$ (1)°, $V = 728{\cdot}7$ (1) Å$^3$, $Z = 2$, $D_x = 1{\cdot}342$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}92$ cm$^{-1}$, $F(000) = 316$, $T = 293$ K, $R = 0{\cdot}0641$ for 1570 reflections. The cycloheptene ring is in a chair conformation, the five-membered cyclopentenone ring is planar while the five-membered lactone ring is in an envelope conformation. The structure contains a molecule of methanol solvent which is within hydrogen-bonded distance of the terpene, O(3)···O(5) ($x$, $y$, $1+z$) = 2·814 (4) Å, O(5)···O(4) ($1-x$, $y-0{\cdot}5$, $-z$) = 2·804 (4) Å and O(5)···O(3′) ($1-x$, $y-0{\cdot}5$, $-z$) = 2·939 (4) Å. The H atoms on the methanol could not be located. The achillin and matricarin series of sesquiterpenes differ only in the stereochemistry at C(11) in the lactone ring. Achillin is identified as the series with an α or *S* configuration at C(11).

**Experimental.** The plant *Dendroseris neriifolia* Hook & Arn. (Compositae-Lactuceae) was collected in the Juan Fernandez Islands (Masatierra) and the sesquiterpene was isolated by standard techniques. The pure sesquiterpene lactone (1) was recrystallized from methanol. A poor-quality colorless crystal of dimensions 0·32 × 0·40 × 0·50 mm was mounted on a Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer; data collected in the $\omega$ scan mode ($3 \le 2\theta \le 55°$), variable scan rate 4 to 29·3° min$^{-1}$, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($38{\cdot}14 \le 2\theta \le 43{\cdot}62°$); monitored reflections ($11\bar{5}$ and $\bar{1}40$) showed variations in intensities of less than $3\sigma(I)$; Laue symmetry $2/m$ and systematic absences ($0k0$, $k = 2n + 1$) consistent with space group $P2_1$; 1793 independent reflections measured ($-13 \le h \le 12$, $0 \le k \le 10$, $0 \le l \le 13$), equivalent reflections averaged, $1570 \ge 3\sigma(I)$; Lorentz–polarization corrections applied, $\psi$-scan-based empirical absorption correction (transmission factors 0·909–0·965); structure solved by direct methods and refined by anisotropic block-cascade least-squares techniques; the hydroxyl H atom was refined isotropically, all other terpene H atoms were located in a difference map but were constrained at a fixed distance from the attached atom; isotropic thermal parameters were refined for each H atom except for the methyl H atoms which were refined with a single isotropic thermal parameter; $R = 0{\cdot}0641$, $wR = 0{\cdot}0724$ for 204 parameters and 1570 reflections ($R = 0{\cdot}0945$ for all 1793 reflections), $S = 1{\cdot}527$, $(\Delta/\sigma)_{max} = 0{\cdot}006$ with the largest peaks in the final difference map of −0·39 and +0·28 e Å$^{-3}$; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00506F_o^2]^{-1}$; extinction correction $F = F_c/[1{\cdot}0 + 0{\cdot}002 \times 0{\cdot}009\ (3)\ F_c^2/\sin(2\theta)]^{0{\cdot}25}$ applied. Computer

* Author to whom correspondence should be addressed.

† On leave from the Faculty of Technology, University of Zagreb, Zagreb, Yugoslavia.

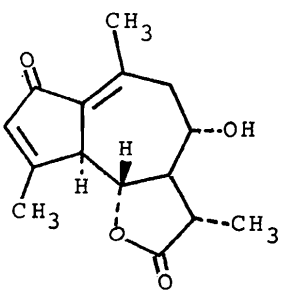
0108-2701/89/040678-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

| | x | y | z | U* |
|---|---|---|---|---|
| C(1) | 396 (3) | 3893 (5) | 242 (4) | 38 (1) |
| C(2) | −1154 (4) | 3557 (5) | −84 (4) | 44 (1) |
| C(3) | −1328 (4) | 3576 (6) | 1276 (4) | 49 (1) |
| C(4) | −83 (4) | 3872 (6) | 2360 (4) | 46 (1) |
| C(5) | 1155 (3) | 4037 (5) | 1852 (3) | 37 (1) |
| C(6) | 2259 (4) | 2595 (5) | 2372 (3) | 40 (1) |
| C(7) | 3697 (4) | 2902 (5) | 2220 (4) | 41 (1) |
| C(8) | 3594 (4) | 2810 (6) | 692 (4) | 43 (1) |
| C(9) | 2630 (4) | 4287 (5) | −202 (4) | 45 (1) |
| C(10) | 1045 (4) | 3975 (5) | −682 (3) | 40 (1) |
| C(11) | 4632 (4) | 1552 (7) | 3260 (4) | 57 (2) |
| C(12) | 4082 (5) | 1676 (8) | 4425 (4) | 64 (2) |
| C(13) | 123 (5) | 4057 (9) | 3878 (5) | 66 (2) |
| C(14) | 279 (5) | 3747 (7) | −2265 (4) | 58 (2) |
| C(15) | 6272 (5) | 1809 (12) | 3847 (6) | 90 (3) |
| O(1) | 2709 (3) | 2324 (5) | 3906 (3) | 55 (1) |
| O(2) | −2120 (3) | 3317 (5) | −1251 (3) | 59 (1) |
| O(3) | 5011 (3) | 3003 (5) | 719 (3) | 58 (1) |
| O(4) | 4667 (4) | 1312 (9) | 5643 (3) | 103 (2) |
| O(5) | 3925 (6) | 1411 (8) | 8026 (5) | 116 (3) |
| C(16) | 5052 (13) | 2845 (22) | 8128 (15) | 192 (8) |

* Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

programs were supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration (Nicolet Instrument Corporation, 1986). Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters and $U_{eq}$ values, Table 2 is a list of interatomic distances and valence angles, while Fig. 1 is a drawing of the title compound.*




(1)

**Related literature.** An excellent review of sesquiterpene lactones by Fischer, Olivier & Fischer (1979) contains references to the phytochemistry, the physical properties and X-ray crystal structures of this class of compounds. The physical properties of 8-α-hydroxyachillin and the C(11) epimer desacetylmatricarin have been reported by White & Winter (1963). Campos, Silva, Jakupovic, Bittner & Stuessey (1988) report the isolation and identification of 8-α-hydroxyachillin from *Dendroseris neriifolia*.

* 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 51556 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

| | | | |
|---|---|---|---|
| C(1)–C(2) | 1·484 (5) | C(1)–C(5) | 1·523 (4) |
| C(1)–C(10) | 1·338 (6) | C(2)–C(3) | 1·462 (7) |
| C(2)–O(2) | 1·224 (4) | C(3)–C(4) | 1·330 (5) |
| C(4)–C(5) | 1·526 (6) | C(4)–C(13) | 1·486 (7) |
| C(5)–C(6) | 1·515 (5) | C(6)–C(7) | 1·530 (6) |
| C(6)–O(1) | 1·465 (4) | C(7)–C(8) | 1·522 (5) |
| C(7)–C(11) | 1·524 (6) | C(8)–C(9) | 1·545 (5) |
| C(8)–O(3) | 1·420 (5) | C(9)–C(10) | 1·493 (5) |
| C(10)–C(14) | 1·506 (5) | C(11)–C(12) | 1·495 (7) |
| C(11)–C(15) | 1·533 (6) | C(12)–O(1) | 1·366 (6) |
| C(12)–O(4) | 1·183 (5) | O(5)–C(16) | 1·557 (17) |

| | | | |
|---|---|---|---|
| C(2)–C(1)–C(5) | 107·4 (3) | C(2)–C(1)–C(10) | 127·2 (3) |
| C(5)–C(1)–C(10) | 125·3 (3) | C(1)–C(2)–C(3) | 106·6 (3) |
| C(1)–C(2)–O(2) | 127·8 (4) | C(3)–C(2)–O(2) | 125·6 (4) |
| C(2)–C(3)–C(4) | 111·8 (4) | C(3)–C(4)–C(5) | 111·1 (4) |
| C(3)–C(4)–C(13) | 126·1 (5) | C(5)–C(4)–C(13) | 122·7 (3) |
| C(1)–C(5)–C(4) | 103·0 (3) | C(1)–C(5)–C(6) | 108·6 (3) |
| C(4)–C(5)–C(6) | 113·7 (3) | C(5)–C(6)–C(7) | 116·3 (3) |
| C(5)–C(6)–O(1) | 111·2 (3) | C(7)–C(6)–O(1) | 102·5 (2) |
| C(6)–C(7)–C(8) | 113·8 (3) | C(6)–C(7)–C(11) | 100·1 (3) |
| C(8)–C(7)–C(11) | 117·4 (4) | C(7)–C(8)–C(9) | 110·4 (3) |
| C(7)–C(8)–O(3) | 107·7 (3) | C(9)–C(8)–O(3) | 109·2 (4) |
| C(8)–C(9)–C(10) | 114·9 (3) | C(1)–C(10)–C(9) | 121·7 (3) |
| C(1)–C(10)–C(14) | 124·2 (3) | C(9)–C(10)–C(14) | 114·1 (4) |
| C(7)–C(11)–C(12) | 101·0 (4) | C(7)–C(11)–C(15) | 117·7 (5) |
| C(12)–C(11)–C(15) | 110·7 (4) | C(11)–C(12)–O(1) | 110·1 (3) |
| C(11)–C(12)–O(4) | 129·2 (5) | O(1)–C(12)–O(4) | 120·8 (5) |
| C(6)–O(1)–C(12) | 108·4 (3) | | |

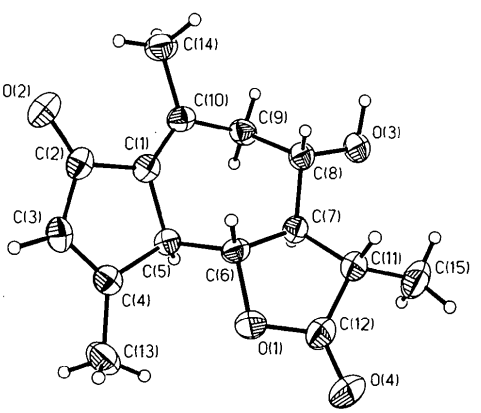



Fig. 1. Compound (1) with thermal ellipsoids drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

We thank Fondo Nacional de Ciencias (Chile), the Welch Foundation (P-074), the TCU Research Fund, the Organization of American States and the Volkswagen Foundation for financial support.

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*Acta Cryst.* (1989). C**45**, 680–681

# Structure of Diphenyl(2-pyridyl)phosphine*

BY JEAN-PIERRE CHARLAND†

*National Research Council of Canada, Division of Chemistry, Ottawa, Canada*

AND JEAN-LOUIS ROUSTAN† AND NASRIN ANSARI

*University of Ottawa, Department of Chemistry, Ottawa, Ontario, Canada K1N 9B5*

(*Received* 28 *July* 1988; *accepted* 20 *October* 1988)

**Abstract.** $C_{17}H_{14}NP$, $M_r = 263{\cdot}28$, monoclinic, $P2_1/n$, $a = 9{\cdot}9023\ (2)$, $b = 9{\cdot}8780\ (3)$, $c = 14{\cdot}8926\ (4)$ Å, $\beta = 92{\cdot}948\ (3)°$, $V = 1454{\cdot}8\ (1)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}202$ Mg m$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}70932$ Å, $\mu = 0{\cdot}17$ mm$^{-1}$, $F(000) = 552$, $T = 293$ K, $R = 0{\cdot}043$ for 1680 unique observed reflections. The phenyl and pyridyl rings are planar and make angles of 71·6 (1), 46·9 (1)° (phenyls) and 45·1 (1)° (pyridyl) with the basal plane of the PCCC pyramid.

**Experimental.** White crystal of $C_{17}H_{14}NP$ (Maisonnet, Far, Olmstead, Hunt & Balch, 1982) from diethyl ether–hexane at 273 K, 0·15 × 0·35 × 0·35 mm; Enraf–Nonius CAD-4 controlled by the NRCCAD software (Le Page, White & Gabe, 1986). Data collected in the $\theta$–$2\theta$ mode up to $2\theta = 55°$, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of the setting angles of 100 reflections ($35 \le 2\theta \le 45°$); systematic absences ($h0l$, $h + l = 2n + 1$, $0k0$, $k = 2n + 1$) consistent with space group $P2_1/n$; no statistically significant change in the intensities of the three standard reflections monitored ($\bar{1}3\bar{1}$, 006, $\bar{3}13$). 3446 unique reflections ($-12 \le h \le 12$, $0 \le k \le 12$, $0 \le l \le 19$), 1680 with $I_{net} \ge 3\sigma(I_{net})$.

Corrections for Lorentz and polarization effects applied but not for absorption. Structure solved by direct methods and difference-map techniques. N atom differentiated from C atoms by refining on occupancies with the ring atoms being defined as C atoms at first and with the isotropic $U$ values fixed at 0·04 Å$^2$. Positions of all the H atoms revealed by subsequent difference maps. Final refinement by full-matrix least squares on all atoms treating isotropically the H atoms and anisotropically all the others; $\sum w(|F_o| - |F_c|)^2$ was minimized with $w^{-1} = \sigma^2(F_o) + 0{\cdot}0002F_o^2$, $R = 0{\cdot}043$, $wR = 0{\cdot}041$, $S = 1{\cdot}5$ for 229 parameters and 1680 reflections ($R = 0{\cdot}094$, $wR = 0{\cdot}051$ for all 3446 reflections), $(\Delta/\sigma)_{max} = 0{\cdot}004$; in the final difference map, general background below 0·24 e Å$^{-3}$. All computations with *NRCVAX* system of programs (Gabe, Lee & Le Page, 1985); atom scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Atomic positions are listed in Table 1, selected bond lengths and

Table 1. *Atomic parameters x, y, z and* $B_{eq}$

E.s.d.'s refer to the last digit printed.

| | $x$ | $y$ | $z$ | $B_{eq}$* (Å$^2$) |
|---|---|---|---|---|
| P | 0·96935 (7) | 0·15507 (7) | 0·29975 (5) | 4·13 (3) |
| N18 | 1·14047 (22) | 0·3508 (3) | 0·26210 (15) | 5·88 (12) |
| C1 | 0·88855 (22) | 0·1617 (3) | 0·18700 (15) | 3·74 (11) |
| C2 | 0·8969 (3) | 0·0488 (3) | 0·13287 (22) | 4·87 (15) |
| C3 | 0·8419 (3) | 0·0493 (5) | 0·04591 (25) | 6·23 (19) |
| C4 | 0·7799 (3) | 0·1627 (5) | 0·01216 (24) | 6·64 (20) |
| C5 | 0·7704 (3) | 0·2755 (4) | 0·06388 (24) | 6·16 (18) |
| C6 | 0·8242 (3) | 0·2750 (3) | 0·15106 (21) | 4·87 (15) |
| C7 | 0·8292 (3) | 0·1559 (3) | 0·37458 (16) | 4·05 (11) |
| C8 | 0·6937 (3) | 0·1622 (3) | 0·34783 (21) | 4·98 (14) |
| C9 | 0·5956 (4) | 0·1565 (4) | 0·4115 (3) | 6·78 (20) |
| C10 | 0·6321 (5) | 0·1470 (4) | 0·5007 (3) | 7·32 (21) |
| C11 | 0·7649 (5) | 0·1405 (4) | 0·52758 (24) | 7·27 (21) |
| C12 | 0·8625 (4) | 0·1436 (3) | 0·46535 (20) | 5·61 (16) |
| C13 | 1·03095 (22) | 0·3302 (3) | 0·30983 (15) | 3·86 (11) |
| C14 | 0·9775 (3) | 0·4308 (3) | 0·36097 (18) | 4·35 (13) |
| C15 | 1·0372 (3) | 0·5563 (4) | 0·36375 (23) | 5·77 (17) |
| C16 | 1·1475 (4) | 0·5793 (4) | 0·31469 (23) | 6·55 (18) |
| C17 | 1·1944 (4) | 0·4745 (5) | 0·26558 (22) | 7·33 (20) |

* $B_{eq}$ is the mean of the principal axes of the thermal ellipsoid.

* Issued as NRCC Publication No. 29360.

† Authors to whom correspondence should be addressed.

0108-2701/89/040680-02$03.00