Table 1. *Experimental conditions*

Crystal size: 0·12 × 0·16 × 0·54 mm
Number and range of reflections used for lattice parameters: 25 (11 to 39° 2θ, Mo $K\alpha$)
Scan method: $2\theta/\theta$
Scan range: $2\theta(\alpha_1)-1$ to $2\theta(\alpha_2)+1°$
Data collection range: 4 to 60° (2θ), $\lambda = 0.71069$ Å
Range of *hkl* values: $\bar{h}k\bar{l}$, $\bar{h}kl$, $hk\bar{l}$, hkl: h −7 to +7, k 0 to +5, l −15 to +15
Scan speed: variable, 3 to 6° (2θ)/min.
Ratio background to scan time: 1·0
Number of measured reflections: 1541
Number of unique reflections: 633
R_{int}: 0·018
Number of observed reflections used in refinement: 563 with $|F_o| > 4\sigma(F_o)$
Number of parameters refined: 45
Final R, wR, S: 0·037, 0·039, 2·4

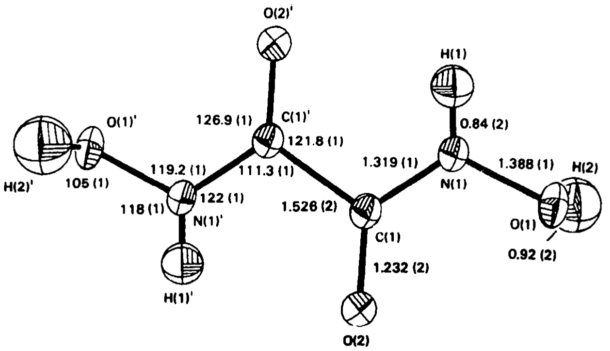

Fig. 1. The title compound plotted with 50% probability thermal ellipsoids. Bond lengths in Å; bond angles in degrees. Atoms with superscripts are related by inversion ($1-x$, $1-y$, $1-z$) to atoms without superscripts.

Table 2. *Atomic coordinates and thermal parameters*

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
C(1)	0·43868 (22)	0·41114 (28)	0·44102 (9)	0·0192 (3)†
N(1)	0·56255 (20)	0·48703 (27)	0·34288 (8)	0·0218 (3)†
O(1)	0·48935 (17)	0·32773 (24)	0·22919 (7)	0·0240 (3)†
O(2)	0·24779 (19)	0·22385 (24)	0·44280 (8)	0·0277 (3)†
H(1)	0·696 (4)	0·610 (4)	0·348 (1)	0·029 (4)‡
H(2)	0·101 (4)	−0·008 (5)	0·318 (2)	0·052 (6)‡

† $U_{\text{eq}} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.
‡ $U_{\text{iso}} = \exp[-8\pi^2u(\sin\theta/\lambda)^2]$.

Related literature. The observed hydrogen-atom positions and the C(1)—O(2) bond length [1·232 (2) Å] indicate that the molecule exists primarily as the keto tautomer (*b*). The C—N bond length [1·319 (1) Å] is similar to the C—N bond lengths observed in other molecules containing an $>$N—C=O moiety (Sutton, 1965) and suggests the possibility of some resonance as $>\overset{+}{\text{N}}$=C—O⁻. Torsional angles about N(1) indicate that N(1) is slightly pyramidal but flattened {ω[O(1)—N(1)—C(1)—O(2)] = 4·9, ω[H(1)—N(1)—C(1)—C(1)$^{\text{i}}$] = 2·9°}. A distance of 1·725 (2) Å between O(2) of one molecule and H(2) of another molecule indicates the presence of intermolecular hydrogen bonding.

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Acta Cryst. (1986). C42, 1649–1651

Redetermination of Pacifenol, a Halogenated Sesquiterpene from the Mediterranean Red Alga *Laurencia majuscula*

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(*Received* 15 *April* 1986; *accepted* 28 *May* 1986)

Abstract. (2*R*,5*R*,5a*R*,7*S*,8*S*,9a*S*)-2,7-Dibromo-8-chloro-2,5,7,8,9,9a-hexahydro-5,8,10,10-tetramethyl-2,5a-methano-6*H*-1-benzoxepin-5-ol, $C_{15}H_{21}Br_2ClO_2$, $M_r = 428.6$, orthorhombic, $P2_12_12_1$, $a = 12.190$ (2), $b = 19.113$ (4), $c = 7.004$ (3) Å, $V = 1631.8$ (13) Å^3, $Z = 4$, $D_x = 1.744$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 50.8$ cm^{-1}, $F(000) = 856$, $T = 299$ K, $R = 0.049$ for 2515 observations with $I > 0$. The C—OH distance, which was stated to be anomalously long [1·50 (1) Å] in the original, lower resolution determination, is

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0108-2701/86/111649-03$01.50

Table 1. *Coordinates and equivalent isotropic thermal parameters for pacifenol*

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

	x	y	z	B_{eq}(Å^2)		x	y	z	B_{eq}(Å^2)
Br(1)	0·61100 (4)	0·18309 (3)	−0·12463 (8)	5·48 (1)	C(6)	0·6233 (3)	0·3503 (2)	0·2563 (6)	2·74 (9)
Br(2)	0·49182 (4)	0·48792 (2)	0·67310 (6)	3·86 (1)	C(7)	0·7403 (3)	0·3815 (2)	0·2297 (7)	3·3 (1)
Cl	0·4390 (1)	0·53601 (5)	0·1581 (2)	4·43 (3)	C(8)	0·7887 (3)	0·3516 (2)	0·0467 (7)	4·3 (1)
O(1)	0·5466 (2)	0·3166 (1)	−0·0492 (4)	3·42 (6)	C(9)	0·7418 (3)	0·3031 (2)	−0·0564 (7)	4·1 (1)
O(2)	0·7274 (2)	0·4555 (1)	0·1912 (5)	4·23 (7)	C(10)	0·6345 (3)	0·2721 (2)	0·0057 (7)	3·6 (1)
C(1)	0·5507 (3)	0·3737 (2)	0·0850 (6)	2·59 (9)	C(11)	0·6266 (3)	0·2676 (2)	0·2243 (6)	3·3 (1)
C(2)	0·4354 (3)	0·3934 (2)	0·1360 (6)	2·95 (9)	C(12)	0·7212 (4)	0·2259 (2)	0·3144 (8)	4·9 (1)
C(3)	0·4269 (3)	0·4533 (2)	0·2841 (6)	2·70 (9)	C(13)	0·5197 (4)	0·2327 (2)	0·2870 (7)	4·2 (1)
C(4)	0·5256 (3)	0·4492 (2)	0·4210 (6)	2·94 (9)	C(14)	0·8164 (3)	0·3747 (2)	0·3985 (8)	4·6 (1)
C(5)	0·5713 (3)	0·3745 (2)	0·4434 (6)	2·82 (9)	C(15)	0·3143 (3)	0·4524 (3)	0·3748 (8)	4·5 (1)

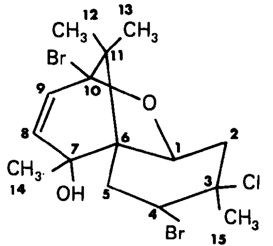
normal, 1·448 (5) Å. The present redetermination, on material from a new natural source, also increases the precision twofold, includes location and refinement of H atoms, places coordinates in the open literature, and confirms the absolute configuration, with correlation to chiroptical data.

Experimental. Colorless prisms of (I) from *n*-hexane–benzene, m.p. 416–417 K. Crystal size 0·28 × 0·40 × 0·40 mm, space group from systematic absences $h00$ with h odd, $0k0$ with k odd, $00l$ with l odd, cell dimensions from setting angles of 25 reflections having $11 < \theta < 12°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time 120 s, scan rates varied 0·43–4·0° min^{-1}. Data having $1 < \theta < 28°$, $0 \leq h \leq 16$, $0 \leq k \leq 25$, $0 \leq l \leq 9$ measured, also nonequivalent $h\bar{k}l$ octant to $\theta = 20°$. Data corrected for background, Lorentz, polarization, absorption by ψ scans, min. relative transmission coefficient 84·75%. Standard reflections 200, 040, 006, ±2·1% variation. 2864 unique data, 2515 observed with $I > 0$. Structure solved by heavy-atom methods, refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H anisotropic, H atoms located from ΔF map and refined isotropically. Final* $R = 0{\cdot}049$ (0·077 for all data), $wR = 0{\cdot}030$, $S = 1{\cdot}445$ for 265 variables. Max. $\Delta/\sigma = 0{\cdot}12$ in final cycle, max. residual density 0·76 e Å^{-3} [near Br(2)], min. −0·53 e Å^{-3}. Enantiomorph yielded $R = 0{\cdot}083$, $wR = 0{\cdot}064$, $S = 2{\cdot}900$. Reported configuration has $[\alpha]_D^{20.0\,°C} = -2{\cdot}5°$[CHCl$_3$, 10·0 g dm^{-3}].

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

O(1)–C(1)	1·441 (4)	C(5)–C(6)	1·527 (5)
O(1)–C(10)	1·421 (5)	C(6)–C(7)	1·558 (5)
O(2)–C(7)	1·448 (5)	C(6)–C(11)	1·596 (5)
O(2)–H(20)	0·81 (4)	C(7)–C(8)	1·523 (7)
C(1)–C(2)	1·498 (5)	C(7)–C(14)	1·508 (7)
C(1)–C(6)	1·556 (5)	C(8)–C(9)	1·307 (6)
C(2)–C(3)	1·549 (5)	C(9)–C(10)	1·500 (6)
C(3)–Cl	1·816 (4)	C(10)–Br(1)	1·952 (4)
C(3)–C(4)	1·540 (5)	C(10)–C(11)	1·537 (6)
C(3)–C(15)	1·513 (6)	C(11)–C(12)	1·538 (6)
C(4)–Br(2)	1·958 (4)	C(11)–C(13)	1·528 (6)
C(4)–C(5)	1·541 (5)	C–H range	0·84 (5)–1·17 (7)
C(1)–O(1)–C(10)	104·5 (3)	C(7)–C(6)–C(11)	109·8 (3)
C(7)–O(2)–H(20)	106 (3)	O(2)–C(7)–C(6)	107·2 (3)
O(1)–C(1)–C(2)	108·3 (3)	O(2)–C(7)–C(8)	104·6 (4)
O(1)–C(1)–C(6)	107·8 (3)	O(2)–C(7)–C(14)	107·3 (4)
C(2)–C(1)–C(6)	115·0 (4)	C(6)–C(7)–C(8)	108·1 (4)
C(1)–C(2)–C(3)	114·1 (3)	C(6)–C(7)–C(14)	115·9 (4)
Cl–C(3)–C(2)	108·2 (3)	C(8)–C(7)–C(14)	112·9 (4)
Cl–C(3)–C(4)	106·5 (3)	C(7)–C(8)–C(9)	124·2 (5)
Cl–C(3)–C(15)	106·7 (3)	C(8)–C(9)–C(10)	120·1 (5)
C(2)–C(3)–C(4)	109·1 (3)	Br(1)–C(10)–O(1)	106·5 (3)
C(2)–C(3)–C(15)	109·4 (4)	Br(1)–C(10)–C(9)	109·7 (3)
C(4)–C(3)–C(15)	116·5 (4)	Br(1)–C(10)–C(11)	114·1 (3)
Br(2)–C(4)–C(3)	112·2 (3)	O(1)–C(10)–C(9)	110·1 (4)
Br(2)–C(4)–C(5)	109·5 (3)	O(1)–C(10)–C(11)	104·8 (3)
C(3)–C(4)–C(5)	113·1 (3)	C(9)–C(10)–C(11)	111·5 (4)
C(4)–C(5)–C(6)	110·1 (3)	C(6)–C(11)–C(10)	94·9 (3)
C(1)–C(6)–C(5)	109·8 (3)	C(6)–C(11)–C(12)	118·4 (4)
C(1)–C(6)–C(7)	108·6 (3)	C(6)–C(11)–C(13)	111·8 (4)
C(1)–C(6)–C(11)	101·0 (3)	C(10)–C(11)–C(12)	113·0 (4)
C(5)–C(6)–C(7)	111·5 (3)	C(10)–C(11)–C(13)	111·4 (4)
C(5)–C(6)–C(11)	115·5 (3)	C(12)–C(11)–C(13)	107·2 (4)

The atomic parameters are given in Table 1, bond distances and angles in Table 2. Fig. 1 shows the absolute configuration and numbering scheme.



(I)

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* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43108 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

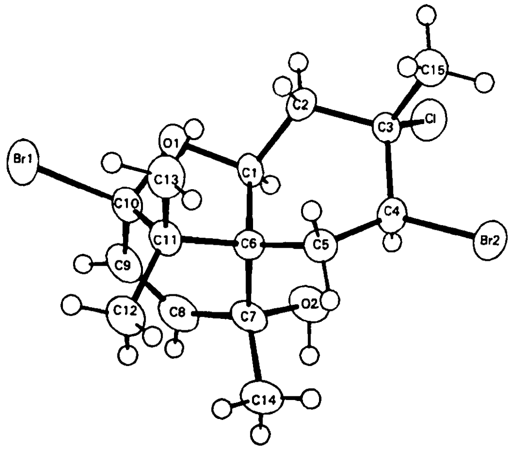

Fig. 1. Absolute configuration and standard sesquiterpene numbering scheme for title compound.

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Acta Cryst. (1986). C**42**, 1651–1652

Structure of Octahydro-2a*H*-azirino[1,2-*a*]indol-2a-ol

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(*Received* 3 *March* 1986; *accepted* 18 *June* 1986)

Abstract. $C_9H_{15}NO$, $M_r = 153{\cdot}2$, monoclinic, $P2_1/c$, $a = 10{\cdot}57\,(2)$, $b = 8{\cdot}93\,(1)$, $c = 9{\cdot}36\,(1)$ Å, $\beta = 105{\cdot}4\,(2)°$, $V = 851{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}20$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}724$ cm^{-1}, $F(000) = 336$, $T = 294$ K, $R = 0{\cdot}059$ for 929 observed reflexions. The stereochemistry of the molecule has been established. Bond lengths: C—C in the three-membered ring, 1·460 Å; other C—C, 1·510–1·533 Å; C—N, 1·479–1·486 Å; C—O, 1·403 Å. Shortest intermolecular contact: O—H···N, 2·801 Å.

Experimental. Material prepared from 2-cyanohexahydroindolium salts by reduction with $LiAlH_4$ and hydrolysis with NaOH, white crystals (m.p. 402 K) from ether (Carlsson, Olesen & Lawesson, 1980). Crystal size 0·2 × 0·4 × 0·8 mm, space group and unit cell from photographs taken with Cu and Mo radiations. Data collection on a computer-controlled Supper diffractometer (Weissenberg geometry) with graphite-monochromated Mo $K\alpha$ radiation, a scintillation counter and a pulse-height analyser. 2484 independent reflexions were measured by the background–peak–background method out to $\sin\theta = 0{\cdot}5$, $-14 \leq h \leq 14$, $0 \leq k \leq 12$, $0 \leq l \leq 13$. Standard reflexion 202 monitored every 25 reflexions, ±4% variation; data rescaled to correct for this. 929 reflexions with $I > 3\sigma(I)$ were used in the refinement. No correction was applied for absorption. Structure solved with *MULTAN* (Germain, Main & Woolfson, 1971). Coordinates, anisotropic thermal parameters

0108-2701/86/111651-02$01.50