

tetrahedral environment, donating two hydrogen bonds as an >NH₂⁺ group. All three hydrogen bonds involving the O⁻ atom are short: 2.566 (3) to O(2), 2.660 (4) to N(1) and 2.672 (4) Å to O(3). These distances, and the virtual equality of the CCO(1) angles [120.7 (3) and 121.4 (3)°], confirm the charged nature and lack of a proton on this atom. For each quinol OH group, the CCO angles are unequal, on account of the repulsion between the hydroxyl H atom and the CH group in the adjacent *ortho* position (see Fig. 3).

The neutral quinol molecules in (II) do not differ significantly in dimensions from each other, or from previously determined quinol dimensions (Wallwork & Powell, 1980, and references therein), or even from those of the Q⁻ ion. However, there is a tendency for C—O⁻ to be shorter than C—OH, for the C—C bonds adjacent to C—O⁻ to be longer, and for the ring CCC angle at this position to be slightly smaller. These changes are in the same direction, but smaller, than those noted (Hough, 1976) for nitrophenolate ions compared with nitrophenols.

We thank the Ministry of Higher Education and Scientific Research of Iraq for a maintenance grant to MMM, the Science and Engineering Research Council for contributing to the cost of the diffractometer, Dr M. J. Begley for helpful discussions, and the late Professor T. J. King for assistance with the computing.

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Acta Cryst. (1991). C47, 1438–1440

Structure of Cheloviolen A from the Sponge *Chelonaphysilla violacea*

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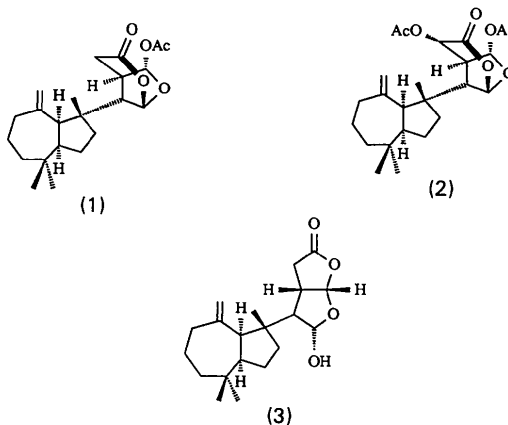
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(Received 18 September 1990; accepted 25 October 1990)

Abstract. 6-(Decahydro-1,4,4-trimethyl-8-methylene-1-azulenyl)-7-hydroxy-2,8-dioxabicyclo[3.3.0]octan-3-one, C₂₀H₃₀O₄, *M_r* = 334.46, orthorhombic, *P*₂₁₂₁₂₁, *a* = 8.614 (3), *b* = 13.441 (1), *c* = 15.948 (2) Å, *V* = 1846.5 (9) Å³, *Z* = 4, *D_m* = 1.17 (1), *D_x* = 1.20 g cm⁻³, *Mo Kα*, λ = 0.71069 Å, μ = 0.88 cm⁻¹, *F*(000) = 712, *T* = 292.5 (5) K, *R* = 0.047 for 1230 observed reflections. The X-ray analysis confirms the overall structure of cheloviolen A and establishes the relative stereochemistries at the seven asymmetric centres.

Introduction. The Dendroceratid sponge *Chelonaphysilla violacea* has yielded a series of related diterpenoid compounds, including aplyviolen (1) for which we have reported a structure determined by X-ray analysis (Buckleton, Bergquist, Cambie, Clark, Karuso & Rickard, 1986). Hambley, Poiner & Taylor (1986) have also reported the structure of aplyviolen and of aplyviolacene (2) obtained from

the same source. The structures of the remaining diterpenoids have been determined from a detailed examination of ¹H and ¹³C NMR spectra (Taylor *et al.*, unpublished). We record here confirmation of the structure of one of these compounds, cheloviolen A (3), m.p. 434–436 K, [α]_D^{17°C} = +6° (*c* 1.0M, CHCl₃), λ_{max} 1785 (γ-lactone), 1630 cm⁻¹ (C=C).



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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors for non-H atoms
$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
C(1)	7470 (7)	5687 (3)	1917 (4)	5.3 (6)
C(2)	8686 (7)	5305 (4)	1299 (4)	5.8 (6)
C(3)	8168 (6)	4510 (5)	690 (3)	5.8 (4)
C(4)	7501 (6)	3540 (3)	1051 (3)	4.2 (3)
C(5)	5947 (5)	3646 (3)	1528 (3)	3.3 (2)
C(6)	4635 (6)	4176 (4)	1070 (3)	4.5 (4)
C(7)	3349 (5)	4198 (4)	1713 (3)	4.4 (3)
C(8)	4151 (5)	4441 (3)	2563 (3)	3.2 (2)
C(9)	5940 (5)	4129 (3)	2425 (2)	2.9 (2)
C(10)	7087 (5)	4941 (3)	2572 (3)	3.7 (3)
C(11)	3175 (5)	3069 (3)	5365 (3)	3.5 (3)
O(11)	2393 (3)	3917 (2)	5328 (2)	4.1 (3)
O(11')	2949 (4)	2496 (3)	5935 (2)	5.4 (4)
C(12)	4277 (5)	2977 (4)	4654 (3)	3.9 (3)
C(13)	4105 (4)	3908 (3)	4144 (2)	3.0 (3)
C(14)	3454 (4)	3765 (3)	3250 (2)	2.5 (2)
C(15)	1703 (4)	3915 (3)	3384 (3)	3.0 (3)
O(15)	1583 (3)	4619 (2)	4064 (2)	3.6 (2)
O(15')	1070 (3)	3003 (2)	3609 (2)	4.0 (3)
C(16)	2832 (5)	4499 (3)	4597 (3)	3.3 (3)
C(17)	3941 (6)	5533 (3)	2774 (4)	4.9 (4)
C(18)	8705 (6)	3048 (4)	1622 (4)	5.2 (5)
C(19)	7194 (7)	2845 (6)	301 (4)	7.4 (8)
C(20)	7862 (5)	4962 (4)	3316 (3)	4.9 (4)

Experimental. Crystallization from ethyl acetate/hexane. The crystal density was measured by flotation in aqueous KI. Crystal $0.44 \times 0.20 \times 0.24$ mm, colourless rectangular prism, mounted on Nonius CAD-4 diffractometer, Zr-filtered Mo $K\alpha$ radiation; unit-cell dimensions from 25 reflections $11.8 < \theta < 13.4^\circ$; systematic absences $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$ defined space group as $P2_12_12_1$; 2098 unique reflections; $\omega/2\theta$ scans, $1 \leq \theta \leq 26^\circ$, $[(\sin\theta)/\lambda]_{\text{max}} 0.6168 \text{ \AA}^{-1}$, 1230 with $I > 2.5\sigma(I)$; $0 \leq h \leq 11$, $0 \leq k \leq 17$, $0 \leq l \leq 20$; three intensity standards checked every 100 reflections showed no non-statistical variation during data collection; Lorentz and polarization corrections applied, absorption corrections by empirical ψ -scan data, max. and min. correction factors 1.0000 and 0.9835, respectively. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) with the best E map revealing positions for all non-H atoms. F_{obs} refinement of atomic positions, non-H atoms assigned anisotropic thermal parameters, H atoms located in difference map and refined with fixed isotropic temperature factors of $U = 0.06 \text{ \AA}^2$. Final weight $w = 1/[\sigma^2(F) + 4.69 \times 10^{-3}F^2]$, $R = 0.047$, $wR = 0.050$, $S = 0.65$; $\Delta_{\text{max}}/\sigma = 0.132$ for positions, max. and min. $\Delta\rho$ excursions in final difference map 0.18 and -0.19 e \AA^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). Calculations performed with the Enraf-Nonius (1981) *Structure Determination Package* on a PDP-11 computer for initial data reduction and with *SHELX76*

Table 2. Bond distances (\AA) and angles ($^\circ$)

C(1)—C(2)	1.528 (8)	C(9)—C(10)	1.491 (6)
C(1)—C(10)	1.485 (7)	C(10)—C(20)	1.362 (7)
C(2)—C(3)	1.511 (8)	C(11)—O(11)	1.325 (5)
C(3)—C(4)	1.536 (8)	C(11)—O(11')	1.207 (6)
C(4)—C(5)	1.546 (7)	C(11)—C(12)	1.484 (6)
C(4)—C(18)	1.531 (7)	O(11)—C(16)	1.455 (5)
C(4)—C(19)	1.540 (9)	C(12)—C(13)	1.499 (6)
C(5)—C(6)	1.522 (6)	C(13)—C(14)	1.545 (5)
C(5)—C(9)	1.571 (6)	C(13)—C(16)	1.534 (6)
C(6)—C(7)	1.509 (6)	C(14)—C(15)	1.537 (5)
C(7)—C(8)	1.556 (6)	C(15)—O(15)	1.442 (5)
C(8)—C(9)	1.612 (6)	C(15)—O(15')	1.389 (5)
C(8)—C(14)	1.545 (6)	O(15)—C(16)	1.380 (5)
C(8)—C(17)	1.517 (6)		
C(2)—C(1)—C(10)	112.3 (4)	C(8)—C(9)—C(10)	114.9 (3)
C(1)—C(2)—C(3)	116.7 (5)	C(1)—C(10)—C(9)	122.1 (4)
C(2)—C(3)—C(4)	118.0 (5)	C(1)—C(10)—C(20)	119.3 (4)
C(3)—C(4)—C(5)	115.4 (4)	C(9)—C(10)—C(20)	118.5 (4)
C(3)—C(4)—C(18)	109.6 (4)	O(11)—C(11)—O(11')	120.0 (4)
C(3)—C(4)—C(19)	106.7 (4)	O(11)—C(11)—C(12)	111.2 (4)
C(5)—C(4)—C(18)	109.5 (4)	O(11')—C(11)—C(12)	128.7 (4)
C(5)—C(4)—C(19)	106.8 (4)	C(11)—O(11)—C(16)	111.5 (3)
C(18)—C(4)—C(19)	108.4 (4)	C(11)—C(12)—C(13)	106.4 (4)
C(4)—C(5)—C(6)	116.8 (4)	C(12)—C(13)—C(14)	115.6 (3)
C(4)—C(5)—C(9)	119.3 (4)	C(12)—C(13)—C(16)	104.4 (3)
C(6)—C(5)—C(9)	103.9 (3)	C(14)—C(13)—C(16)	103.8 (3)
C(5)—C(6)—C(7)	103.2 (4)	C(8)—C(14)—C(13)	116.2 (3)
C(6)—C(7)—C(8)	105.5 (4)	C(8)—C(14)—C(15)	113.8 (3)
C(7)—C(8)—C(9)	104.5 (3)	C(13)—C(14)—C(15)	102.2 (3)
C(7)—C(8)—C(14)	108.8 (3)	C(14)—C(15)—O(15)	105.1 (3)
C(7)—C(8)—C(17)	110.1 (4)	C(14)—C(15)—O(15')	107.8 (3)
C(9)—C(8)—C(14)	108.4 (3)	O(15)—C(15)—O(15')	110.9 (3)
C(9)—C(8)—C(17)	113.3 (3)	C(15)—O(15)—C(16)	109.3 (3)
C(14)—C(8)—C(17)	111.4 (4)	O(11)—C(16)—C(13)	106.5 (3)
C(5)—C(9)—C(8)	103.6 (3)	O(11)—C(16)—O(15)	110.7 (3)
C(5)—C(9)—C(10)	116.3 (3)	C(13)—C(16)—O(15)	109.1 (3)

(Sheldrick, 1976) on the University of Auckland IBM 4341 computer for refinement. Diagrams were produced using *ORTEP* (Johnson, 1965).

Discussion. Atomic coordinates for non-H atoms are listed in Table 1.* Bond distances and angles are given in Table 2. The atomic numbering and molecular geometry are shown in Fig. 1, which does not necessarily represent the true chirality of the molecule.

The X-ray analysis confirms the overall structure for cheloviolene A (3) and establishes the relative stereochemistries at the seven asymmetric centres. The bond length C(8)—C(9) is quite long [1.612 (6) \AA], presumably as a result of sterically induced strain, but all other bonds are normal. The bond angles at the sp^3 -hybridized C atoms reveal evidence for considerable strain in the molecule, especially in the region of the fusion of the seven- and five-membered rings and the link to the other half of the molecule. Thus C(4)—C(5)—C(9) is

* Lists of structure factors, thermal parameters and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53688 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

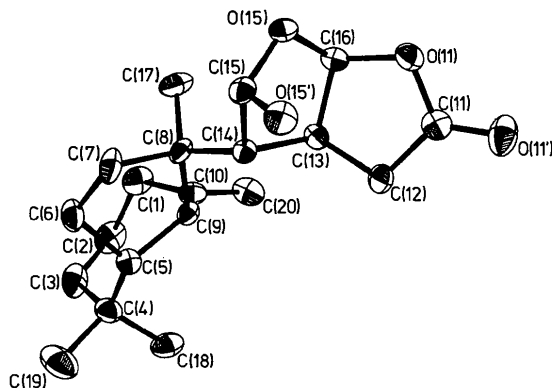


Fig. 1. The molecular geometry and atomic numbering for cheloviolen A (3). Atoms are represented as 50% probability ellipses.

119.3 (4)° whereas C(6)—C(5)—C(9) is 103.9 (3)°; C(5)—C(9)—C(10) is 116.3 (3)° whereas C(5)—C(9)—C(8) is 103.6 (3)°; C(8)—C(14)—C(13) is 116.2 (3)° whereas C(13)—C(14)—C(15) is 102.2 (3)°.

The molecules pack into the unit cell in the manner shown in the stereopair diagrams of Fig. 2. The only significant intermolecular interaction is a hydrogen bond between O(15') and O(11') [O...O 2.86 (1) Å]. Seven other intermolecular contacts are in the range 3.31–3.46 Å.

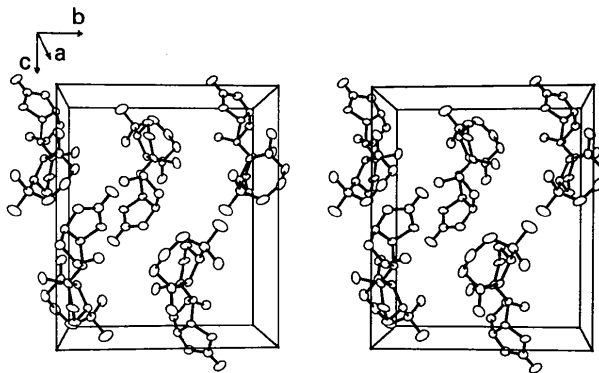


Fig. 2. Stereopair diagrams for cheloviolen A (3).

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Acta Cryst. (1991). **C47**, 1440–1442

2,2,4,4,6,6-Hexamethyl-1,3,5-triphenylcyclotrisilazane

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(Received 10 August 1990; accepted 25 October 1990)

Abstract. $C_{24}H_{33}N_3Si_3$, $M_r = 447.81$, triclinic, $P\bar{1}$, $a = 6.8234$ (11), $b = 11.1822$ (19), $c = 17.751$ (3) Å, $\alpha = 75.900$ (12), $\beta = 79.226$ (13), $\gamma = 81.998$ (9)°, $V = 1284$ Å³, $Z = 2$, $D_x = 1.158$ Mg m⁻³, $\bar{\lambda}(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.194$ mm⁻¹, $F(000) = 480$, $T = 298$ K, $R = 0.0488$ for 2374 unique observed reflections. The six-membered Si_3N_3 ring is markedly non-planar and the *N*-phenyl rings are almost perpendicular to the local Si—N—Si planes.

Introduction. The structure of the title compound (I) had been incorrectly assigned to a reaction product

which was subsequently shown to be the homologous four-membered ring compound 2,2,4,4-tetramethyl-1,3-diphenylcyclodisilazane (II). The crystal structure of (II) has been published (Párkányi, Argay, Hencsei & Nagy, 1976) and since the synthesis of (I) has recently been reported (Dejak, Kulpiński, Lasocki & Piechucki, 1987), we felt that it was timely to determine the structure of this compound.

Experimental. Colourless needle (0.89 × 0.15 × 0.11 mm), Stoe Stadi-4 four-circle diffractometer, unit-cell parameters from 2θ values of 22 reflections measured at $\pm\omega$ ($2\theta = 30$ – 32°). For data collection, graphite-monochromated Mo $K\alpha$ X-radiation, ω - 2θ

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