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Structure of 9-Annulene Derivatives. 3. 7*H*-Benzocyclononen-7-ol, $C_{13}H_{12}O$

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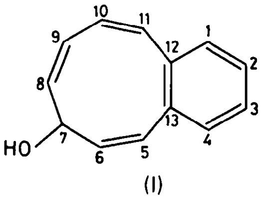
Abstract. $M_r = 184{\cdot}2$, $F(000) = 392$, triclinic, $P\bar{1}$, $a = 5{\cdot}688\ (2)$, $b = 12{\cdot}878\ (4)$, $c = 14{\cdot}138\ (6)$ Å, $\alpha = 80{\cdot}85\ (4)$, $\beta = 85{\cdot}52\ (3)$, $\gamma = 89{\cdot}79\ (3)°$, $U = 1019{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}20$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}080$ mm^{-1}, $T = 295$ K, final $R = 0{\cdot}038$, 2139 observed reflections. The asymmetric unit contains two independent molecules. The nine-membered ring is in the tub conformation. The crystal structure is built up of $C_{13}H_{12}O$ units linked into tetramers by O···O′ hydrogen bonds.

Introduction. In this paper we investigate the structural properties of a 9-annulenol (I).



(I)

Experimental. Suitable crystals of the title compound grown at room temperature by slow evaporation from an ether solution. Crystal 0·38 × 0·55 × 0·41 mm. Enraf–Nonius CAD-4 diffractometer, take-off angle 5°, graphite-crystal-monochromatized Mo $K\alpha$ radiation. Cell dimensions: least-squares refinement of the setting angles of 25 accurately centred reflections ($\theta \leq 16°$). 3585 independent reflections ($R_{\rm int} = 0{\cdot}015$), ω–2θ scans ($\theta \leq 25°$), background scans on each side of the peaks. Three standard reflections: no significant intensity variation. Automatic multisolution routine of *SHELX*76 (Sheldrick, 1976) used to generate a series of *E* maps, one of which contained recognizable fragments of two crystallographically independent molecules; successive difference syntheses phased on these fragments enabled all non-hydrogen atoms to be located. 2139 reflections considered observed $[I \geq 2\sigma(I)]$ and used in subsequent steps. Application of full-matrix least-squares refinement with isotropic temperature factors gave $R = 0{\cdot}125$; incorporation of anisotropic temperature factors reduced R to 0·088. All H-atoms located at this stage from a difference map. Further refinement of non-H atoms anisotropically and H atoms isotropically reduced R to 0·040. No absorption correction. Refinement of an empirical extinction coefficient caused a significant reduction in R to 0·038. Weighting scheme based on counting statistics; $w = 1{\cdot}10/(\sigma^2|F_o| + 7{\cdot}46 \times 10^{-4}\,F_o^2)$; $\sum w\Delta F^2$ minimized; $R_w = 0{\cdot}040$. $S = 1{\cdot}18$. A final difference map showed no peaks or depressions larger than 0·13 e Å^{-3}. $(\Delta/\sigma)_{\max} = 0{\cdot}0$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Fig. 1 shows the least-squares plane of the molecule in isometric projection, with 20% probability vibration ellipsoids for the non-H atoms and spheres of arbitrary radii 0·10 Å for H atoms. Fig. 2 shows the packing arrangement in the crystal. Both figures were produced using *ORTEP* (Johnson, 1976). Table 1 lists the fractional atomic coordinates and Table 2 lists bond distances and angles involving non-hydrogen atoms together with the endocyclic torsion angles for the nine-membered ring.†

The asymmetric unit in the present study contains two independent molecules. These molecules show some differences in their structural parameters, presumably due to their different intermolecular environments, with the largest deviation occurring in the absolute magnitude of the torsion angle C(8)—C(9)—C(10)—C(11) which is 30·6 (5)° in molecule *A* and 40·5 (5)° in molecule *B*.

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[†] Lists of coordinates and bond lengths and angles involving H atoms, and structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38901 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The general pattern established for the nine-membered ring from parts 1 and 2 (Mazhar-ul-Haque, Ahmed & Horne, 1983) of this series is maintained in the present compound, which exhibits a tub conformation, and detailed structural properties closely paralleling those of the 9-annulene (part 1). Thus the localized C=C bond distance (averaged over molecules *A* and *B*) is 1·320 (5) Å, which is identical with the value for 9-annulene. Similarly, the bond and torsion angles, again averaged over molecules *A* and *B*, compare well with the values for the corresponding angles in the 9-annulene (r.m.s. differences 2·3 and 5·1° respectively).

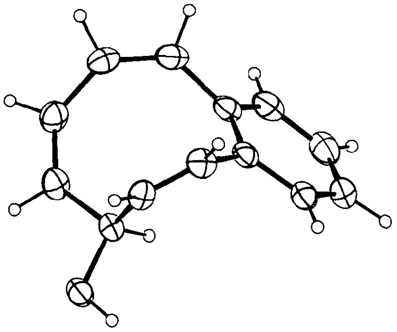

Fig. 1. View of the molecule.

Table 1. *Fractional coordinates* ($\times 10^4$) *and* U_{eq} *values* ($Å^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$(trace of orthogonalized U_{ij} matrix).

	x	*y*	*z*	U_{eq}
Molecule *A*				
C(1)	2873 (5)	4699 (2)	6548 (2)	63
C(2)	3257 (6)	4167 (2)	5778 (2)	69
C(3)	5145 (5)	3505 (2)	5742 (2)	67
C(4)	6586 (5)	3346 (2)	6488 (2)	58
C(5)	7661 (4)	3596 (2)	8112 (2)	54
C(6)	6888 (4)	3008 (2)	8922 (2)	50
C(7)	4495 (4)	2515 (2)	9108 (2)	46
C(8)	2862 (4)	3083 (2)	9722 (2)	55
C(9)	2417 (4)	4098 (2)	9663 (2)	59
C(10)	3247 (4)	5035 (2)	9002 (2)	58
C(11)	3971 (4)	5224 (2)	8074 (2)	60
C(12)	4308 (4)	4547 (2)	7317 (2)	51
C(13)	6179 (4)	3845 (2)	7287 (2)	48
O(14)	4628 (3)	1470 (1)	9619 (1)	67
Molecule *B*				
C(1)	8973 (5)	1384 (2)	4422 (2)	72
C(2)	10640 (6)	2141 (2)	4445 (2)	78
C(3)	11410 (6)	2790 (2)	3615 (2)	74
C(4)	10513 (5)	2674 (2)	2761 (2)	64
C(5)	8053 (5)	1708 (2)	1787 (2)	56
C(6)	8868 (4)	936 (2)	1358 (2)	52
C(7)	10768 (4)	219 (2)	1736 (2)	49
C(8)	9903 (5)	−817 (2)	2274 (2)	57
C(9)	8067 (5)	−1071 (2)	2904 (2)	62
C(10)	6160 (5)	−468 (2)	3297 (2)	66
C(11)	6120 (5)	462 (2)	3576 (2)	68
C(12)	8047 (4)	1240 (2)	3565 (2)	56
C(13)	8858 (4)	1895 (2)	2720 (1)	52
O(14)	12321 (3)	−28 (1)	960 (1)	72

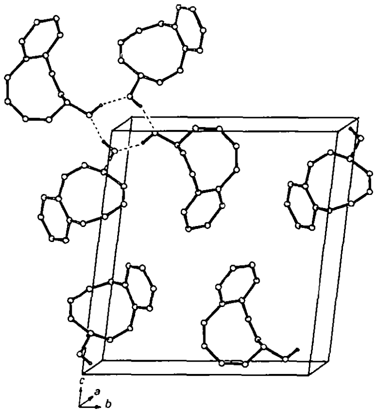

Fig. 2. The packing arrangement. Hydrogen bonds are indicated by broken lines.

Table 2. *Bond lengths, angles and selected torsion angles*

(*a*) Distances (Å)

	Molecule *A*		Molecule *B*	
		corrected		corrected
C(2)–C(1)	1·378 (3)	1·384	1·368 (4)	1·375
C(12)–C(1)	1·400 (3)	1·405	1·396 (3)	1·402
C(3)–C(2)	1·371 (4)	1·377	1·372 (4)	1·377
C(4)–C(3)	1·376 (4)	1·381	1·376 (3)	1·383
C(13)–C(4)	1·389 (3)	1·394	1·389 (3)	1·395
C(6)–C(5)	1·314 (3)	1·318	1·310 (3)	1·315
C(13)–C(5)	1·486 (3)	1·492	1·484 (3)	1·491
C(7)–C(6)	1·491 (3)	1·499	1·495 (3)	1·503
C(8)–C(7)	1·491 (3)	1·496	1·491 (3)	1·497
O(14)–C(7)	1·427 (2)	1·433	1·429 (2)	1·436
C(9)–C(8)	1·321 (3)	1·327	1·325 (3)	1·332
C(10)–C(9)	1·459 (3)	1·465	1·458 (3)	1·465
C(11)–C(10)	1·329 (3)	1·333	1·318 (3)	1·323
C(12)–C(11)	1·486 (3)	1·492	1·486 (3)	1·493
C(13)–C(12)	1·396 (3)	1·402	1·396 (3)	1·402

(*b*) Angles (°)

	Molecule *A*	Molecule *B*
C(12)–C(1)–C(2)	121·5 (3)	121·7 (3)
C(3)–C(2)–C(1)	119·6 (3)	119·8 (3)
C(4)–C(3)–C(2)	119·9 (3)	119·9 (3)
C(13)–C(4)–C(3)	121·4 (3)	121·1 (2)
C(13)–C(5)–C(6)	122·3 (2)	122·1 (2)
C(7)–C(6)–C(5)	124·4 (2)	122·9 (2)
C(8)–C(7)–C(6)	112·7 (2)	114·5 (2)
O(14)–C(7)–C(6)	110·5 (2)	110·2 (2)
O(14)–C(7)–C(8)	105·7 (2)	105·2 (2)
C(9)–C(8)–C(7)	130·3 (2)	131·4 (2)
C(10)–C(9)–C(8)	134·1 (2)	133·6 (2)
C(11)–C(10)–C(9)	134·1 (2)	132·1 (3)
C(12)–C(11)–C(10)	133·4 (2)	130·3 (3)
C(11)–C(12)–C(1)	119·1 (2)	120·2 (2)
C(13)–C(12)–C(1)	118·3 (2)	118·3 (2)
C(13)–C(12)–C(11)	122·2 (2)	121·4 (2)
C(5)–C(13)–C(4)	120·0 (2)	121·0 (2)
C(12)–C(13)–C(4)	119·2 (2)	119·2 (2)
C(12)–C(13)–C(5)	120·7 (2)	119·6 (2)

(*c*) Endocyclic torsion angles for the nine-membered ring (°)

	Molecule *A*	Molecule *B*
C(12)–C(13)–C(5)–C(6)	72·6 (5)	−75·4 (5)
C(13)–C(5)–C(6)–C(7)	0·9 (6)	−3·7 (6)
C(5)–C(6)–C(7)–C(8)	−103·1 (6)	99·9 (6)
C(6)–C(7)–C(8)–C(9)	47·4 (7)	−41·1 (7)
C(7)–C(8)–C(9)–C(10)	1·4 (8)	2·1 (8)
C(8)–C(9)–C(10)–C(11)	30·6 (7)	−40·5 (7)
C(9)–C(10)–C(11)–C(12)	−2·1 (6)	1·5 (6)
C(10)–C(11)–C(12)–C(13)	−73·3 (6)	78·1 (6)
C(11)–C(12)–C(13)–C(5)	11·6 (5)	−9·5 (5)

The hydroxyl group is *exo* to the nine-membered ring, confirming the results of a recent NMR study in solution (Anastassiou, 1983). The phenyl rings are planar (r.m.s. deviation from planarity 0·011 Å for molecule *A* and 0·006 Å for molecule *B*), with normal bond lengths and angles.

Analysis of the thermal-motion tensors in terms of the rigid-body model (Schomaker & Trueblood, 1968) results in reasonable agreement between the observed and calculated tensor components [$\langle(\Delta U_{ij}^2)\rangle^{1/2} =$ 0·0034 Å^2, $\langle\sigma(U_{ij})\rangle = 0{\cdot}0014$ Å^2; both molecules]. Bond lengths corrected for libration are listed in Table 2. Librational corrections to the bond angles are negligible.

An examination of intermolecular contact distances indicates that the crystal structure is built up of $C_{13}H_{12}O$ units linked into tetramers by O···O′ hydrogen bonds [O···O′ distances 2·714 (5) and 2·740 (5) Å; O—H···O′ angle 163 (2) and O···H′—O′ angle 158 (2)°]. The hydrogen-bonding scheme is incorporated in Fig. 2. With the exception of these O···O distances, all intermolecular contacts are of the normal van der Waals type.

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The Structure of Monacolin K,* $C_{24}H_{36}O_5$

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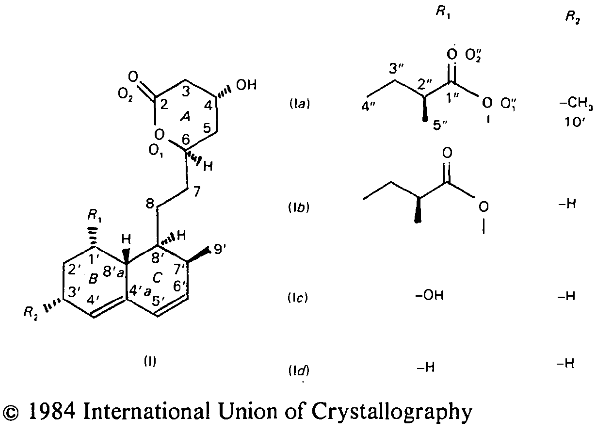
Abstract. $M_r = 404{\cdot}5$, orthorhombic, space group $P2_12_12_1$, $a = 22{\cdot}154$ (4), $b = 17{\cdot}321$ (3), $c = 5{\cdot}968$ (1) Å, $V = 2290{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}17$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}1$ cm^{-1}, $F(000) = 880$, $T = 297$ K; $R = 0{\cdot}088$, $wR = 0{\cdot}082$ for 1515 observed reflections. The six-membered rings of the hexahydronaphthalene system containing a *transoid* conjugated diene are in a distorted half-chair form with an envelope conformation.

Introduction. In our continuing studies to detect specific inhibitors of cholesterol synthesis produced by microorganisms, the metabolite monacolin K (I*a*) was isolated from the culture of *Monascus ruber*. Some compounds (I*b,c,d*) isolated previously from the strains of *Penicillium citrium* had been recognized to have a similar biological activity to that of monacolin K (Endo, Kuroda, Tsujita, Terahara & Tamura, 1974, 1976).



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* (1*S*, 3*R*, 7*S*, 8*S*, 8a*R*)-1,2,3,7,8,8a-Hexahydro-3,7-dimethyl-8-{2-[(4*R*, 6*R*)-3,4,5,6-tetrahydro-4-hydroxy-2-oxo-2*H*-pyran-6-yl]-ethyl}-1-naphthyl (2*S*)-2-methylbutanoate.

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