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Structure of (±)-(1*R**,2*S**,3a*R**,7a*R**)-Perhydro-1,2-indenediol, $C_9H_{16}O_2$

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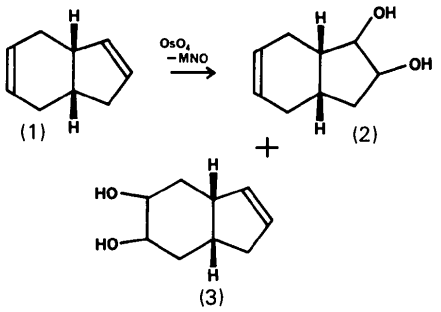
Abstract. $M_r = 156{\cdot}2$, triclinic, $P\bar{1}$, $a = 11{\cdot}368\,(3)$, $b = 14{\cdot}833\,(4)$, $c = 5{\cdot}319\,(1)$ Å, $\alpha = 95{\cdot}76\,(1)$, $\beta = 89{\cdot}98\,(1)$, $\gamma = 106{\cdot}13\,(1)°$, $V = 856{\cdot}8\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}21$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 0{\cdot}78$ mm^{-1}, $F(000) = 344$, $T = 295$ K, $R = 0{\cdot}046$ based on 2436 reflections. All the hydroxyl groups form intermolecular hydrogen bonds, but not intramolecular ones. The cyclohexane and cyclopentane rings respectively adopt the chair and half-chair forms, though they are somewhat distorted. There are no unusual bond distances or angles.

Introduction. Catalytic *cis*-hydroxylation of (1) by OsO_4 with *N*-methylmorpholine *N*-oxide (MNO) yields (2) and (3) in a product ratio of 3:1 (Matoba, Ohnishi, Kagohashi, Ishii & Ogawa, 1983).



A ^{13}C NMR study has revealed that hydrogenation of (2) on Pd–carbon gives a single component of the two diastereomers obtainable for *cis*-perhydro-1,2-indenediol (*syn* or *anti* configurations). The structure determination of the product was carried out to elucidate its stereochemistry and the mechanism of the hydroxylation.

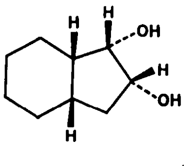


Table 1. *Atomic coordinates* (×10^4) *and isotropic temperature factors* (Å^2 × 10^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Molecule *A*				
C(1)	6742 (2)	3119 (1)	4658 (4)	323 (5)
C(2)	7900 (2)	3935 (1)	4486 (3)	270 (4)
C(3)	8750 (1)	3516 (1)	2755 (3)	257 (4)
C(3a)	8279 (1)	2448 (1)	2750 (3)	258 (4)
C(4)	8742 (2)	2074 (1)	5005 (3)	316 (5)
C(5)	8023 (2)	1052 (1)	5251 (4)	407 (6)
C(6)	6673 (2)	968 (1)	5533 (4)	397 (6)
C(7)	6147 (2)	1318 (1)	3306 (4)	364 (5)
C(7a)	6889 (1)	2305 (1)	2764 (3)	293 (5)
O(10)	8415 (1)	4258 (1)	6976 (2)	325 (3)
O(11)	10027 (1)	3918 (1)	3401 (2)	299 (3)
Molecule *B*				
C(1′)	5922 (2)	6592 (1)	10440 (4)	358 (5)
C(2′)	6628 (2)	5864 (1)	10695 (3)	306 (5)
C(3′)	7768 (1)	6402 (1)	12401 (3)	279 (5)
C(3′a)	7897 (1)	7447 (1)	12298 (3)	274 (4)
C(4′)	8581 (2)	7850 (1)	10004 (4)	334 (5)
C(5′)	8442 (2)	8829 (1)	9721 (4)	438 (6)
C(6′)	7102 (2)	8788 (1)	9418 (4)	435 (6)
C(7′)	6375 (2)	8400 (1)	11659 (4)	384 (6)
C(7′a)	6556 (2)	7462 (1)	12259 (3)	302 (5)
O(10′)	6950 (1)	5553 (1)	8240 (2)	405 (4)
O(11′)	8838 (1)	6101 (1)	11804 (2)	353 (4)

Experimental. Columnar colorless crystals (m.p. 397–399 K) obtained from ethanol solution. D_m not determined. Approximate dimensions 0·3 × 0·3 × 0·2 mm. Rigaku AFC-5UD diffractometer, graphite monochromator. Cell parameters determined by least squares from 2θ values for 20 reflections ($35 \le 2\theta \le 45°$). Intensities measured up to $2\theta = 130°$ in h −13~13, k −17~17 and l 0~6. Three standard reflections showed no significant variation. 2923 unique reflections measured, 2600 observed [$|F_o| > \sigma_1(F_o)$, where $\sigma_1(F_o)$ is the e.s.d. due to counting errors]. No correction for absorption. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located on a difference electron density map. Positional parameters for all atoms and anisotropic thermal parameters for non-H atoms refined by

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Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)–C(2)	1·531 (3)	1·530 (3)	C(3)–C(3a)	1·525 (2)	1·522 (2)	C(4)–C(5)	1·528 (3)	1·526 (3)
C(1)–C(7a)	1·539 (3)	1·534 (3)	C(3)–O(11)	1·435 (2)	1·433 (2)	C(5)–C(6)	1·513 (3)	1·516 (3)
C(2)–C(3)	1·545 (3)	1·556 (3)	C(3a)–C(4)	1·523 (3)	1·529 (3)	C(6)–C(7)	1·525 (3)	1·519 (3)
C(2)–O(10)	1·430 (3)	1·423 (3)	C(3a)–C(7a)	1·535 (2)	1·531 (3)	C(7)–C(7a)	1·529 (3)	1·526 (3)
C(2)–C(1)–C(7a)	106·1 (2)	106·5 (2)	C(3a)–C(3)–O(11)	115·5 (1)	116·0 (1)	C(5)–C(6)–C(7)	111·7 (2)	111·4 (2)
C(1)–C(2)–C(3)	105·0 (2)	104·6 (2)	C(3)–C(3a)–C(4)	113·7 (1)	113·8 (1)	C(6)–C(7)–C(7a)	112·9 (2)	113·1 (2)
C(1)–C(2)–O(10)	108·7 (2)	108·5 (2)	C(3)–C(3a)–C(7a)	101·2 (1)	101·7 (1)	C(1)–C(7a)–C(3a)	103·6 (1)	103·5 (2)
C(3)–C(2)–O(10)	112·7 (2)	112·5 (2)	C(4)–C(3a)–C(7a)	112·4 (1)	112·4 (1)	C(1)–C(7a)–C(7)	115·4 (2)	116·4 (2)
C(2)–C(3)–C(3a)	106·2 (1)	105·9 (1)	C(3a)–C(4)–C(5)	111·5 (2)	110·8 (2)	C(3a)–C(7a)–C(7)	114·2 (1)	113·9 (2)
C(2)–C(3)–O(11)	113·5 (1)	113·5 (1)	C(4)–C(5)–C(6)	110·6 (2)	110·5 (2)			

block-diagonal least squares. Temperature factor of each H equal to B_{eq} of attached non-H atom. $R = 0{\cdot}046$, $wR = 0{\cdot}055$, $S = 1{\cdot}11$ for 2436 reflections with $w \neq 0$. $\sum(w|\Delta F|^2)$ minimized, $w = 1/\sigma^2(F_o)$ for observed reflections with $|F_c| \geq \sigma(F_o)$ and $|\Delta F| < 3\sigma(F_o)$, $w = 0$ otherwise, $\sigma(F_o) = [\sigma_1^2(F_o) + 0{\cdot}00124|F_o|^2]^{1/2}$. Final $\Delta/\sigma < 0{\cdot}5$. Max. $\Delta\rho$ excursions in final difference map $0{\cdot}2$ e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i =$ 1–4) (*International Tables for X-ray Crystallography*, 1974). Calculation carried out on a FACOM M-150F computer at Shionogi Research Laboratories.

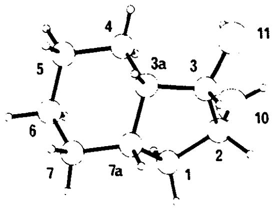

Fig. 1. Perspective view of molecule *A* showing the crystallographic numbering scheme (which differs from that used to name the compound).

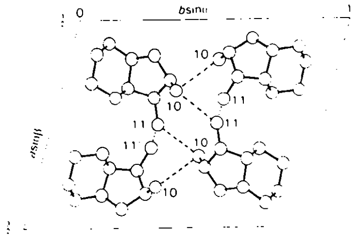

Fig. 2. Crystal structure viewed down the *c* axis. Broken and dotted lines represent intermolecular hydrogen bonds.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.† Bond lengths and angles are given in Table 2. The two independent molecules in an asymmetric unit are referred to as molecule *A* and molecule *B*. Each atom in molecule *B* is allotted a primed number of the corresponding atom in molecule *A*. The structure of molecule *B* is almost the same as that of molecule *A* for which the perspective view is shown in Fig. 1. The relative configuration is 1*R**,2*S**,3a*R**,7a*R**. The cyclohexane ring is in a slightly distorted chair form and the cyclopentane ring in a twisted half-chair form.

The crystal structure is shown in Fig. 2. All the hydroxyl groups participate in the intermolecular hydrogen bonds O(10)···O(11)$(2-x,1-y,1-z)$ [2·818 (2) Å], O(10)···O(10′)(x,y,z) [2·904 (2) Å], O(11)···O(11′)$(2-x,1-y,1-z)$ [3·055 (2) Å], and O(11)···O(11′)$(2-x,1-y,2-z)$ [2·865 (2) Å]. The latter two bonds, represented by dotted lines in Fig. 2, form an infinite zigzagging chain extending along the *c* axis. No intramolecular hydrogen bonds are formed.

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† Lists of anisotropic thermal parameters, positional parameters of H atoms, torsion angles necessary to describe the ring conformations, and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39529 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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