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Structure of (1*R*,3*R*)- and (1*S*,3*S*)-1-(1-Methylindol-2-yl)-1,3-diphenylbutane-1,3-diol

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Abstract. $C_{25}H_{25}NO_2$, $M_r = 371\cdot478$, monoclinic, Pc, $a = 8\cdot3906\ (4)$, $b = 13\cdot4356\ (8)$, $c = 8\cdot9449\ (3)$ Å, $\beta = 91\cdot518\ (7)°$, $V = 1008\cdot03\ (8)$ Å^3, $Z = 2$, $D_x = 1\cdot2239$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1\cdot5418$ Å, $\mu = 5\cdot698$ cm^{-1}, $F(000) = 396$, room temperature, $R = 0\cdot042$ for 1498 reflections with $I > 2\sigma(I)$. The lattice cell comprises a pair of the *R,R* and *S,S* enantiomers with the planes of the two phenyl rings of each stereoisomer orientated at *ca* 90° to the coplanar structure of the indole ring and attached carbon chain. Intramolecular hydrogen bonding is evident in the crystal structure between the proton of the 1-hydroxy group and the O atom of the 3-hydroxy group within each enantiomer, whilst the enantiomeric pairs are linked by weak hydrogen bonds between the proton of the 3-hydroxy group of one stereoisomer and the O atom of the 1-hydroxy group of the second isomer.

Experimental. An enantiomeric mixture of the (*S,S*)- and (*R,R*)-diols was obtained as a by-product of the reaction of 1-methylindol-2-yl lithium and acetophenone (Jones, Fresneda, Aznar-Saliente & Sepulveda-Arques, 1984). A transparent, colourless, prismatic crystal of the racemate was obtained from cyclohexane, with dimensions of *ca* 0·40 × 0·13 × 0·08 mm. Intensities were measured on a Philips PW1100 diffractometer, with graphite-monochromated Cu $K\alpha$ radiation, with $\omega/2\theta$ scans and 1·5° width. Lattice parameters were obtained from a least-squares fit of the angular 2θ positions of 40 reflections, with $\theta < 45°$. Of the 1724 independent data, 1498 were considered as

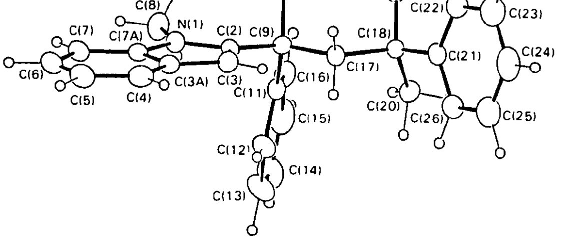

Fig. 1. *ORTEP*II (Johnson, 1976) view of the *R,R* stereoisomer showing atomic numbering.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}$ trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
N(1)	0·37040	0·28494 (25)	−0·06550	0·039 (2)
C(2)	0·46171 (62)	0·25181 (30)	0·05606 (56)	0·036 (2)
C(3)	0·61426 (68)	0·28537 (34)	0·04453 (61)	0·043 (2)
C(3A)	0·62060 (67)	0·34245 (31)	−0·09027 (60)	0·041 (2)
C(4)	0·74212 (77)	0·39593 (39)	−0·15992 (65)	0·056 (3)
C(5)	0·70390 (91)	0·44532 (41)	−0·29157 (72)	0·065 (4)
C(6)	0·55342 (91)	0·44242 (38)	−0·35577 (70)	0·062 (3)
C(7)	0·43003 (78)	0·39037 (33)	−0·28897 (66)	0·051 (2)
C(7A)	0·46787 (68)	0·34112 (30)	−0·15543 (60)	0·041 (3)
C(8)	0·20426 (75)	0·26377 (48)	−0·10025 (72)	0·056 (3)
C(9)	0·38748 (60)	0·19414 (29)	0·18377 (56)	0·035 (2)
O(10)	0·32567 (55)	0·10262 (22)	0·12242 (49)	0·042 (2)
C(11)	0·25359 (64)	0·25613 (32)	0·25012 (58)	0·039 (2)
C(12)	0·28227 (78)	0·35417 (36)	0·29170 (67)	0·053 (3)
C(13)	0·16454 (102)	0·41035 (48)	0·35667 (86)	0·079 (4)
C(14)	0·01739 (96)	0·36849 (65)	0·38180 (88)	0·084 (4)
C(15)	−0·01158 (74)	0·27271 (63)	0·34147 (77)	0·074 (4)
C(16)	0·10399 (65)	0·21566 (44)	0·27439 (64)	0·054 (3)
C(17)	0·52102 (61)	0·17116 (32)	0·30133 (58)	0·036 (2)
C(18)	0·48489 (64)	0·10395 (29)	0·43686 (56)	0·036 (2)
O(19)	0·42131 (57)	0·01298 (23)	0·37432 (50)	0·046 (2)
C(20)	0·36731 (70)	0·14704 (36)	0·54567 (59)	0·044 (3)
C(21)	0·64596 (67)	0·08614 (33)	0·51557 (58)	0·041 (2)
C(22)	0·73524 (82)	0·00162 (46)	0·48564 (68)	0·062 (3)
C(23)	0·88396 (95)	−0·01047 (62)	0·55267 (85)	0·080 (4)
C(24)	0·94771 (81)	0·05867 (56)	0·64703 (82)	0·072 (4)
C(25)	0·86092 (79)	0·14254 (48)	0·67826 (78)	0·065 (3)
C(26)	0·71221 (72)	0·15600 (39)	0·61290 (67)	0·051 (3)

observed within a $2\sigma(I)$ criterion. $2\theta_{max} = 65°$, range of *hkl*: *h* 0→9, *k* 0→15, *l* −10→10. No intensity variation of standard reflection. No correction for absorption. The structure was solved by direct methods, using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined on F_o by one-block full-matrix least-squares methods (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). All H atoms were located in a difference synthesis and refined isotropically. Weights were chosen empirically to optimize the dependence of $\langle w\Delta^2F\rangle$ *vs* $\langle |F_o| \rangle$ and $\langle(\sin\theta)/\lambda\rangle$. The final *R* and *wR* factors were 0·042 and 0·050 and the final difference synthesis showed no peaks above 0·14 e Å^{-3}. $(\Delta/\sigma)_{max} = 0{\cdot}07$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Table 1 gives the final atom coordinates and Table 2 lists selected bond distances, angles, and torsion angles.* Fig. 1 shows the projection of the *R,R* isomer of the molecule and the atomic numbering scheme, and Table 3 provides the evidence for intramolecular and intermolecular hydrogen bonding.

Table 2. *Selected bond lengths* (Å), *angles* (°), *and torsion angles* (°) *for the R,R stereoisomer*

N(1)—C(2)	1·387 (5)	C(9)—C(11)	1·531 (7)
N(1)—C(8)	1·448 (6)	C(9)—C(17)	1·547 (7)
N(1)—C(7A)	1·386 (6)	C(9)—O(10)	1·438 (5)
C(7A)—C(3A)	1·394 (8)	C(17)—C(18)	1·548 (7)
C(3)—C(3A)	1·431 (7)	C(18)—O(19)	1·441 (6)
C(3)—C(2)	1·364 (8)	C(18)—C(20)	1·519 (8)
C(2)—C(9)	1·527 (7)	C(18)—C(21)	1·526 (8)

C(7)—C(7A)—N(1)	129·1 (5)	C(2)—C(9)—C(17)	107·9 (4)
C(4)—C(3A)—C(3)	133·7 (5)	C(2)—C(9)—O(10)	107·4 (4)
C(7A)—N(1)—C(8)	124·2 (3)	C(11)—C(9)—C(17)	111·8 (4)
C(8)—N(1)—C(2)	127·9 (3)	C(11)—C(9)—O(10)	110·6 (4)
N(1)—C(2)—C(9)	121·5 (4)	O(10)—C(9)—C(17)	109·6 (3)
C(3)—C(2)—C(9)	128·8 (5)	C(9)—C(17)—C(18)	119·7 (4)
C(2)—C(3)—C(3A)	107·3 (5)	C(17)—C(18)—O(19)	105·6 (4)
C(3)—C(3A)—C(7A)	106·9 (5)	C(17)—C(18)—C(20)	115·1 (4)
C(3A)—C(7A)—N(1)	108·3 (4)	C(17)—C(18)—C(21)	105·3 (4)
C(7A)—N(1)—C(2)	107·9 (3)	C(20)—C(18)—C(21)	110·3 (4)
N(1)—C(2)—C(2)	109·6 (4)	C(20)—C(18)—O(19)	109·4 (4)
C(2)—C(9)—C(11)	109·4 (4)		

C(1)—C(2)—C(9)—O(10)	+63·2 (5)	C(9)—C(17)—C(18)—O(19)	+54·8 (5)
C(1)—C(2)—C(9)—C(17)	−178·7 (4)	C(9)—C(17)—C(18)—C(20)	−65·9 (6)
C(1)—C(2)—C(9)—C(11)	−56·9 (5)	C(9)—C(17)—C(18)—C(21)	+172·5 (4)
C(12)—C(11)—C(9)—C(2)	−50·8 (6)	C(22)—C(21)—C(18)—C(17)	−95·0 (6)
C(12)—C(11)—C(9)—O(10)	−168·9 (5)	C(22)—C(21)—C(18)—O(19)	+18·8 (7)
C(12)—C(11)—C(9)—C(17)	+68·6 (6)	C(22)—C(21)—C(18)—C(20)	+140·3 (5)
C(18)—C(17)—C(9)—C(2)	−173·9 (4)	O(10)—C(9)/C(18)—O(19)	−0·4 (4)
C(18)—C(17)—C(9)—O(10)	−57·3 (6)	C(11)—C(9)/C(18)—C(20)	+1·5 (4)
C(18)—C(17)—C(9)—C(11)	+65·8 (6)	C(2)—C(9)/C(18)—C(21)	−1·6 (7)

Table 3. *Intramolecular and intermolecular hydrogen-bond distances* (Å) *and angles* (°)

Intramolecular hydrogen bond			
O(10)—H(10)	0·89 (4)	O(10)···O(19)	2·660 (6)
H(10)···O(19)	1·83 (4)	O(10)—H(10)···O(19)	154 (4)
Intermolecular hydrogen bond			
O(19)—H(19)	0·86 (6)	O(19)···(10′)	2·842 (6)
H(19)···O(19′)	2·01 (4)	O(19)—H(19)···O(10′)	170 (6)

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* Lists of structure factors, anisotropic thermal parameters, and atomic coordinates for the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44470 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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