Table 5. *Hydrogen-bond* (O—H···O′) *distances* (Å)

The hydrogen positions are calculated with H—O 1·08 Å and H—O—C 109°. The average e.s.d. for O—O′ is 0·01 Å.

O	O′	O—O′	H—O′	H—O[(a)]	x	y	z
O(1)*A*	O(2)*A*	2·84	1·85	1·84	0·53	0·19	0·05
O(2)*A*	O(2)*B*	2·75	1·78	1·93	0·52	0·72	−0·22
O(2)*B*	O(1)*B*	2·77	1·77	1·77	0·50	1·19	0·39
O(1)*B*	O(3)*B*	2·79	1·81	1·96	0·54	0·66	0·46
O(3)*B*	O(3)*A*	2·73	1·75	1·73	0·49	1·12	0·71
O(3)*A*	O(1)*A*	2·79	1·86	1·84	0·51	0·62	0·09

(*a*) Values corresponding to the alternative, less probable, hydrogen-bond system.

OH groups are involved in hydrogen bonding (Table 5). The positions of the six H atoms of the OH groups were not clearly indicated as individual peaks in the difference Fourier map but their expected positions were at positive electron densities. The expected positions for the six H atoms (Table 5) were obtained by rotating the hydroxyls about their C—O bonds and examining neighbouring interactions. One other possibility for the hydrogen-bond system can be created by substituting O′—H···O for O—H···O′ (Table 5). However, this possibility corresponds to lower electron density at the sites of H, and two of the H···O distances are too long compared with their O—O′ distances. It is notable that even if the conformations of the two independent molecules were equal, the space group could not be transformed into *A*2 without assumption of disorder in the hydrogen-bond system.

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Three Crystal Structures of 1*β*-(*p*-Methoxybenzyl)-9*α*,10*β*-dihydroxydecahydroisoquinoline and 1*β*-(*p*-Methoxybenzyl)-9*β*,10*α*-dihydroxydecahydroisoquinoline, $C_{17}H_{25}NO_3$*

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The first isomer (9α,10β) of the title compound has two crystal forms: (I*A*) is orthorhombic, $Pna2_1$, a = 8·571 (4), b = 30·754 (2), c = 5·957 (2) Å, Z = 4; and (I*B*) is orthorhombic, $P2_12_12_1$, a = 8·730 (5), b = 30·094 (4), c = 5·950 (2) Å, Z = 4. Crystals of the 9β,10α isomer (II) are triclinic, $P\bar{1}$, a = 9·467 (3), b = 14·132 (3), c = 5·983 (3) Å, α = 101·93 (4), β = 94·18 (4), γ = 85·05 (4)°, Z = 2. Their structures have been refined to R = 0·048, 0·069, 0·041 for the 928, 1259, 2517 observed reflexions of (I*A*), (I*B*), and (II) respectively. In each of the three structures, the isoquinoline consists of two *trans*-fused six-membered rings in the chair form, and the methoxybenzyl is nearly planar. The latter is axial to the isoquinoline in (I*A*) and (I*B*), and equatorial in (II). The terminal methyl group is $\rangle\!\text{C—O}^{\diagup\text{CH}_3}$ in (I*A*) and $\rangle\!\text{C—O}_{\diagdown\text{CH}_3}$ in (I*B*), with approximately 180° rotation about the $\rangle$C—O bond and a slight shift of O in the plane of the aromatic ring. Each molecule has an intramolecular O—H···N bond, and the molecules in each structure are interlinked by O—H···O bonds into endless chains parallel to c, the shortest axis.

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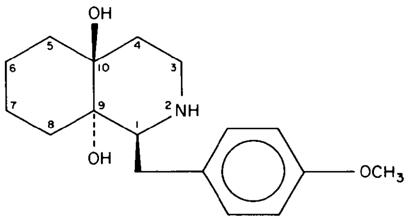
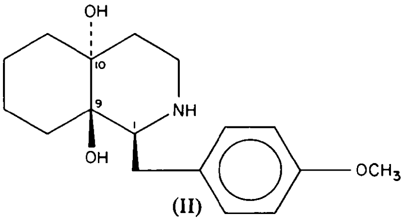
Introduction

Two crystal samples [1*β*-(*p*-methoxybenzyl)-9*α*,10*β*-dihydroxydecahydroisoquinoline (I) and 1*β*-(*p*-methoxybenzyl)-9*β*,10*α*-dihydroxydecahydroisoquinoline (II), $C_{17}H_{25}NO_3$] were supplied by I. Monkovic of the Bristol Laboratories of Canada for an X-ray study of their molecular conformations. The structure of (I) is of particular interest since it is an important intermediate in the synthesis of 3,14-dihydroxymorphinans; (II) was studied for comparison since it is closely related to (I).



(I)



(II)

Sample (I) contained transparent colourless needles in two crystal forms, both orthorhombic but with slightly different cell dimensions and in different space groups: (I*A*) in $Pna2_1$; (I*B*) in $P2_12_12_1$. Crystals of (II) were thick, transparent, colourless plates in $P\bar{1}$. All three crystal structures have been determined, and are reported in this paper.

Experimental

Crystal data for the three structures are summarized in Table 1. The space groups were identified from the systematic absences in precession photographs. However, for the (I*A*) crystals, four (out of a possible 25) weak and diffuse *h*0*l* reflexions with *h* odd were present in violation of the systematic absences, and were ignored. Similarly, for the (I*B*) crystals three very weak and diffuse spots appearing near the (300), (500) and (700) lattice sites, contrary to the space-group requirements, were also ignored. There were no such anomalies in photographs of the type (II) crystals.

The cell parameters were derived from the angular settings of some high-order axial reflexions measured at 2θ and -2θ. The densities were obtained by flotation in aqueous KI solution at 20 °C for (I*A*) and (I*B*), and at 24 °C for (II). Intensities were collected on a Picker diffractometer by the θ–2θ scan method with filtered Cu radiation. 2θ was scanned over 2·0° for $2\theta < 80°$, 2·4° for $2\theta = 80$–100°, and 3·0° for $2\theta = 100$–129°, at a speed of 2° min^{-1}. Backgrounds were measured for 10 s at the upper 2θ limit of each scan. Two monitor reflexions were measured at frequent intervals for scaling of each data set. The net intensities were corrected for Lorentz and polarization effects, but not for absorption. In (II), the 18 strongest intensities appeared to be affected by extinction, and were corrected near the end of the refinement by factors of 1·02–1·40 which were derived from a plot of I_c/I_o *versus* I_o.

Structure determination

The phases were estimated with the tangent formula (Karle & Hauptman, 1956) for the noncentrosymmetric structures (I*A*) and (I*B*), and the symbolic addition procedure (Karle & Karle, 1963) for (II). The *E* maps included 170 reflexions ($|E| \geq 1·50$) for (I*A*), 160 reflexions ($|E| \geq 1·50$) for (I*B*), and 522 reflexions ($|E| \geq 1·20$) for (II). The maps for (I*A*) and (II) contained the correct structures, but that of (I*B*) gave two molecular images separated by a *meta* vector nearly parallel to **a**. For the latter, only 14 atomic positions were accepted from the trial structure, which was assumed to be the average of the two images (Huber, 1975); the remaining seven non-hydrogen atoms were determined from a Fourier map. The H atoms were located from difference maps after partial refinement of the other atoms.

The refinement, based solely on the observed reflexions, was by block-diagonal least squares, mini-

Table 1. *Crystal data of* $C_{17}H_{25}NO_3$ ($M_r = 291·39$)

	(I*A*)	(I*B*)	(II)
Space group	$Pna2_1$	$P2_12_12_1$	$P1$
a (Å)	8·571 (4)	8·730 (5)	9·467 (3)
b (Å)	30·754 (2)	30·094 (4)	14·132 (3)
c (Å)	5·957 (2)	5·950 (2)	5·983 (3)
α (°)			101·93 (4)
β (°)			94·18 (4)
γ (°)			85·05 (4)
V (Å^3)	1570·2	1563·2	779·0
Z	4	4	2
d_c (g cm^{-3})	1·232	1·238	1·242
d_m (g cm^{-3})	1·232	1·238	1·241
μ(Cu $K\alpha$) (cm^{-1})	6·345	6·376	6·397
Dimensions (mm)	0·4 × 0·1 × 0·07	0·4 × 0·2 × 0·3	0·6 × 0·3 × 0·4
Measured reflexions	1448	1564	2611
Observed reflexions	928	1259	2517
R for observed reflexions	0·048	0·069*	0·041
R_w	0·053	0·080	0·041
$[\sum w(\Delta F)^2/(m-n)]^{1/2}$	0·89	1·24	0·34
Mean $[\Delta(p)/\sigma(p)]$	0·22	0·16	0·08
Maximum $[\Delta(p)/\sigma(p)]$	1·39†	0·86	0·59

* R = 0·067 for the 1255 reflexions included in the least-squares refinement.

† The maximum $\Delta(p)/\sigma(p)$ for C, N, O is 0·83.

Table 2. *Atomic positional parameters* ($\times 10^4$, *for* H $\times$ 10^3)

	(I*A*)			(I*B*)			(II)		
	x	*y*	*z*	*x*	*y*	*z*	*x*	*y*	*z*
O(1)	2790(4)	852(1)	4697(0)	6866(5)	1921(1)	11529(6)	3968(1)	2214(1)	-511(2)
O(2)	1398(4)	715(1)	10402(7)	7617(5)	1573(1)	5755(6)	2715(1)	2172(1)	5062(2)
O(3)	-5361(5)	2417(1)	5202(10)	-620(5)	244(2)	7285(8)	1615(1)	8127(1)	2913(2)
C(1)	409(7)	1126(2)	6167(10)	5441(7)	1379(2)	9502(9)	2788(2)	3502(1)	2140(2)
N(2)	1216(6)	1548(1)	6437(10)	4324(6)	1730(2)	9112(8)	4217(1)	3839(1)	2633(2)
C(3)	1888(8)	1602(2)	8646(14)	4552(7)	1969(2)	6949(10)	4911(2)	3529(1)	4662(3)
C(4)	3165(7)	1271(2)	9024(12)	6060(7)	2204(2)	7019(9)	5099(2)	2433(1)	4186(3)
C(5)	3824(7)	471(2)	8804(12)	8889(7)	2144(2)	7890(10)	3996(2)	861(1)	2577(3)
C(6)	3188(7)	9(2)	8508(13)	10208(7)	1838(2)	8455(11)	2642(2)	374(1)	1655(4)
C(7)	2333(8)	-26(2)	6246(13)	9846(8)	1553(2)	10547(12)	1927(2)	809(1)	-294(3)
C(8)	1031(7)	320(2)	6108(11)	8329(8)	1303(2)	10245(10)	1664(2)	1907(1)	392(3)
C(9)	1621(6)	765(2)	6439(9)	7012(7)	1610(2)	9666(8)	3009(2)	2396(1)	1324(2)
C(10)	2545(6)	807(2)	8684(11)	7391(6)	1892(2)	7536(8)	3726(2)	1956(1)	3322(2)
C(11)	-1105(7)	1076(2)	7548(12)	5336(7)	978(2)	7873(11)	1976(2)	4020(1)	383(3)
C(12)	-2288(7)	1420(2)	6897(12)	3768(8)	791(2)	7701(11)	1886(2)	5112(1)	1105(3)
C(13)	-3131(6)	1396(2)	4915(11)	3099(8)	530(2)	9437(11)	2423(2)	5692(1)	-178(3)
C(14)	-4173(7)	1713(2)	4299(12)	1666(8)	362(2)	9265(11)	2317(2)	6687(1)	468(3)
C(15)	-4390(7)	2072(2)	5683(13)	777(7)	436(2)	7349(11)	1662(2)	7139(1)	2430(3)
C(16)	-3590(8)	2096(2)	7738(12)	1354(8)	701(2)	5637(11)	1120(2)	6581(1)	3747(3)
C(17)	-2542(7)	1777(2)	8265(11)	2813(8)	868(2)	5836(11)	1239(2)	5583(1)	3066(3)
C(18)	-6254(9)	2392(3)	3206(15)	-1521(8)	292(3)	5276(13)	979(2)	8615(1)	4944(4)
H(01)	303(8)	116(2)	466(15)	568(6)	200(2)	1147(9)	450(2)	272(2)	-14(4)
H(02)	219(6)	78(1)	1174(10)	758(9)	176(3)	499(14)	313(2)	213(1)	630(3)
H(1)	9(5)	110(1)	443(9)	532(8)	127(2)	1128(13)	227(1)	361(1)	355(2)
H(2)	48(9)	181(2)	603(14)	319(6)	163(2)	916(9)	416(2)	448(1)	279(3)
H(3,1)	108(5)	156(1)	1002(8)	446(7)	176(2)	573(10)	435(2)	378(1)	608(3)
H(3,2)	231(5)	187(1)	885(8)	364(6)	220(2)	693(10)	586(2)	382(1)	495(3)
H(4,1)	346(7)	130(2)	1038(12)	586(8)	245(2)	799(13)	583(2)	220(1)	306(3)
H(4,2)	401(7)	128(2)	778(11)	649(6)	237(2)	556(10)	550(2)	221(1)	562(3)
H(5,1)	437(7)	51(2)	1059(13)	875(8)	237(2)	867(12)	470(2)	76(1)	140(3)
H(5,2)	450(7)	53(2)	774(12)	925(9)	232(2)	621(14)	444(2)	61(1)	388(3)
H(6,1)	240(6)	-3(2)	1011(10)	1057(7)	163(2)	693(11)	198(2)	44(1)	293(3)
H(6,2)	397(6)	-20(2)	839(11)	1133(6)	200(2)	864(10)	287(2)	-35(1)	114(3)
H(7,1)	308(7)	0(2)	508(13)	956(7)	174(2)	1185(10)	256(2)	64(1)	-166(3)
H(7,2)	188(6)	-34(2)	604(11)	1049(7)	133(2)	1075(11)	98(2)	54(1)	-82(3)
H(8,1)	55(7)	30(2)	458(11)	798(7)	111(2)	1145(10)	94(2)	208(1)	156(3)
H(8,2)	28(7)	24(2)	744(12)	828(7)	106(2)	939(11)	124(2)	219(1)	-97(3)
H(11,1)	-98(6)	106(1)	923(10)	583(8)	106(2)	613(14)	244(2)	383(1)	-106(3)
H(11,2)	-162(7)	78(2)	720(13)	612(10)	70(3)	834(16)	97(2)	381(1)	19(3)
H(13)	-285(7)	118(2)	357(13)	402(7)	44(2)	1038(11)	286(2)	540(1)	-154(3)
H(14)	-456(7)	171(2)	259(10)	122(6)	13(2)	1022(10)	273(2)	707(1)	-51(3)
H(16)	-366(9)	238(2)	893(14)	50(9)	77(3)	460(14)	67(2)	689(1)	515(3)
H(17)	-198(8)	180(2)	944(13)	293(8)	113(2)	523(12)	86(2)	519(1)	401(3)
H(18,1)	-542(5)	242(1)	172(9)	-265(7)	14(2)	531(10)	-4(2)	847(1)	497(3)
H(18,2)	-689(8)	213(2)	301(13)	-186(7)	62(2)	518(11)	148(2)	842(2)	636(4)
H(18,3)	-681(8)	268(2)	329(14)	-63(8)	17(2)	457(12)	104(2)	931(1)	497(3)

Table 3. *Bond lengths* (Å)

The e.s.d.'s of (II) may be underestimated by 50%.

	(I*A*)	(I*B*)	(II)
O(1)—C(9)	1·467 (6)	1·457 (6)	1·443 (2)
O(2)—C(10)	1·447 (7)	1·443 (6)	1·435 (2)
O(3)—C(15)	1·377 (7)	1·351 (8)	1·364 (2)
O(3)—C(18)	1·416 (11)	1·439 (9)	1·416 (3)
C(1)—N(2)	1·479 (7)	1·456 (8)	1·463 (2)
C(1)—C(9)	1·529 (8)	1·540 (9)	1·539 (2)
C(1)—C(11)	1·544 (9)	1·551 (9)	1·528 (2)
N(2)—C(3)	1·446 (10)	1·487 (8)	1·467 (2)
C(3)—C(4)	1·510 (9)	1·496 (9)	1·513 (3)
C(4)—C(10)	1·537 (8)	1·526 (8)	1·523 (2)
C(5)—C(6)	1·530 (8)	1·512 (9)	1·521 (3)
C(5)—C(10)	1·508 (8)	1·526 (8)	1·522 (2)
C(6)—C(7)	1·538 (11)	1·544 (10)	1·519 (3)
C(7)—C(8)	1·545 (9)	1·534 (10)	1·523 (3)
C(8)—C(9)	1·472 (8)	1·514 (9)	1·516 (2)
C(9)—C(10)	1·559 (8)	1·561 (7)	1·547 (2)
C(11)—C(12)	1·514 (8)	1·483 (9)	1·511 (2)
C(12)—C(13)	1·386 (9)	1·424 (9)	1·381 (2)
C(12)—C(17)	1·385 (8)	1·407 (9)	1·380 (2)
C(13)—C(14)	1·373 (9)	1·353 (9)	1·377 (3)
C(14)—C(15)	1·390 (9)	1·398 (9)	1·378 (2)
C(15)—C(16)	1·405 (10)	1·387 (9)	1·375 (2)
C(16)—C(17)	1·367 (9)	1·375 (10)	1·382 (2)
⟨O—H⟩	1·03	0·90	0·86
N—II	1·05	1·04	0·89
⟨C—H⟩	1·02	1·02	1·00

mizing $\sum w(|F_o| - |F_c|)^2$, where $w = \{1 + [(|F_o| - p_2)/p_1]^4\}^{-1}$. The p_1 and p_2 parameters, chosen to make $\langle w(\Delta F)^2\rangle$ independent of $|F_o|$, were respectively 25 and 20 for (I*A*), 27 and 17 for (I*B*), and 20 and 15 for (II).

Anisotropic thermal motion was assumed for C, N, and O atoms; H atoms were assumed to be isotropic. The agreement indices for the final cycles of the three structures are listed at the end of Table 1.

Residual electron densities in the final difference maps were in the ranges −0·15 to 0·26 e Å^{-3} for (I*A*), −0·26 to 0·31 e Å^{-3} for (I*B*), and −0·14 to 0·13 e Å^{-3} for (II). There were no large discrepancies among the observed and calculated structure amplitudes, except for four observed reflexions in (I*B*) which were excluded from the later stages of refinement. All unobserved reflexions had small $|F_c|$ values.

The scattering factors were those of Hanson, Herman, Lea & Skillman (1964) for C, N, O, and of Stewart, Davidson & Simpson (1965) for H. All calculations were carried out with the NRC System of Crystallographic Computer Programs (Ahmed, Hall, Pippy & Huber, 1973).

The positional parameters are listed in Table 2;* bond lengths and angles, not corrected for thermal vibration, are in Tables 3 and 4. The e.s.d.'s shown in Tables 3 and 4 for (II), derived from the least-squares refinement, appear to be underestimated and should be incremented by at least 50% to allow for the omission of the parameters' covariance from these calculations (Rollett, 1970).

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33525 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Valency angles* (°)

The e.s.d.'s of (II) may be underestimated by 50%.

	(I*A*)	(I*B*)	(II)
C(15)–O(3)–C(18)	117·4 (6)	118·2 (6)	117·5 (1)
N(2)–C(1)–C(9)	108·0 (5)	106·3 (5)	105·3 (1)
N(2)–C(1)–C(11)	115·0 (5)	115·1 (5)	110·1 (1)
C(9)–C(1)–C(11)	116·2 (5)	116·3 (5)	113·9 (1)
C(1)–N(2)–C(3)	112·6 (5)	113·5 (5)	111·5 (1)
N(2)–C(3)–C(4)	110·4 (5)	108·8 (5)	108·8 (1)
C(3)–C(4)–C(10)	110·8 (5)	112·6 (5)	112·9 (1)
C(6)–C(5)–C(10)	111·8 (5)	112·4 (5)	112·0 (1)
C(5)–C(6)–C(7)	109·7 (5)	111·2 (5)	110·9 (2)
C(6)–C(7)–C(8)	110·0 (5)	110·6 (6)	111·9 (2)
C(7)–C(8)–C(9)	112·7 (5)	112·5 (5)	112·2 (1)
O(1)–C(9)–C(1)	104·9 (4)	105·1 (4)	107·6 (1)
O(1)–C(9)–C(8)	107·9 (4)	106·6 (4)	107·1 (1)
O(1)–C(9)–C(10)	104·2 (4)	106·6 (4)	108·3 (1)
C(1)–C(9)–C(8)	115·3 (5)	114·6 (5)	114·2 (1)
C(1)–C(9)–C(10)	112·2 (4)	112·5 (4)	108·6 (1)
C(8)–C(9)–C(10)	111·4 (4)	110·8 (5)	110·9 (1)
O(2)–C(10)–C(4)	108·8 (4)	111·4 (4)	110·4 (1)
O(2)–C(10)–C(5)	109·1 (5)	108·3 (4)	109·7 (1)
O(2)–C(10)–C(9)	104·2 (4)	105·3 (4)	104·7 (1)
C(4)–C(10)–C(5)	112·3 (5)	112·0 (5)	111·3 (1)
C(4)–C(10)–C(9)	111·4 (5)	109·7 (4)	110·1 (1)
C(5)–C(10)–C(9)	110·7 (5)	109·9 (4)	110·4 (1)
C(1)–C(11)–C(12)	110·9 (5)	113·1 (5)	113·5 (1)
C(11)–C(12)–C(13)	122·0 (5)	122·5 (6)	121·9 (1)
C(11)–C(12)–C(17)	120·5 (5)	122·6 (6)	121·6 (1)
C(13)–C(12)–C(17)	117·4 (6)	114·8 (6)	116·6 (1)
C(12)–C(13)–C(14)	121·9 (6)	122·1 (6)	121·6 (2)
C(13)–C(14)–C(15)	119·6 (6)	121·0 (6)	120·6 (2)
O(3)–C(15)–C(14)	124·6 (6)	117·1 (6)	115·9 (1)
O(3)–C(15)–C(16)	115·8 (6)	123·6 (6)	125·0 (1)
C(14)–C(15)–C(16)	119·5 (6)	119·3 (6)	119·1 (2)
C(15)–C(16)–C(17)	118·9 (6)	118·9 (6)	119·3 (2)
C(12)–C(17)–C(16)	122·6 (6)	123·8 (6)	122·8 (2)

Results and discussion

Equivalent perspective views of the three molecular structures are presented in Fig. 1. In each molecule, the decahydroisoquinoline consists of two *trans*-fused six-membered rings in the chair conformation, while the methoxybenzyl is nearly planar. The C(1)–C(11) bond linking the two groups is axial to the isoquinoline in (I*A*) and (I*B*), and equatorial in (II). The molecules of the (I*A*) and (I*B*) crystal structures are almost identical except for a 180° rotation of the terminal O(3)–CH_3 bond about the adjoining O(3)–C(15) bond.

Bond lengths and angles

The effect of conformational differences on the equivalent bond lengths and angles has been examined by the *t*-distribution test. Significant discrepancies between the (I*A*) and (I*B*) structures occur in the N(2)–C(3) and C(8)–C(9) lengths, and the O(1)–C(9)–C(10), O(2)–C(10)–C(4), O(3)–C(15)–C(14) and O(3)–C(15)–C(16) angles. The largest of these discrepancies is in the two exocyclic angles at C(15), and appears to be a direct result of the position of the methyl group. The larger angles of 124·6 (6) and 123·6 (6)° are those on the same side of C(15)–O(3) as the methyl group, and the smaller angles of 115·8 (6) and 117·1 (6)° are on the opposite side. Similar imbalance of these exocyclic angles is also present in (II) where the angles are 125·0 (1) and 115·9 (1)°, respectively, and in the two structures reported by Cotrait, Sy & Ptak (1975) (124·8 and 115·0°), and by Koetzle & Williams (1976) (124·6 and 115·1°). The remaining significant discrepancies between (I*A*) and (I*B*) are much smaller: 0·04 Å ($t = 3·2$, $3·5$) in the bond lengths, and about 2·5° ($t = 6·0$, $6·5$) in the two angles involving the O(1) and O(2) substituents.

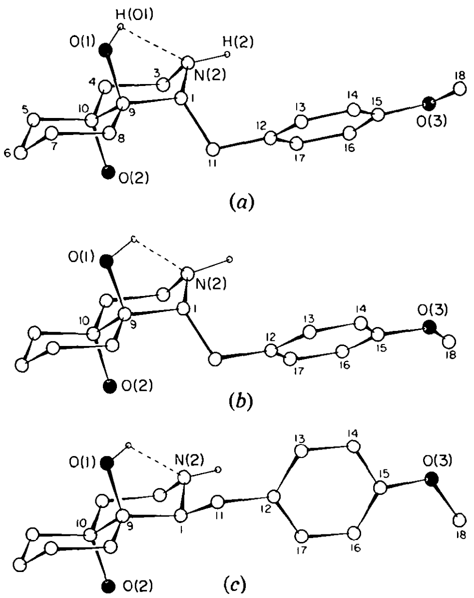

Fig. 1. Perspective drawings of the molecular structures of (*a*) (I*A*), (*b*) (I*B*) and (*c*) (II), as viewed from approximately the same direction relative to the isoquinoline group.

The largest discrepancies between (I) and (II) appear to be directly related to the orientation of C(1)–C(11) in the two structures. For this comparison, the dimensions of (I) are taken as the mean values of (I*A*) and (I*B*). Thus, in (I) where C(1)–C(11) is axial, the C(11)–C(1)–N(2) angle is 115·0° while in (II) it is only 110·1°. This change of 4·9° is accompanied by smaller but significant changes (≤3·7°) in some of the valency angles at the neighbouring C(9). Also, small but significant discrepancies (0·019–0·024 Å) are observed in the O(1)–C(9), C(8)–C(9) and C(12)–C(13) lengths; however, these discrepancies should more appropriately be considered random errors in view of the underestimated e.s.d.'s of (II).

The ranges of the bond lengths in the three structures, their weighted mean values, and the corresponding mean values given by Sutton (1965) are summarized in Table 5.

Torsion angles and mean planes

It is evident from the torsion angles listed in Table 6, and from the displacements of the atoms from the mean planes given in Table 7, that both rings of the isoquinoline are in the chair conformation and that the aromatic ring is very nearly planar in each of the three structures. While only small variations (within 2·5°) are observed in the equivalent torsion angles of (I*A*) and (I*B*), larger differences are noted in some of the corresponding angles of (II). The largest differences of 8·8 and 6·7° occur in C(10)–C(9)–C(1)–N(2) and C(9)–C(1)–N(2)–C(3), which are the two bonds closest to where the methoxybenzyl is substituted on ring (*a*). In both cases, the larger torsion angles are in (II) where C(1)–C(11) is equatorial. Also, from the details given in Table 7, it can be seen that each of the chair rings has a planar seat except for ring (*a*) of (II). Thus, ring (*a*) appears to be under more strain when the methoxybenzyl substituent is equatorial than when it is axial. It is also evident from Table 7 that C(11), O(3) and C(18) of the methoxybenzyl have preferred positions near the mean plane of the aromatic rings.

Table 5. *Ranges and weighted mean values of the bond lengths* (Å), *and the corresponding mean values of Sutton* (1965)

	Number	Range	Weighted mean	Sutton (1965)
HO—C$\leqq$	6	1·435–1·467	1·442	1·426 ± 5
—O—CH_3	3	1·416–1·439	1·418	1·426 ± 5
O—C(sp^2)	3	1·351–1·377	1·364	1·36 ± 1
N—C	6	1·446–1·487	1·466	1·472 ± 5
C(sp^3)—C(sp^3)	30	1·472–1·561	1·527	1·537 ± 5
C(sp^3)—C(sp^2)	3	1·483–1·514	1·510	1·505 ± 5
C(sp^2)—C(sp^2)	18	1·353–1·424	1·380	1·394 ± 5

Table 6. *Torsion angles* (°) *within the rings*

	(I*A*)	(I*B*)	(II)
Ring (*a*)			
C(9)–C(1)–N(2)–C(3)	−62·6	63·4	−69·7
C(1)–N(2)–C(3)–C(4)	64·7	−64·1	63·5
N(2)–C(3)–C(4)–C(10)	−56·3	55·6	−52·2
C(3)–C(4)–C(10)–C(9)	49·1	−50·6	49·9
C(4)–C(10)–C(9)–C(1)	−49·3	51·4	−55·5
C(10)–C(9)–C(1)–N(2)	54·0	−56·5	64·0
Mean $\lvert\tau\rvert$	56·0	56·9	59·1
Ring (*b*)			
C(10)–C(5)–C(6)–C(7)	57·9	−56·3	55·7
C(5)–C(6)–C(7)–C(8)	−56·3	53·9	−54·1
C(6)–C(7)–C(8)–C(9)	56·4	−54·4	54·4
C(7)–C(8)–C(9)–C(10)	−54·6	55·3	−54·5
C(8)–C(9)–C(10)–C(5)	54·2	−55·3	54·9
C(9)–C(10)–C(5)–C(6)	−55·9	56·5	−56·0
Mean $\lvert\tau\rvert$	55·9	55·3	54·9
Ring (*c*)			
C(17)–C(12)–C(13)–C(14)	0·5	1·5	0·0
C(12)–C(13)–C(14)–C(15)	0·6	0·3	−0·2
C(13)–C(14)–C(15)–C(16)	−2·8	−2·4	0·4
C(14)–C(15)–C(16)–C(17)	3·9	2·5	−0·4
C(15)–C(16)–C(17)–C(12)	−3·0	−0·7	0·2
C(16)–C(17)–C(12)–C(13)	0·8	−1·3	0·1
Mean $\lvert\tau\rvert$	1·9	1·4	0·2

Hydrogen bonds

Projections along **a** of the three crystal structures, showing the molecular orientations and the hydrogen bonds, are presented in Fig. 2. The projections for (I*A*) and (I*B*) show half the unit cell, and that for (II) is of the full unit cell. The relevant dimensions of the hydrogen bonds are also given in the figure.

In each of the three structures, there appear to be two types of hydrogen bonds: one intramolecular between O(1) and N(2), and the other intermolecular between O(2) of the molecule at (x,y,z) and O(1′) of the adjacent molecule at (x, y, $1 + z$). The latter bond links the molecules into continuous chains parallel to *c*, which is the shortest cell axis. There are no other short van der Waals contacts in any of the three structures.

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Table 7. *Deviations* (×10³ Å) *from the mean planes of the atoms marked with an asterisk*

	(I*A*)	(I*B*)	(II)
Ring (*a*)			
C(1)*	2	4	−45
N(2)	687	−690	724
C(3)*	−3	−4	46
C(4)*	2	4	−44
C(10)	−614	638	−655
C(9)*	−2	−4	43
Ring (*b*)			
C(5)*	3	−8	8
C(6)	−692	658	−653
C(7)*	−3	8	−8
C(8)*	3	−8	8
C(9)	644	−669	660
C(10)*	−3	8	−8
Methoxybenzyl			
C(11)	44	11	27
C(12)*	5	12	0
C(13)*	−5	−6	0
C(14)*	−5	−7	−1
C(15)*	17	15	2
C(16)*	−18	−9	−1
C(17)*	6	−4	0
O(3)	65	58	0
C(18)	26	166	−29
χ^2	19·4	16·0	3·6

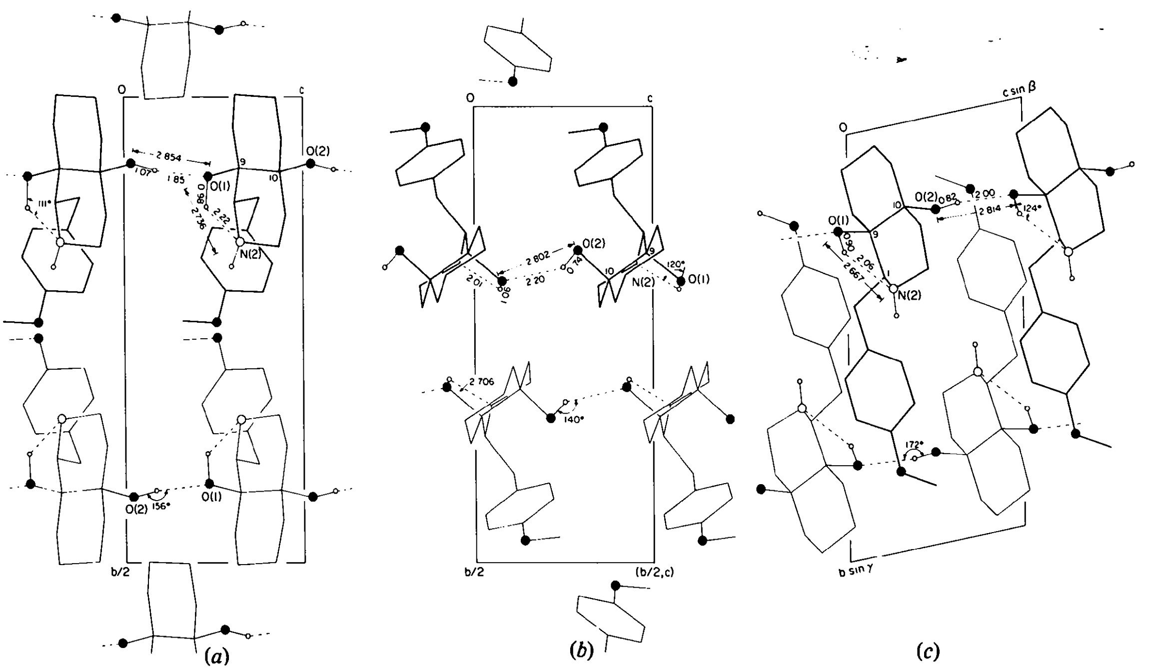

Fig. 2. Projections of (*a*) (I*A*), (*b*) (I*B*) and (*c*) (II) along a showing the molecular orientations and the hydrogen-bonding schemes. The distances are in Å, and the projections are of half the unit cells of (I*A*) and (I*B*) and of the full unit cell of (II).

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Structure Cristalline du Diperchlorate d'*o*-Phénanthroline Monohydraté: $C_{12}H_8N_2.2HClO_4.H_2O$

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Crystals of the title compound are monoclinic, space group $P2_1/b$, with $a = 11{\cdot}515$ (7), $b = 20{\cdot}12$ (1), $c = 13{\cdot}620$ (8) Å and $\gamma = 90{\cdot}05$ (5)°. There are eight formula units $[(C_{12}H_{10}N_2)^{2+}.2ClO_4^-.H_2O]$ per unit cell. Data were collected on an Enraf–Nonius CAD-4 diffractometer using Mo $K\alpha$ radiation. The crystal structure was solved by direct methods and refined by least-squares procedures to a final $R = 0{\cdot}043$ for 3230 independent reflections. Perchlorate and phenanthroline groups alternate in layers and are linked to each other and to water molecules by hydrogen bonds.