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ABSOLUTE CONFIGURATION OF CRYPTOSTYLINES I, II, AND III BY X-RAY ANALYSIS AND AROMATIC CHIRALITY METHOD

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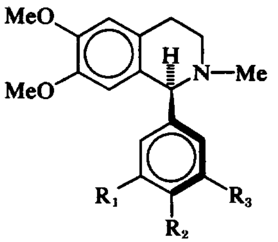
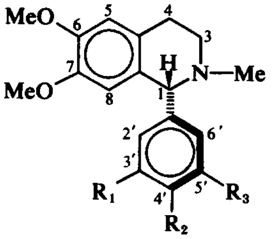
Abstract—The *S*-configuration of the alkaloids cryptostylines I, II, and III was assigned on the basis of a single crystal X-ray analysis of unnatural cryptostyline II hydrobromide (**5**.HBr: orthorhombic, $P2_12_12_1$, $a = 10{\cdot}162$, $b = 12{\cdot}352$, $c = 16{\cdot}456$ Å) and confirmed by application of the aromatic chirality method. Further evidence for the configurational assignment was provided by the CD spectra of the monophenolic derivatives of unnatural cryptostyline II (**14**).

The syntheses of the substituted 1-phenyl-tetrahydroisoquinoline alkaloids cryptostylines I, II, and III (**1**, **2**, and **3**, respectively) have been recently described and the structures assigned the *S* configuration based on a single crystal X-ray analysis.[1] We now report the details of the X-ray study and in addition present confirmation of the *S* configuration* by application of the aromatic chirality method.[3]





1: $R_1{=}H$; $R_2 + R_3{=}OCH_2O$ **4**
2: $R_1{=}H$; $R_2{=}R_3{=}OMe$ **5**
3: $R_1{=}R_2{=}R_3{=}OMe$ **6**

The crystal structure analysis of unnatural cryptostyline II hydrobromide[1] (**5**.HBr) established its absolute configuration as *R* (Fig 1). Thus, the natural isomer II (**2**) and the related alkaloids cryptostylines I and III possess the *S* configuration.

In crystalline **5**.HBr, the 3-carbon is displaced 0·58 Å above the plane (A) of the phenyl ring of the isoquinoline moiety whereas the N is 0·23 Å below this plane. All C atoms comprising the phenyl ring substituted at the 1-position are located below plane A. The angle between the 1′-4′ axis of the 1-substituent and plane A is 36°. The two aromatic rings are nearly perpendicular to each other; the angle between their normals is 84°. All four OMe groups lie approximately in the plane of their respective phenyl rings as expected when there is no steric interference.[4] The maximum displacement of the carbon of any OMe group from the plane of its phenyl ring is 0·3 Å.

The unnatural cryptostylines I, II, and III (**4**, **5**, and **6**, respectively) exhibit UV spectra[1,5] with characteristic[6] maxima or inflections at about 282–287 (^1L_b transition), 230–235 (^1L_a transition), and 205–208 nm (^1B transition)[7]. Their intensities and the positions of their maxima closely resemble the expected arithmetic sum of the two similar chromophores; the differences in the positions of the maxima are about 3–5 nm.

Analysis of the ORD spectra[1] of **4**, **5**, and **6** was rendered uncertain by the closely located overlapping Cotton effects and strong background rotations†. However, their CD spectra[1] (Fig 2) are well resolved and have extrema in the 200–220, 230–250, and 270–290 nm regions which correspond to the UV absorption maxima. In contrast to their single long wave length UV maxima, their

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*Initially, the opposite configuration was suggested by an indirect comparison with model compounds.[2]

†It is worthwhile to emphasize that the sign of the conventional $[\alpha]_D$ value does not necessarily correspond to the sign of the longest wave length Cotton effect in the ORD mode. Further, since the $[\alpha]_D$ value is influenced by solvent and pH, it should be used cautiously when assigning an absolute configuration.

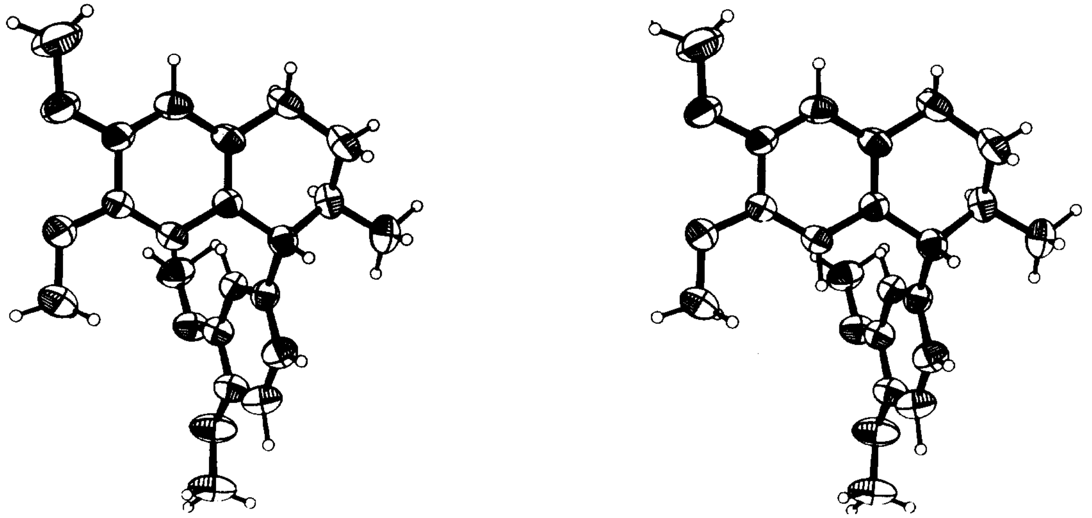


Fig 1. Stereodrawing illustrating the absolute configuration of unnatural cryptostyline II as determined from the X-ray analysis of 5.HBr.

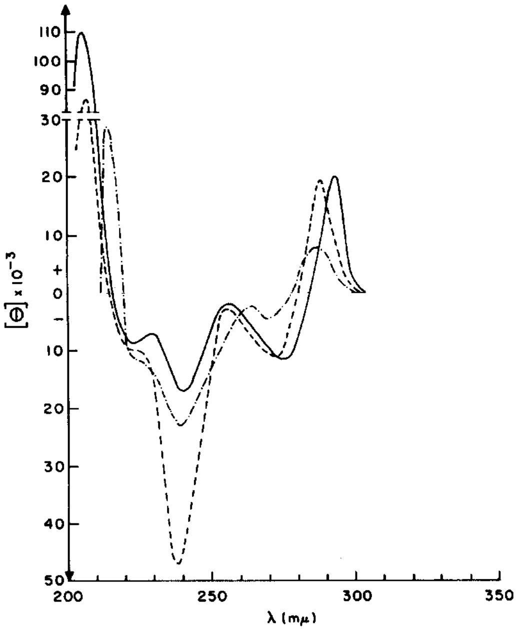


Fig 2. CD spectra of unnatural cryptostylines I (4) [———], II (5), [-----], and III (6) |—·—·—| in methanol.

CD spectra clearly show Davydov exciton splitting[8] in the ^1L_b transition band characterized by two strong extrema ($\Delta\lambda = 15$–16 nm) of about the same amplitude but opposite signs. These bands are probably derived from an electric dipole–dipole interaction between the long axis charge-transfer transition moments of the two similar non-coplanar aromatic chromophores. However, at the ^1L_a band region of 230–250 nm, the Cotton effects do not indicate an exciton splitting since these transition moments are approximately parallel[9]. Finally, although the negative CD maximum below 200 nm could not be reached because of instrumental limitation, the very strong positive extreme in the 200–210 nm region suggest that the ^1B Cotton effect also exhibits a Davydov splitting with the same sequence as shown by the first pair.

Since the first of the split Cotton effects of the ^1L_b transitions of the unnatural cryptostylines **4**, **5**, and **6** are positive, the two non-coplanar aromatic chromophores must show right-handed or positive chirality (Fig 3) on the basis of the aromatic chirality method[3]. Thus, they possess the *R* configuration in agreement with the X-ray analysis. Had there been no electric dipole–dipole interaction, the sums of the closely related Cotton effect of opposite signs would have cancelled each other with resulting weak intensities.

In connection with this study, unnatural 4′-desmethyl cryptostyline II (**14**) was synthesized. Cyclodehydration of the benzyloxy-substituted amide **9**, obtained by condensation of homoveratrylamine **7** and 4-benzyloxy-3-methoxybenzoyl chloride (**8**),[10] with phosphorus oxychloride yielded

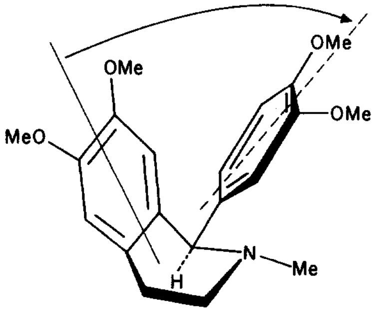
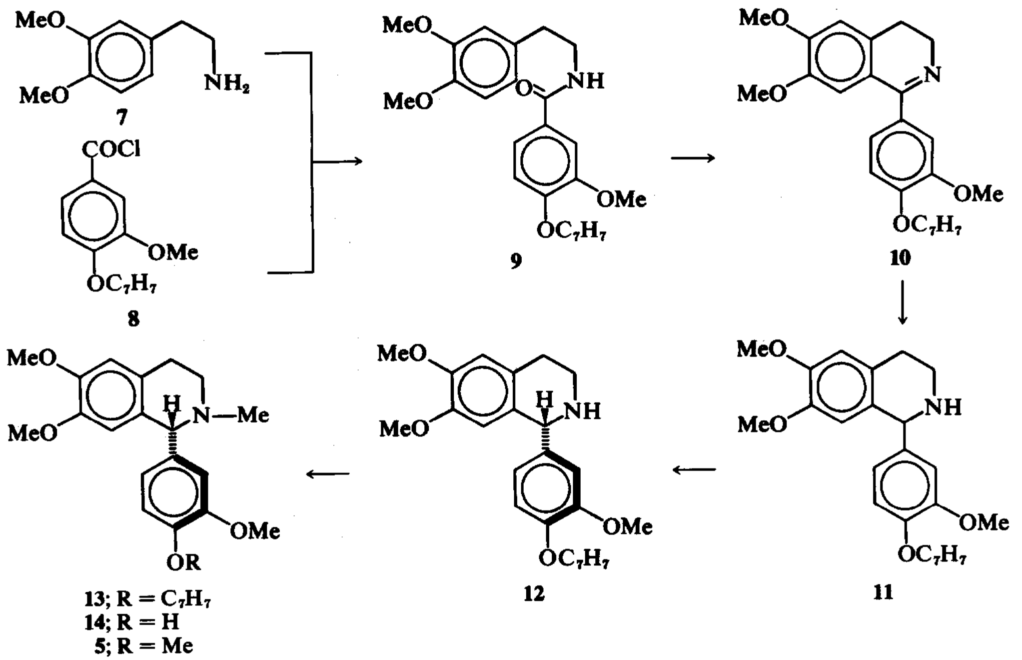


Fig 3. Positive chirality of unnatural cryptostyline II (5).

the 3,4-dihydroisoquinoline **10** which was reduced with sodium borohydride to the tetrahydroisoquinoline **11**. Resolution of **11** with (−)-diacetone-2-keto-L-gulonic acid[11] yielded the dextrorotatory isomer **12** which was reductively condensed with formaldehyde and the resulting N-methyl derivative **13** O-debenzylated with mineral acid to afford the desired monophenol **14**. The configuration of **14** was established since O-methylation with diazomethane yielded unnatural cryptostyline II (**5**).



The UV, ORD and CD spectra (Table 1) of the monophenol **14** provided additional confirmation of the configuration assignment. Once again, Davydov splitting was observed in the ^1L_b transition (Fig 4). Both the first and second extreme showed approximately the same bathochromic shift of 6–7 nm in 0·1 N KOH. This indicates a strong coupling between the long axis polarized transition moments of the veratrole and methoxy phenolate moieties. If there were no such interaction between the two alkoxy benzene transitions, only one of the two Cotton effects in the 280–300 nm region would have shifted with alkali to the longer wave length.

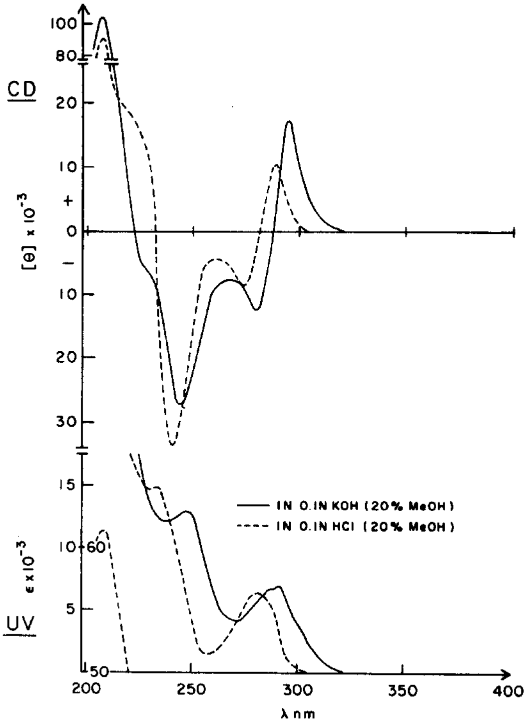


Fig 4. UV and CD curves of the monophenol **14**.

EXPERIMENTAL

Crystallography. Crystals of **5**.HBr are orthorhombic, space group $P2_12_12_1$. The crystal data are $a = 10{\cdot}162(3)$, $b = 12{\cdot}352(5)$, $c = 16{\cdot}456(4)$ Å, $d_{obs} = 1{\cdot}37$, $d_{calc} = 1{\cdot}364$ g cm^{-3} for $Z = 4$, $\mu = 31{\cdot}9$ cm^{-1} (CuKα). All intensity data were collected by θ-2θ scans on a Hilger & Watts model Y290 four circle diffractometer. Nickel filtered CuKα radiation and pulse height discrimination were used. Of the 2252 accessible reflections with $2\theta < 140°$, 1882 had intensities significantly greater than background. These data were collected from a crystal which was approximately 0·15 × 0·15 × 0·45 mm. The data were corrected for absorption[12].

Table 1. UV maxima, ORD and CD Cotton effects of the monophenol **14**

Solvent	UV[a] λ max, nm (i) = inflection	ε	ORD[b] λ nm	[φ]	CD[b] λ nm (i) = inflection	[θ]
Methanol	282	6140	294	+ 4500°pk[c]	291	+ 10900
	235 (i)	14000	281	− 13250°tr[c]	275	− 8800
	207	66700	266	− 500°pk	261	− 3800
			251	− 8500°tr	243	− 28000
			236	+ 36240°pk	225	+ 15000 (i)
					208	+ 80000
0·1 N HCl (20% Methanol)	281	6200	293	+ 3500°pk	289	+ 10300
	234	14820	281	− 12500°tr	274	− 8800
	208	61500	265	− 750°pk	260	− 4200
			250	− 8870°tr	241	− 34000
			233	+ 37490°pk	222	+ 18000 (i)
					207	+ 90000
0·1 n KOH (20% Methanol)	291	6800	304	+ 2140°pk	296	+ 17200
	247	12850	287	− 16250°tr	280	− 12300
			266	− 4000°pk	267	− 7600
			255	− 6500°tr	245	− 27000
			235	+ 10000°pk	230	− 6000 (i)
					203	+ 103000

[a] UV spectra were recorded on a Cary 14 Spectrophotometer.

[b] ORD and CD spectra were recorded on a JASCO Spectropolarimeter, Model ORD/CD/UV-5 at 23°C. Concentrations were in the range of 0·01–0·001 M.

[c] pk = peak; tr = trough.

The function minimized in the least squares refinement was

$$\sum w \big| |F_o| - |F_c| \big|^2$$

where $w = 1/(7{\cdot}1 + |F_o| + 0{\cdot}012|F_o|^2)$. Standard atomic scattering curves were used for Br^-, C, N, O[13] and H[14]. The Br^- curve was corrected for the real and imaginary parts of the anomalous scattering.[15] The refinement calculations were made with a local modification of the program ORFLS[16].

The structure of **5**.HBr was solved by the heavy atom method. Full matrix least squares with isotropic temperature factors for all atoms was used for the initial refinement. All atoms were assigned anisotropic thermal parameters and the refinement was continued by block diagonal least squares (BDLS) in which the matrix was partitioned into eight blocks. A difference Fourier calculated at this point revealed all but one of the hydrogen atoms. The hydrogen atoms were added at their calculated positions and several additional cycles of BDLS were run. The refinement was stopped when all shifts were less than $\frac{1}{3}$ of their respective standard deviations ($\frac{1}{3}\sigma$ for the hydrogens). The final difference Fourier has no features greater than 0·4 e $Å^{-1}$ in magnitude. The final unweighted R is 0·031 for the 1882 observed data.

The final atomic parameters and their standard deviations are given in Tables 2 and 3. The bond lengths and angles are in reasonable agreement with the expected values. A listing of the observed and calculated structure factors is given in Table 4.

The absolute configuration of **5**.HBr was established by comparison of the observed and calculated structure factors of selected Bijvoet pairs of reflections. The intensities of 27 pairs of reflections, $hk\ell$ and $hk\bar{\ell}$, for which $F_c(hk\ell) > 20$ e and $|\Delta F_c| = \big||F_c(hk\ell)| - |F_c(hk\bar{\ell})|\big| > 2{\cdot}0$ e were measured. In all cases the sign of the difference ΔF_o was the same as that of the corresponding ΔF_c, calculated for the configuration shown in Fig 1. In order to confirm that the crystal used for the structure and absolute configuration determinations was truly representative of the sample, the same set of Bijvoet reflections was measured for each of two additional crystals chosen from the original batch. The intensity differences for these two crystals are in agreement with those observed for the first crystal.

N-(*4-Benzyloxy-3-methoxybenzoyl*)*homoveratrylamine* (**9**). A soln of **8**[10] (17·5 g, 0·063 mol) in 50 ml CH_2Cl_2 was added over 30 min to a vigorously stirred mixture of homoveratrylamine (11·4 g, 0·063 mol) in 50 ml CH_2Cl_2 and 10 ml H_2O, maintained at 4° and slightly alkaline by the addition of 10% NaOH or as needed. The mixture was stirred at 25° for 2 hr and the crystals collected to give 16 g (60%) of **9**, m.p. 162–164°. The organic layer was separated, washed with 3N HCl, evaporated and the residue crystallized from EtOH to give another 7 g (26%) of **9**. An analytical specimen prepared from EtOH exhibited: m.p. 164–165°; IR (KBr): 3400, 1628, 1598, 1590, 1580, 1540, 1515, 1510 cm^{-1}. (Found: C, 71·39; H, 6·67; N, 3·27. Calc. for $C_{25}H_{27}NO_5$: C, 71·24; H, 6·46; N, 3·32%).

1-(*4-Benzyloxy-3-methoxyphenyl*)-*6,7-dimethoxy-3,4-dihydroisoquinoline hydrochloride* (**10**.HCl). A mixture of **9** (16 g, 0·038 mol) and 16 ml of $POCl_3$ in 160 ml of MeCN was stirred and refluxed for 2 hr and evaporated under reduced pressure. The residue was dissolved in H_2O, EtOAc added, and the mixture rendered alkaline with

Table 2. Final atomic parameters for **5**.HBr with standard deviations in parentheses

ATOM	X	Y	Z	B
Br	0·89428(5)	0·88371(4)	0·85773(3)	*
O(1)	0·7517(4)	0·6861(3)	0·5371(2)	*
O(2)	0·5592(3)	0·5752(3)	0·5944(2)	*
O(3)	−0·0720(3)	0·4129(3)	0·3910(3)	*
O(4)	0·0412(3)	0·5811(3)	0·3323(2)	*
N(2)	0·5061(4)	0·4112(3)	0·2410(2)	*
C(1)	0·4755(4)	0·3974(4)	0·3310(3)	*
C(3)	0·6513(5)	0·4194(5)	0·2282(3)	*
C(4)	0·7015(5)	0·5246(5)	0·2640(3)	*
C(4)A	0·6561(4)	0·5374(3)	0·3506(3)	*
C(5)	0·7241(4)	0·6086(4)	0·4021(3)	*
C(6)	0·6903(4)	0·6189(4)	0·4827(2)	*
C(7)	0·5846(4)	0·5583(3)	0·5141(2)	*
C(8)	0·5170(4)	0·4889(3)	0·4646(2)	*
C(8)A	0·5513(4)	0·4789(3)	0·3809(2)	*
C(9)	0·4471(7)	0·3226(6)	0·1922(4)	*
C(11)	0·3286(4)	0·3992(3)	0·3455(2)	*
C(12)	0·2663(5)	0·3087(4)	0·3750(3)	*
C(13)	0·1323(5)	0·3100(4)	0·3919(3)	*
C(14)	0·0601(4)	0·4024(4)	0·3772(3)	*
C(15)	0·1222(4)	0·4951(3)	0·3460(2)	*
C(16)	0·2560(4)	0·4932(4)	0·3314(3)	*
C(17)	0·8476(10)	0·7581(9)	0·5057(5)	*
C(18)	0·4575(7)	0·5116(6)	0·6300(3)	*
C(19)	−0·1338(6)	0·3322(6)	0·4403(5)	*
C(20)	0·0996(6)	0·6782(4)	0·3018(4)	*
H(1)	0·509(4)	0·320(3)	0·344(2)	2·2(8)
HN(2)	0·467(6)	0·471(5)	0·221(3)	4·9(13)
H(3)A	0·667(5)	0·417(4)	0·176(3)	4·7(12)
H(3)B	0·695(7)	0·340(6)	0·250(5)	8·5(19)
H(4)A	0·668(5)	0·587(4)	0·235(3)	4·0(10)
H(4)B	0·793(6)	0·526(4)	0·264(3)	4·6(12)
H(8)	0·799(5)	0·649(4)	0·381(3)	4·4(11)
H(9)A	0·493(6)	0·263(5)	0·204(4)	6·4(17)
H(9)B	0·466(7)	0·334(6)	0·124(4)	7·6(16)
H(9)C	0·346(6)	0·314(5)	0·201(4)	5·2(13)
H(11)	0·450(5)	0·438(4)	0·483(3)	3·6(10)
H(12)	0·314(5)	0·247(4)	0·386(3)	3·3(9)
H(13)	0·087(6)	0·245(5)	0·420(3)	5·7(13)
H(16)	0·297(4)	0·555(4)	0·317(3)	2·9(9)
H(17)A	0·891(8)	0·795(6)	0·553(5)	8·3(18)
H(17)B	0·811(8)	0·806(6)	0·461(5)	9·6(22)
H(17)C	0·920(8)	0·732(7)	0·477(5)	7·9(23)
H(18)A	0·445(6)	0·531(5)	0·682(4)	5·8(13)
H(18)B	0·465(6)	0·438(5)	0·618(4)	6·2(16)
H(18)C	0·374(5)	0·533(4)	0·594(3)	4·9(12)
H(19)A	−0·229(6)	0·368(5)	0·453(4)	6·0(14)
H(19)B	−0·081(7)	0·323(6)	0·497(4)	8·0(18)
H(19)C	−0·141(7)	0·267(6)	0·413(4)	7·3(19)
H(20)A	0·025(6)	0·732(5)	0·290(3)	5·5(13)
H(20)B	0·162(5)	0·656(5)	0·247(3)	5·3(12)
H(20)C	0·160(7)	0·713(5)	0·338(4)	7·2(17)

*Anisotropic thermal parameters are given in Table 3.

10% NaOH aq. The organic extract was washed with H_2O, acidified with ethanolic HCl and evaporated. The residue was crystallized from EtOH to give 12·5 g (75%) of **10**.HCl: m.p. 209–210°; NMR (DMSO-d_6) δ3·06 (t,2,J = 7 Hz,CH_2Ar), 3·67 (s,3,OCH_3), 3·82 (t,2,J = 7 Hz,CH_2N=), 3·87 (s,3,OCH_3), 3·92 (s,3,OCH_3), 5·22 (s,2,OCH_2), 6·98 (s,1,aromatic), 7·20–7·60 (m,9,aromatic). (Found: C, 68·32; H, 6·17; N, 3·18. Calc. for $C_{25}H_{25}NO_4$·HCl: C, 68·26; H, 5·96; N, 3·18%).

(±)-1-(4-*Benzyloxy*-3-*methoxyphenyl*)-6,7-*dimethoxy*-1,2,3,4-*tetrahydroisoquinoline* (**11**). To a stirred soln of **10**.HCl (44 g, 0·1 mol) in 600 ml of MeOH at 15–20°, 16·5 g of $NaBH_4$ was added over 20 min. After stirring at 25° for 3 hr, the mixture was evaporated and the residue

Table 3. Final anisotropic thermal parameters for **5**.HBr with standard deviations in parentheses

ATOM	B11 × 10⁴	B22 × 10⁵	B33 × 10⁵	B12 × 10⁵	B13 × 10⁵	B23 × 10⁵
Br	117(1)	775(4)	493(2)	85(4)	49(3)	147(2)
O(1)	135(4)	943(29)	409(13)	−539(29)	−34(20)	−9(16)
O(2)	133(4)	861(25)	299(10)	−302(27)	74(17)	−31(14)
O(3)	66(3)	1119(32)	678(18)	−7(24)	28(18)	317(21)
O(4)	87(3)	707(22)	509(14)	92(23)	55(18)	108(15)
N(2)	96(3)	671(28)	310(13)	151(28)	−5(19)	−60(16)
C(1)	75(4)	593(30)	326(14)	75(29)	11(19)	−20(17)
C(3)	102(5)	1015(45)	325(17)	156(40)	114(25)	−61(23)
C(4)	73(4)	992(42)	325(17)	5(36)	91(23)	48(23)
C(4)A	58(3)	741(29)	311(14)	79(27)	5(19)	39(19)
C(5)	64(3)	791(33)	369(16)	−77(33)	−23(20)	98(21)
C(6)	76(4)	648(28)	333(15)	−153(33)	−30(20)	−24(19)
C(7)	74(4)	542(26)	281(13)	−7(27)	52(19)	27(15)
C(8)	67(4)	587(28)	275(13)	24(27)	33(18)	36(16)
C(8)A	64(3)	526(26)	300(14)	63(25)	−7(19)	2(15)
C(9)	155(8)	908(48)	401(21)	−109(51)	−13(35)	−197(27)
C(11)	80(4)	581(27)	286(14)	−27(28)	−66(19)	−27(17)
C(12)	101(5)	604(30)	388(19)	−14(32)	−69(25)	87(19)
C(13)	88(5)	742(35)	488(20)	−115(33)	−73(25)	155(22)
C(14)	83(4)	787(35)	354(16)	−36(32)	−14(21)	98(20)
C(15)	73(4)	663(27)	314(15)	36(28)	−49(21)	43(17)
C(16)	84(4)	519(26)	312(15)	−28(28)	−2(21)	−19(16)
C(17)	224(12)	1661(84)	509(28)	−1279(94)	−119(42)	108(44)
C(18)	172(8)	1016(51)	356(20)	−358(53)	227(35)	52(27)
C(19)	92(6)	1103(55)	702(32)	−146(45)	150(35)	334(37)
C(20)	105(5)	568(31)	623(25)	38(39)	21(35)	67(23)

The anisotropic temperature factor has the form

$$\exp\left[-\left(h^2\mathrm{B}11+k^2\mathrm{B}22+l^2\mathrm{B}33+2hk\mathrm{B}12+2hl\mathrm{B}13+2kl\mathrm{B}23\right)\right].$$

distributed between a mixture of CH_2Cl_2–H_2O. The organic layer was washed with H_2O, evaporated, and the residue crystallized from EtOH to give 39·8 g (97%) of **11**: m.p. 127–129°; NMR ($CDCl_3$) δ1·92 (s,1,NH), 2·60–3·40 (m,4,CH_2CH_2), 3·60, 3·79, 3·83 (3s,9,3 OCH_3), 4·95 (s,1,CH), 5·13 (s,2,CH_2O), 6·25, 6·60 (2s,2,aromatic), 6·60–7·00 (m,3,aromatic), 7·20–7·60 (m,5,aromatic). (Found: C, 74·11; H, 6·72; N, 3·46. Calc. for $C_{25}H_{27}NO_4$: C, 74·05; H, 6·71; N, 3·45%).

(+)-1(R)-1-(*4-Benzyloxy-3-methoxyphenyl*)-6,7-*dimethoxy*-1,2,3,4-*tetrahydroisoquinoline* (**12**). A soln of **11** (20·3 g, 0·05 mol) and (−)-diacetone-2-keto-L-gulonic acid hydrate[11] (14·6 g, 0·05 mol) in 400 ml of EtOH was stored at 25° overnight. The crystals were collected and recrystallized twice from 300 ml of EtOH to give 12·4 g (73% based on 0·025 mol) of **12** · (−)-diacetone-2-keto-L-gulonate: m.p. 192–194°- $[\alpha]^{25}+1\cdot9^\circ$ (*c* 1,MeOH); $[\alpha]^{25}_{365}+19\cdot4^\circ$ (*c* 1,MeOH). (Found: C, 65·32; H, 6·71; N, 1·95. Calc. for $C_{25}H_{27}NO_4$. $C_{12}H_{18}O_7$: C, 65·38; H, 6·67; N, 2·06%).

An aqueous soln of the above salt (10·9 g, 0·016 mol) was rendered alkaline with 10% NaOH aq. and extracted with CH_2Cl_2, the extract washed with H_2O and evaporated. The residue was crystallized from EtOH to give 6·3 g (97%) of **12**: m.p. 129–130°; $[\alpha]^{25}+25\cdot0^\circ$ (*c* 1,$CHCl_3$); UV max (MeOH) 206 nm (ϵ 75000), 230 (19250) (infl), 282 (7090); ORD (*c* 0·405, MeOH) $[\phi]_{700}+73^\circ$, $[\phi]_{589}+111^\circ$, $[\phi]_{293}+8000^\circ$ (pk), $[\phi]_{288}$ 0° (int), $[\phi]_{279}-13380^\circ$ (tr), $[\phi]_{263}$ 0° (pk), $[\phi]_{245}-8750^\circ$ (tr), $[\phi]_{240}$ 0° (int), $[\phi]_{230}+27500^\circ$ (pk); CD (*c* 0·01 *M*, MeOH) $[\phi]_{310}$ 0, $[\theta]_{288}+14200$, $[\theta]_{280}$ 0, $[\theta]_{273}-7600$, $[\theta]_{256}-1800$, $[\theta]_{240}-12000$; $[\theta]_{232}$ 0; $[\theta]_{224}+17000$ (sh), $[\theta]_{207}+125000$; NMR similar to that of **11**. (Found: C, 74·23; H, 6·76; N, 3·35. Calc. for $C_{25}H_{27}NO_4$: C, 74·05; H, 6·71; N, 3·45%).

(−)-1(R)-1-(*4-Benzyloxy-3-methoxyphenyl*)-*2-methyl*-6,7-*dimethoxy*-1,2,3,4-*tetrahydroisoquinoline* (**13**). A mixture of **12** (12·4 g, 0·03 mol) and 20 ml of 37% CH_2O in 100 ml MeOH and 60 ml dioxane was stored overnight at 25°. To the stirred soln at 15–20°, 12 g $NaBH_4$ was added over 30 min. After stirring at 25° for 2 hr, the mixture was evaporated, the residue dissolved in H_2O and extracted with CH_2Cl_2. The extract was washed with H_2O, evaporated, and the residue crystallized from a mixture of EtOH–H_2O to give 11·4 g (82%) of **13**: m.p. 109–110°; $[\alpha]^{25}-56\cdot0^\circ$ (*c* 1,$CHCl_3$); UV max (MeOH) 207 nm (ϵ 74000), 230 (20000) (infl), 282 (6720); ORD (*c* 0·419 MeOH) $[\phi]_{700}-111^\circ$, $[\phi]_{589}-174^\circ$, $[\phi]_{303}$ 0° (int), $[\phi]_{294}+3500^\circ$ (pk), $[\phi]_{291}$ 0° (int), $[\phi]_{281}-19800^\circ$ (tr), $[\phi]_{264}-6250^\circ$ (pk), $[\phi]_{245}-23700^\circ$ (tr), $[\phi]_{238}$ 0° (infl), $[\phi]_{230}$ 32500° (pk); CD (*c* 0·01*M*, MeOH) $[\theta]_{310}$ 0, $[\theta]_{289}+15200$, $[\theta]_{281}$ 0, $[\theta]_{274}-9400$, $[\theta]_{257}-3000$, $[\theta]_{240}-47000$, $[\theta]_{225}-5000$ (sh), $[\theta]_{220}$ 0, $[\theta]_{210}+85000$, $[\theta]_{202}$ 0; NMR ($CDCl_3$) δ2·25 (s,3,NCH_3), 2·50–3·30 (m,4,CH_2CH_2), 3·58, 3·84, 3·86 (3s,9,3 OCH_3), 4·15 (s,1,CH), 5·18 (s,2,OCH_2), 6·26, 6·64 (2s,2,aromatic), 6·70–7·00 (m,3,aromatic), 7·20–7·70 (m,5, aromatic). (Found: C, 74·45; H, 7·24; N, 3·33. Calcd. for $C_{26}H_{29}NO_4$: C, 74·44; H, 6·97; N, 3·34%).

(−)-1(R)-6,7-*Dimethoxy-2-methyl-1-*(*4-hydroxy-3-methoxyphenyl*)-1,2,3,4-*tetrahydroisoquinoline* (**14**). A mixture of **13** (6 g, 0·0143 mol) and 60 ml conc HCl in 60 ml C_6H_6 was stirred in a N_2 atm at 25° for 18 hr. The aqueous layer was washed with ether, neutralized with $NaHCO_3$,

Table 4. Observed and calculated structure factors (× 10) for 5.HBr.

H	K	L	FO	FC
2	0	0	866	837
4	0	0	464	497
6	0	0	487	494
8	0	0	166	166
10	0	0	50	47
1	1	0	106	103
2	1	0	163	163
3	1	0	785	797
4	1	0	75	72
5	1	0	38	32
6	1	0	603	610
7	1	0	446	429
8	1	0	145	146
9	1	0	86	69
10	1	0	60	60
11	1	0	140	146
12	1	0	62	68
0	2	0	1327	1347
1	2	0	755	799
2	2	0	627	803
3	2	0	962	952
4	2	0	215	207
5	2	0	1045	1049
7	2	0	306	315
8	2	0	154	150
9	2	0	231	231
11	2	0	66	62
1	3	0	426	461
2	3	0	71	63
3	3	0	997	956
4	3	0	158	151
5	3	0	104	102
6	3	0	571	575
8	3	0	255	253
9	3	0	106	103
10	3	0	113	109
11	3	0	71	77
12	3	0	77	62
0	4	0	1046	1075
1	4	0	332	355
2	4	0	319	311
3	4	0	153	156
4	4	0	549	559
5	4	0	402	392
6	4	0	312	305
7	4	0	447	450
10	4	0	196	195
11	4	0	59	63
2	5	0	336	336
3	5	0	529	512
4	5	0	215	212
6	5	0	102	98
7	5	0	100	98
8	5	0	174	172
11	5	0	116	116
0	6	0	355	342
1	6	0	476	467
2	6	0	517	459
3	6	0	54	64
4	6	0	159	152
5	6	0	386	367
7	6	0	124	123
8	6	0	191	168
9	6	0	225	217
10	6	0	48	46
1	7	0	216	225
2	7	0	562	575
3	7	0	317	323
4	7	0	213	217
6	7	0	431	429
7	7	0	295	292
8	7	0	251	238
0	8	0	313	307
1	8	0	124	132
4	8	0	269	255
5	8	0	239	236
7	8	0	242	240
8	8	0	113	106
9	8	0	46	49
10	8	0	126	127
1	9	0	363	369
2	9	0	247	246
3	9	0	412	402
4	9	0	297	299
5	9	0	72	72
6	9	0	77	73
7	9	0	213	215
0	10	0	618	614
1	10	0	288	260
2	10	0	66	78
3	10	0	235	229
4	10	0	66	64
5	10	0	100	90
6	10	0	106	100
8	10	0	87	87
9	10	0	113	115
1	11	0	96	96
2	11	0	312	316
4	11	0	350	349
6	11	0	141	145
8	11	0	73	73
2	12	0	113	104
3	12	0	111	115
4	12	0	176	169
5	12	0	107	116
6	12	0	44	44
1	13	0	76	79
3	13	0	217	219
4	13	0	53	60
4	14	0	62	74
1	0	1	763	760
2	0	1	1076	1051
3	0	1	595	565
4	0	1	643	623
5	0	1	245	225
6	0	1	75	91
7	0	1	138	129
8	0	1	192	192
9	0	1	266	257
10	0	1	66	67
11	0	1	87	68
12	0	1	86	62
0	1	1	159	160
1	1	1	270	267
2	1	1	428	436
3	1	1	804	810
4	1	1	615	615
5	1	1	676	665
6	1	1	313	362
7	1	1	298	309
8	1	1	317	314
9	1	1	154	153
10	1	1	79	77
11	1	1	68	72
12	1	1	80	60
0	2	1	114	114
1	2	1	1133	1161
2	2	1	669	658
3	2	1	462	457
4	2	1	660	675
5	2	1	141	144
6	2	1	361	361
7	2	1	308	303
8	2	1	157	156
9	2	1	83	71
10	2	1	218	218
11	2	1	78	81
12	2	1	62	53
0	3	1	400	459
1	3	1	1103	1129
2	3	1	825	841
3	3	1	884	894
4	3	1	416	411
5	3	1	406	402
6	3	1	175	169
7	3	1	261	254
8	3	1	115	118
9	3	1	73	63
10	3	1	91	93
11	3	1	63	50
12	3	1	54	43
1	4	1	673	689
2	4	1	625	642
3	4	1	530	537
4	4	1	125	118
5	4	1	415	426
6	4	1	160	147
7	4	1	170	165
8	4	1	237	230
9	4	1	285	280
11	4	1	72	72
0	5	1	478	479
1	5	1	551	534
2	5	1	806	807
3	5	1	197	182
4	5	1	479	497
5	5	1	367	353
6	5	1	306	305
7	5	1	242	244
8	5	1	160	165
9	5	1	66	58
11	5	1	66	61
0	6	1	543	570
1	6	1	260	272
2	6	1	118	122
3	6	1	454	459
4	6	1	325	329
5	6	1	103	104
6	6	1	339	332
7	6	1	228	226
8	6	1	207	206
9	6	1	168	165
10	6	1	59	74
11	6	1	132	132
0	7	1	363	370
1	7	1	333	338
2	7	1	186	182
3	7	1	411	404
4	7	1	257	265
5	7	1	180	174
6	7	1	256	259
7	7	1	216	207
9	7	1	69	75
10	7	1	63	76
0	8	1	55	50
1	8	1	422	418
2	8	1	207	211
3	8	1	204	213
4	8	1	63	66
5	8	1	275	276
6	8	1	220	216
8	8	1	129	119
9	8	1	119	114
10	8	1	56	59
0	9	1	103	94
1	9	1	144	146
2	9	1	307	304
3	9	1	53	61
4	9	1	107	109
5	9	1	358	361
6	9	1	179	175
7	9	1	79	72
8	9	1	81	81
9	9	1	50	50
0	10	1	329	326
1	10	1	174	168
2	10	1	141	150
3	10	1	136	136
4	10	1	193	182
5	10	1	101	104
6	10	1	69	62
7	10	1	100	88
8	10	1	69	71
9	10	1	74	79
0	11	1	292	286
2	11	1	187	184
3	11	1	186	180
4	11	1	132	128
5	11	1	77	80
6	11	1	124	116
7	11	1	127	133
8	11	1	46	42
1	12	1	226	215
2	12	1	145	136
3	12	1	98	98
4	12	1	134	133
5	12	1	137	147
6	12	1	86	91
7	12	1	57	64
0	13	1	91	94
1	13	1	94	96
2	13	1	138	137
4	13	1	71	71
5	13	1	91	107
1	14	1	114	139
2	14	1	76	73
3	14	1	70	78
4	14	1	51	46
0	0	2	386	457
1	0	2	230	240
2	0	2	367	385
3	0	2	951	962
4	0	2	51	47
5	0	2	207	205
6	0	2	211	203
7	0	2	345	345
8	0	2	64	65
9	0	2	182	199
11	0	2	50	104
0	1	2	665	663
1	1	2	700	710
2	1	2	375	368
3	1	2	505	517
4	1	2	579	588
5	1	2	503	505
6	1	2	270	274
7	1	2	120	115
8	1	2	173	162
9	1	2	177	178
10	1	2	235	235
11	1	2	95	93
0	2	2	627	668
1	2	2	936	983
2	2	2	1489	1516
3	2	2	92	92
4	2	2	126	131
5	2	2	243	238
6	2	2	314	320
8	2	2	297	294
9	2	2	81	77
10	2	2	120	112
12	2	2	103	102
0	3	2	437	455
1	3	2	1126	1147
2	3	2	659	633
4	3	2	262	273
5	3	2	480	479
6	3	2	224	227
7	3	2	109	112
8	3	2	163	161
9	3	2	136	139
10	3	2	67	69
11	3	2	73	63
0	4	2	291	295
1	4	2	681	698
2	4	2	651	657
3	4	2	375	382
4	4	2	154	152
5	4	2	225	221
6	4	2	78	65
7	4	2	698	690
8	4	2	122	129
9	4	2	112	112
11	4	2	126	117
0	5	2	687	690
1	5	2	75	82
2	5	2	109	112
3	5	2	322	329
4	5	2	720	731
5	5	2	254	253
6	5	2	263	258
7	5	2	116	124
8	5	2	352	345
9	5	2	186	187
10	5	2	142	145
11	5	2	76	77
0	6	2	66	95
1	6	2	219	222
2	6	2	720	727
3	6	2	190	186
4	6	2	346	344
5	6	2	317	314
6	6	2	276	277
7	6	2	462	458
8	6	2	214	212
9	6	2	73	75
10	6	2	82	77
0	7	2	192	205
1	7	2	458	465
2	7	2	231	225
3	7	2	325	319
4	7	2	152	149
5	7	2	365	357
6	7	2	59	61
7	7	2	183	181
8	7	2	90	86
9	7	2	138	133
10	7	2	46	40
0	8	2	100	90
1	8	2	436	433
2	8	2	280	267
3	8	2	376	381
4	8	2	175	169
5	8	2	234	242
6	8	2	193	193
7	8	2	212	208
8	8	2	100	91
0	9	2	147	156
1	9	2	127	118
2	9	2	94	98
4	9	2	364	359
5	9	2	145	147
6	9	2	116	107
8	9	2	112	106
0	10	2	131	130
1	10	2	257	257
2	10	2	291	284
3	10	2	158	153
4	10	2	83	78
5	10	2	108	103
6	10	2	145	143
7	10	2	92	94
8	10	2	85	89
9	10	2	100	103
0	11	2	263	245
1	11	2	244	235
3	11	2	56	63
4	11	2	57	49
5	11	2	176	176
6	11	2	69	74
8	11	2	71	66
1	12	2	190	183
2	12	2	155	152
3	12	2	156	150
7	12	2	76	80
0	13	2	119	118
2	13	2	106	106
3	13	2	56	36
4	13	2	102	113
5	13	2	46	48
6	13	2	81	94
1	14	2	80	81
2	14	2	100	101
3	14	2	60	74
4	14	2	44	44
1	0	3	671	689
2	0	3	1417	1471
3	0	3	167	191
4	0	3	636	631
5	0	3	94	106
6	0	3	542	560
8	0	3	193	192
9	0	3	198	197
10	0	3	121	128
11	0	3	55	38
0	1	3	1183	1143
1	1	3	1006	1068
2	1	3	710	706
3	1	3	421	421
4	1	3	335	350
5	1	3	710	697
6	1	3	294	295
7	1	3	142	146
8	1	3	206	202
9	1	3	201	212
10	1	3	121	118
11	1	3	102	99
12	1	3	52	48
0	2	3	144	125
1	2	3	701	708
2	2	3	201	197
3	2	3	534	532
4	2	3	107	107
5	2	3	417	415
6	2	3	142	132
7	2	3	387	389
8	2	3	173	170
10	2	3	155	151
11	2	3	117	116
0	3	3	728	685
1	3	3	640	644
2	3	3	547	527
3	3	3	685	688
4	3	3	569	590
5	3	3	346	348
6	3	3	344	349
7	3	3	166	168
9	3	3	93	102
10	3	3	214	213
0	4	3	244	249
1	4	3	267	243
2	4	3	580	588
3	4	3	427	411
4	4	3	532	540
5	4	3	206	200
6	4	3	233	237
7	4	3	126	124
8	4	3	434	434
9	4	3	166	181
10	4	3	120	110
0	5	3	412	432
1	5	3	502	534
2	5	3	545	546
3	5	3	106	112
4	5	3	398	392
5	5	3	436	422
6	5	3	146	148
7	5	3	231	223
8	5	3	106	107
9	5	3	113	101
0	6	3	749	717
1	6	3	455	468
2	6	3	132	120
3	6	3	733	734
4	6	3	170	170
5	6	3	124	123
6	6	3	200	202
7	6	3	290	290
8	6	3	82	65
9	6	3	101	95
10	6	3	76	82
11	6	3	85	87
0	7	3	767	769
2	7	3	297	269
3	7	3	328	340
4	7	3	506	501
5	7	3	171	175
6	7	3	133	134
7	7	3	203	195
8	7	3	52	42
9	7	3	126	124
10	7	3	117	120
0	8	3	273	273
1	8	3	353	360
2	8	3	334	331
3	8	3	216	208
4	8	3	107	114
5	8	3	131	133
6	8	3	314	304
8	8	3	230	223
9	8	3	57	55
10	8	3	70	69
0	9	3	86	67
1	9	3	744	231
2	9	3	116	122
3	9	3	213	212
4	9	3	140	140
5	9	3	211	207
6	9	3	122	125
8	9	3	48	49
9	9	3	110	115
0	10	3	192	188
1	10	3	246	242
2	10	3	126	126
3	10	3	266	268
4	10	3	199	212
5	10	3	54	54
6	10	3	172	175
7	10	3	155	153
8	10	3	114	122
9	10	3	47	51
0	11	3	394	390
1	11	3	50	60
3	11	3	99	104
4	11	3	140	130
6	11	3	102	100
1	12	3	178	177
2	12	3	102	98
3	12	3	100	99
6	12	3	81	93
7	12	3	51	53
1	13	3	65	67
2	13	3	46	33
3	13	3	69	70
5	13	3	111	126
1	14	3	56	60
2	14	3	90	91
3	14	3	67	91
0	0	4	1645	1723
1	0	4	181	175
2	0	4	606	623
3	0	4	197	204
4	0	4	678	675
6	0	4	628	630
7	0	4	236	242
8	0	4	76	68
9	0	4	123	122
12	0	4	61	54
0	1	4	112	97
1	1	4	1178	1201
2	1	4	495	503
3	1	4	587	606
4	1	4	401	390
5	1	4	218	224
6	1	4	349	345
7	1	4	256	249
8	1	4	216	213
9	1	4	124	126
10	1	4	110	107
11	1	4	173	177
12	1	4	92	94
0	2	4	627	640
1	2	4	1198	1187
2	2	4	682	678
3	2	4	209	220
4	2	4	391	380
5	2	4	745	751
6	2	4	227	217
8	2	4	55	54
9	2	4	270	264
10	2	4	133	132
12	2	4	78	82
0	3	4	557	554
1	3	4	534	514
2	3	4	562	572
3	3	4	327	321
4	3	4	446	436
5	3	4	65	61
6	3	4	328	321
7	3	4	216	222
8	3	4	361	309
9	3	4	67	58
0	4	4	786	811
1	4	4	364	348
2	4	4	396	389
3	4	4	239	246
4	4	4	456	452
5	4	4	51	43
6	4	4	363	366
7	4	4	279	276
8	4	4	148	152
9	4	4	50	28
10	4	4	162	161
11	4	4	43	47
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3	5	4	723	722
4	5	4	196	188
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7	5	4	74	67
8	5	4	137	135
9	5	4	145	142
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0	6	4	211	222
1	6	4	399	414
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5	6	4	359	356
6	6	4	245	244
7	6	4	236	239
8	6	4	117	113
9	6	4	204	200
10	6	4	49	56
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1	7	4	360	335
2	7	4	263	272
3	7	4	159	156
4	7	4	66	62
6	7	4	224	223
7	7	4	64	65
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10	7	4	47	30
0	8	4	435	431
1	8	4	292	294
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3	8	4	234	238
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5	8	4	225	234
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6	9	4	133	130
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6	10	4	178	173
0	11	4	128	123
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6	11	4	72	75
7	11	4	54	48
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7	12	4	54	59
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3	13	4	86	90
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6	0	5	124	129
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10	0	5	79	82
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1	1	5	145	135
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4	1	5	317	316
5	1	5	171	163
6	1	5	197	194
7	1	5	215	214
8	1	5	242	242
9	1	5	152	158
10	1	5	57	49
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2	2	5	269	281
3	2	5	333	325
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6	2	5	287	295
7	2	5	126	128
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8	3	5	146	142
9	3	5	122	129
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2	4	5	416	396
3	4	5	329	344
4	4	5	110	116
5	4	5	456	465
6	4	5	50	46
7	4	5	105	105
8	4	5	66	66
9	4	5	257	254
10	4	5	61	86
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0	5	5	105	87
1	5	5	346	346
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3	5	5	327	330
4	5	5	117	121
5	5	5	266	287
6	5	5	390	385
7	5	5	201	201
8	5	5	97	101
11	5	5	61	59
0	6	5	394	412
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3	6	5	155	151
4	6	5	365	365
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7	6	5	111	112
8	6	5	158	150
9	6	5	102	98
10	6	5	135	143
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4	7	5	175	178
5	7	5	160	98
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10	7	5	43	44
0	8	5	160	166
1	8	5	270	271
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3	8	5	123	121
4	8	5	142	136
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6	8	5	133	129
8	8	5	66	88
9	8	5	72	75
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1	9	5	228	232
2	9	5	360	370
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7	9	5	73	65
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3	10	5	97	88
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7	10	5	75	77
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6	11	5	55	64
7	11	5	121	125
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4	12	5	73	85
5	12	5	84	91
1	13	5	60	85
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3	13	5	42	18
4	13	5	65	78
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6	0	6	131	130
7	0	6	374	377
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9	0	6	67	73
10	0	6	61	54
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2	1	6	275	294
3	1	6	519	519
4	1	6	221	216
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6	1	6	224	219
7	1	6	62	61
8	1	6	213	216
9	1	6	124	119
10	1	6	209	212
11	1	6	72	77
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6	2	6	214	210
7	2	6	117	110
8	2	6	267	268
9	2	6	172	174
10	2	6	49	39
11	2	6	126	131
1	3	6	363	352
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3	3	6	481	466
4	3	6	655	661
5	3	6	276	276
6	3	6	179	176
7	3	6	106	105
8	3	6	289	290
9	3	6	142	141
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11	3	6	48	50
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3	4	6	368	369
4	4	6	285	286
5	4	6	162	158
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8	4	6	107	104
9	4	6	85	85
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1	5	6	289	291
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3	5	6	467	462
4	5	6	272	271
5	5	6	153	143
6	5	6	285	274
8	5	6	156	150
9	5	6	83	82
10	5	6	63	62
11	5	6	44	37
0	6	6	57	66
1	6	6	306	304
2	6	6	366	369
3	6	6	221	225
4	6	6	235	230
5	6	6	269	270
6	6	6	230	225
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8	6	6	141	153
9	6	6	134	135
10	6	6	47	47
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1	7	6	285	279
2	7	6	256	261
3	7	6	252	245
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5	7	6	284	271
6	7	6	160	162
7	7	6	272	273
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9	7	6	125	128
0	8	6	409	403
1	8	6	130	131
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3	8	6	251	247
4	8	6	243	237
6	8	6	69	62
7	8	6	136	130
8	8	6	177	173
9	8	6	77	76
0	9	6	296	267
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2	9	6	170	168
3	9	6	127	122
4	9	6	107	110
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6	9	6	83	78
7	9	6	141	142
8	9	6	69	72
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2	10	6	205	209
3	10	6	120	114
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5	10	6	164	155
6	10	6	136	137
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8	10	6	41	39
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1	11	6	159	158
2	11	6	113	110
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5	11	6	150	147
6	11	6	56	62
7	11	6	74	75
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1	12	6	55	47
2	12	6	81	66
3	12	6	106	110
4	12	6	107	109
5	12	6	58	58
0	13	6	62	55
2	13	6	56	52
3	13	6	46	49
4	13	6	114	125
1	14	6	80	67
2	14	6	64	76
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2	0	7	876	869
3	0	7	100	94
4	0	7	281	272
6	0	7	520	510
8	0	7	155	148
9	0	7	116	124
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3	1	7	259	255
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6	1	7	255	263
7	1	7	217	215
8	1	7	109	108
9	1	7	238	237
10	1	7	103	98
0	2	7	243	259
1	2	7	715	712
2	2	7	557	557
3	2	7	654	666
4	2	7	122	127
5	2	7	185	181
6	2	7	119	114
7	2	7	342	337
8	2	7	90	91
9	2	7	90	94
11	2	7	93	89
0	3	7	802	837
1	3	7	387	372
2	3	7	203	205
3	3	7	155	149
4	3	7	395	407
5	3	7	274	269
6	3	7	202	209
8	3	7	169	162
10	3	7	118	121
0	4	7	272	266
1	4	7	87	65
2	4	7	642	648
3	4	7	117	112
4	4	7	297	283
5	4	7	111	107
6	4	7	208	208
7	4	7	119	122
8	4	7	219	225
9	4	7	109	113
10	4	7	109	103
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1	5	7	555	554
2	5	7	155	161
4	5	7	318	314
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6	5	7	146	155
7	5	7	172	165
8	5	7	79	72
9	5	7	116	115
10	5	7	71	76
1	6	7	272	281
2	6	7	144	138
3	6	7	350	345
4	6	7	165	156
5	6	7	126	125
7	6	7	166	166
8	6	7	105	105
9	6	7	115	111
10	6	7	48	52
0	7	7	473	460
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2	7	7	173	172
3	7	7	184	182
4	7	7	366	362
5	7	7	125	126
6	7	7	114	106
7	7	7	69	62
8	7	7	89	91
9	7	7	90	95
10	7	7	54	58
0	8	7	139	147
1	8	7	82	87
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3	8	7	102	98
4	8	7	174	165
5	8	7	63	75
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7	8	7	50	56
8	8	7	147	151
9	8	7	53	63
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3	9	7	176	170
5	9	7	339	341
6	9	7	113	110
0	10	7	93	95
1	10	7	157	151
2	10	7	119	123
3	10	7	192	179
4	10	7	106	101
5	10	7	89	84
6	10	7	88	94
7	10	7	85	90
8	10	7	56	56
0	11	7	301	298
2	11	7	66	62
4	11	7	147	150
6	11	7	110	119
0	12	7	55	51
1	12	7	111	111
2	12	7	152	151
3	12	7	89	86
4	12	7	53	56
6	12	7	55	66
0	13	7	66	72
1	13	7	71	79
3	13	7	60	56
4	13	7	41	42
1	14	7	63	73
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3	0	8	644	635
4	0	8	342	327
5	0	8	347	352
6	0	8	157	160
7	0	8	145	150
8	0	8	57	59
10	0	8	100	94
11	0	8	78	82
0	1	8	200	169
1	1	8	476	497
2	1	8	500	507
3	1	8	230	222
4	1	8	350	361
5	1	8	233	240
6	1	8	419	435
7	1	8	232	230
9	1	8	118	111
10	1	8	149	149
11	1	8	47	50
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1	2	8	611	628
2	2	8	704	700
3	2	8	121	120
4	2	8	209	217
5	2	8	393	369
6	2	8	125	130
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9	2	8	63	52
11	2	8	59	59
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Table 4 (continued).

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2	3	8	219	240
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6	3	8	183	179
7	3	8	152	145
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10	3	8	71	75
11	3	8	36	39
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2	4	8	99	80
3	4	8	356	345
4	4	8	216	216
5	4	8	162	159
6	4	8	189	193
7	4	8	336	333
8	4	8	107	106
9	4	8	74	66
10	4	8	106	110
0	5	8	131	118
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3	5	8	319	319
4	5	8	282	277
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6	5	8	256	249
7	5	8	175	174
8	5	8	177	169
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10	5	8	67	62
0	6	8	355	349
1	6	8	222	216
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3	6	8	64	67
4	6	8	186	192
5	6	8	163	160
6	6	8	159	159
7	6	8	142	134
8	6	8	133	126
9	6	8	61	65
10	6	8	40	43
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1	7	8	325	302
2	7	8	216	217
3	7	8	223	224
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5	7	8	183	176
6	7	8	91	98
8	7	8	77	74
9	7	8	59	58
0	8	8	249	256
1	8	8	179	167
2	8	8	141	141
3	8	8	190	161
4	8	8	196	193
5	8	8	122	128
6	8	8	62	62
7	8	8	109	102
9	8	8	47	47
1	9	8	117	117
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5	9	8	67	69
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7	9	8	75	82
8	9	8	40	38
0	10	8	169	168
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3	10	8	136	134
4	10	8	75	72
6	10	8	123	135
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1	11	8	152	137
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4	11	8	115	120
5	11	8	55	58
6	11	8	50	56
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1	12	8	146	143
2	12	8	86	84
3	12	8	71	66
4	12	8	49	57
5	12	8	64	77
0	13	8	166	166
1	13	8	71	73
2	13	8	72	64
3	13	8	45	56
1	0	9	775	756
3	0	9	314	320
5	0	9	192	197
6	0	9	164	166
9	0	9	151	144
11	0	9	95	97
0	1	9	175	167
1	1	9	259	258
2	1	9	165	164
3	1	9	254	256
4	1	9	148	150
5	1	9	106	105
6	1	9	349	357
7	1	9	281	280
8	1	9	263	266
9	1	9	48	39
10	1	9	53	59
11	1	9	69	69
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1	2	9	221	202
2	2	9	182	180
3	2	9	63	61
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6	2	9	207	204
7	2	9	61	55
8	2	9	91	86
9	2	9	48	49
10	2	9	108	102
0	3	9	270	258
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3	3	9	331	345
4	3	9	121	117
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8	3	9	128	131
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10	3	9	93	100
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1	4	9	434	415
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4	4	9	144	140
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7	5	9	81	84
8	5	9	91	90
9	5	9	56	61
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1	6	9	235	205
2	6	9	85	88
3	6	9	138	134
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5	6	9	86	94
6	6	9	207	210
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1	7	9	258	251
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4	7	9	73	76
5	7	9	125	147
6	7	9	115	116
7	7	9	215	214
8	7	9	65	56
0	8	9	90	84
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2	8	9	169	160
3	8	9	101	106
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6	8	9	205	199
0	9	9	153	153
2	9	9	217	209
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6	9	9	127	127
7	9	9	45	39
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1	10	9	153	156
2	10	9	73	66
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1	11	9	115	112
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4	11	9	70	84
1	12	9	59	55
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4	12	9	94	105
5	12	9	76	91
4	13	9	110	124
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3	0	10	327	311
4	0	10	204	206
5	0	10	156	159
6	0	10	89	86
7	0	10	65	69
8	0	10	251	250
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1	1	10	302	297
2	1	10	301	305
3	1	10	430	431
4	1	10	272	280
5	1	10	191	197
6	1	10	70	71
7	1	10	186	188
8	1	10	240	236
9	1	10	107	104
0	2	10	296	294
1	2	10	369	369
2	2	10	194	197
3	2	10	220	222
4	2	10	101	96
5	2	10	467	465
6	2	10	192	189
7	2	10	52	59
8	2	10	165	160
9	2	10	170	171
0	3	10	56	56
1	3	10	136	138
2	3	10	427	420
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4	3	10	237	236
5	3	10	296	290
6	3	10	117	110
8	3	10	193	192
9	3	10	99	104
10	3	10	57	56
0	4	10	306	312
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9	4	10	73	72
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7	5	10	102	84
8	5	10	63	76
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0	6	10	63	65
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3	6	10	92	87
4	6	10	105	99
5	6	10	307	308
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9	6	10	102	107
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9	7	10	94	98
0	8	10	394	386
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7	8	10	55	62
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2	0	11	270	277
3	0	11	104	88
4	0	11	100	100
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6	0	11	229	223
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1	3	11	243	246
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4	8	11	143	150
6	8	11	120	129
7	8	11	46	56
1	9	11	167	163
2	9	11	106	106
3	9	11	91	92
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5	9	11	128	136
6	9	11	67	67
7	9	11	59	62
0	10	11	59	52
1	10	11	56	51
2	10	11	111	108
3	10	11	101	107
4	10	11	121	125
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1	1	12	204	200
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6	1	12	98	95
7	1	12	50	58
8	1	12	85	89
9	1	12	125	126
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4	9	12	90	84
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6	9	12	93	96
0	10	12	77	73
2	10	12	107	98
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2	11	12	63	71
3	11	12	76	65
1	12	12	75	81
1	0	13	223	218
2	0	13	128	131
3	0	13	82	82
5	0	13	367	365
6	0	13	118	116
7	0	13	55	53
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1	1	13	190	193
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3	3	13	131	142
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6	3	13	49	47
7	3	13	121	117
8	3	13	91	92
9	3	13	52	48
0	4	13	203	189
1	4	13	230	226
2	4	13	118	114
3	4	13	63	67
4	4	13	128	132
5	4	13	207	197
6	4	13	70	64
8	4	13	55	54
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3	5	13	199	199
4	5	13	90	83
5	5	13	69	72
6	5	13	56	61
8	5	13	81	85
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2	6	13	103	97
3	6	13	50	51
4	6	13	171	154
5	6	13	133	133
6	6	13	59	56
7	6	13	111	118
8	6	13	53	57
0	7	13	102	92
1	7	13	206	197
2	7	13	106	99
3	7	13	96	92
4	7	13	60	73
5	7	13	106	101
7	7	13	69	75
0	8	13	101	100
1	8	13	55	53
2	8	13	165	176
4	8	13	60	58
5	8	13	109	115
6	8	13	99	106
1	9	13	126	127
2	9	13	155	157
4	9	13	76	73
5	9	13	98	104
1	10	13	106	110
3	10	13	70	73
4	10	13	49	56
0	11	13	40	39
1	11	13	91	99
0	0	14	74	71
1	0	14	51	52
3	0	14	262	264
4	0	14	430	423
5	0	14	63	75
7	0	14	46	45
8	0	14	42	43
0	1	14	73	76
1	1	14	168	167
2	1	14	224	233
3	1	14	201	200
4	1	14	113	113
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6	1	14	66	72
7	1	14	82	87
8	1	14	67	71
0	2	14	51	36
1	2	14	274	280
2	2	14	107	106
3	2	14	106	103
4	2	14	138	139
5	2	14	164	157
6	2	14	90	89
7	2	14	50	55
8	2	14	66	84
0	3	14	66	86
1	3	14	156	160
2	3	14	236	230
3	3	14	82	81
4	3	14	154	151
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6	3	14	69	70
7	3	14	119	132
8	3	14	110	118
0	4	14	193	187
1	4	14	105	95
4	4	14	178	175
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6	4	14	105	106
7	4	14	62	63
8	4	14	60	63
1	5	14	151	149
2	5	14	108	109
3	5	14	149	152
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6	5	14	50	45
7	5	14	139	147
0	6	14	77	74
1	6	14	159	151
2	6	14	90	86
3	6	14	72	61
5	6	14	151	150
0	7	14	105	96
1	7	14	60	62
2	7	14	230	222
3	7	14	118	116
4	7	14	69	65
5	7	14	55	46
6	7	14	72	60
0	8	14	164	167
3	8	14	45	42
4	8	14	127	135
0	9	14	131	132
1	9	14	135	133
3	9	14	136	140
1	10	14	114	113
2	10	14	58	58
3	10	14	53	56
1	0	15	120	99
2	0	15	127	118
3	0	15	225	245
4	0	15	91	86
5	0	15	64	92
6	0	15	117	111
0	1	15	155	155
1	1	15	126	129
2	1	15	132	129
3	1	15	137	146
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6	1	15	78	84
7	1	15	77	91
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3	2	15	136	133
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6	2	15	91	101
7	2	15	76	79
8	2	15	43	49
0	3	15	245	246
1	3	15	116	108
2	3	15	107	118
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4	3	15	102	101
5	3	15	112	114
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7	3	15	102	105
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1	4	15	175	177
2	4	15	201	195
3	4	15	150	152
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7	4	15	53	59
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3	5	15	89	93
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6	5	15	70	77
7	5	15	47	42
0	6	15	135	142
1	6	15	100	96
2	6	15	112	113
3	6	15	112	108
4	6	15	88	97
6	6	15	60	64
0	7	15	150	146
1	7	15	55	49
2	7	15	60	64
3	7	15	103	108
4	7	15	83	63
1	8	15	100	104
2	8	15	47	51
3	8	15	63	78
4	8	15	41	34
0	9	15	69	73
1	9	15	83	65
2	9	15	65	66
0	0	16	103	113
1	0	16	176	173
2	0	16	78	73
3	0	16	129	121
7	0	16	54	55
0	1	16	263	255
1	1	16	112	118
2	1	16	86	69
3	1	16	54	61
5	1	16	143	143
6	1	16	130	142
0	2	16	92	94
2	2	16	161	154
6	2	16	111	115
1	3	16	141	148
2	3	16	86	89
3	3	16	97	98
4	3	16	151	151
5	3	16	136	143
6	3	16	87	88
1	4	16	122	122
2	4	16	47	41
3	4	16	163	166
0	5	16	93	91
2	5	16	127	124
3	5	16	50	45
4	5	16	126	126
5	5	16	55	56
6	5	16	92	98
2	6	16	112	123
3	6	16	90	83
0	7	16	51	58
1	7	16	121	119
2	7	16	54	56
3	7	16	53	53
0	8	16	56	57
1	8	16	67	73
3	8	16	78	82
0	9	16	107	106
1	0	17	73	80
2	0	17	200	205
4	0	17	45	46
5	0	17	65	63
6	0	17	100	95
2	1	17	77	75
3	1	17	80	67
4	1	17	91	94
5	1	17	112	114
6	1	17	81	76
0	2	17	115	105
1	2	17	116	121
3	2	17	101	104
4	2	17	92	87
5	2	17	48	47
6	2	17	39	34
0	3	17	87	92
1	3	17	64	66
4	3	17	72	67
1	4	17	84	76
2	4	17	100	104
3	4	17	62	59
4	4	17	51	54
0	5	17	43	43
4	5	17	77	77
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3	6	17	51	58
4	6	17	38	43
0	7	17	116	117
1	7	17	106	102
3	7	17	39	36
0	0	18	90	82
2	0	18	55	52
4	0	18	53	38
1	1	18	73	70
2	1	18	55	41
3	1	18	106	100
4	1	18	42	49
1	2	18	99	106
2	2	18	52	65
3	2	18	46	42
4	2	18	39	39
5	2	18	57	58
0	3	18	93	102
1	3	18	76	80
2	3	18	94	97
3	3	18	62	52
4	3	18	67	71
0	4	18	96	99
1	4	18	82	91
3	4	18	57	63
4	4	18	67	71
1	5	18	93	94
2	5	18	104	104
1	6	18	70	76
2	6	18	42	53
1	0	19	85	94
1	1	19	54	58
2	1	19	61	76
3	1	19	35	34
0	2	19	106	112
1	3	19	56	54
2	3	19	65	71
3	3	19	69	75
0	4	19	51	46
1	4	19	57	62
2	4	19	51	50
0	0	20	35	41
0	1	20	126	130

the ppt collected and crystallized from ether to give 3·9 g (82%) of **14**: m.p. 92–93°; $[\alpha]_D^{25} - 35{\cdot}0°$ (*c* 1,$CHCl_3$); UV, ORD, and CD: see Table 1; NMR (DMSO-d_6) δ2·12 (s,3,NCH_3), 2·40–3·20 (m,4,CH_2CH_2), 3·46, 3·70, 3·72 (3s,9,3 OCH_3), 4·03 (s,1,CH), 6·13 (s,1,aromatic), 6·60–6·90 (m,4,aromatic). (Found: C, 69·30; H, 7·43; N, 3·79. Calc. for $C_{19}H_{23}NO_4$: C, 69·28; H, 7·04; N, 4·25%).

Conversion of **14** *into unnatural cryptostyline* II (**5**). A mixture of **14** (1 g, 0·003 mol) in 20 ml of MeOH was treated with an excess of CH_2N_2 in Et_2O, the volatiles evaporated, the residue suspended in water and extracted with EtOAc. The extract was evaporated and the residue crystallized from Et_2O to give 890 mg (85%) of **5**: m.p. 117–118°; $[\alpha]^{25} - 59{\cdot}1°$ (*c* 1,$CHCl_3$); identical in UV, NMR, ORD and CD with authentic **5**.[1]

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