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STEREOCHEMISTRY, ABSOLUTE CONFIGURATION, AND CRYSTAL STRUCTURE OF *cis*-(4*S*,9*S*,10*R*)-1-(*S*-1-PHENYLETHYL)DECAHYDROQUINOLIN-4-OL. RELATIVE STEREOCHEMISTRY OF CYCLOALKANO-2,3-PIPERIDIN-4-OLS

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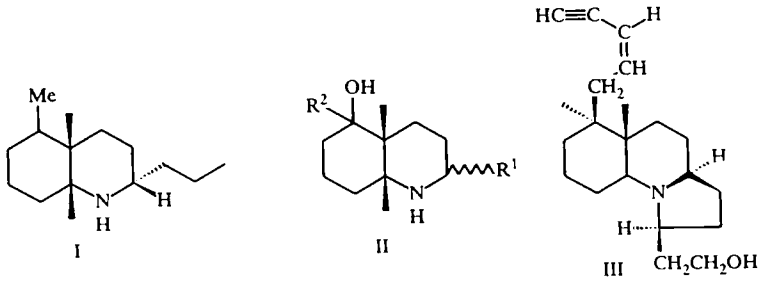
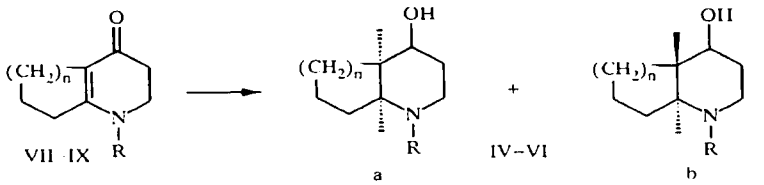
According to x-ray crystallographic data, the cis-*isomer of 1-(1-phenylethyl)decahydroquinolin-4-ol, the predominant product of the stereoselective hydrogenation of 1-(1-*S*-phenylethyl)-$\Delta^{9,10}$-octahydroquinoline-4-one with sodium tetrahydroborate, has the (4*S*,9*S*,10*R*) configuration. The x-ray crystallographic data for this cis-decahydroquinolin-4-ol have been compared with the previously studied piperidin-4-ones of known absolute configuration. The stereochemical rules for cycloalkano-2,3-piperidine-4-ols are discussed.*

The *cis*-decahydroquinoline system, which has not been found previously in natural alkaloids, is the structural basis of a family of neurotoxic alkaloids isolated from the cuticular secretion of the toxic Panamanian tree frog *Dendrobates pumilio*, pumiliotoxin C (I), hydroxypumiliotoxin (II), and hefirotoxin (III) [1].



The development of sterically directed syntheses of simpler analogs of this family of alkaloids was stimulated by the possibility of using them as molecular probes for the study of functionally important components of biological membranes [2].

We have previously described the stereoselective synthesis of optically active analogs of *cis*-decahydroquinoline alkaloids [3]. We showed that asymmetric reduction of the enaminoketones VII-IX with sodium tetrahydroborate gave only one of the diastereomeric pairs of the cycloalkano-2,3-piperidine-4-ols IVa, b to VIa, b in a ratio of 3:1 although three new chiral centers are formed in the cycloalkano-2,3-piperidine system.



VII n = 1, VIII n = 2, IX n = 3 IV n = 1, V n = 2, VI n = 3

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TABLE 1. Coordinates of Nonhydrogen Atoms ($\times 10^4$) in the Structure of Va

Atom	x	y	z	B_{iso}^{eq}, Å^2	x	y	z	B_{iso}^{eq}, Å^2	x	y	z	B_{iso}^{eq}, Å^2
	Molecule VA				Molecule VB				Molecule VC			
O	-1062(9)	2299(3)	613(4)	3,8(2)	2269(8)	2843(3)	5(4)	3,9(2)	-4423(8)	2251(3)	-388(4)	4,2(2)
$N_{(1)}$	-160(10)	789(3)	2155(5)	3,0(2)	2948(10)	4294(3)	1697(5)	3,1(2)	-3714(11)	2315(4)	-3290(4)	3,1(2)
$C_{(2)}$	-1625(14)	1205(5)	2465(7)	4,2(3)	1506(13)	4306(4)	972(6)	3,3(3)	-5189(15)	1910(4)	-2946(7)	4,0(3)
$C_{(3)}$	-2217(13)	1611(4)	1707(7)	3,3(3)	977(14)	3721(4)	627(7)	4,1(3)	-5656(13)	2092(4)	-1911(6)	3,6(3)
$C_{(4)}$	-466(12)	1918(4)	1349(6)	2,9(3)	2783(14)	3404(4)	297(6)	3,5(3)	-3923(14)	2049(4)	-1339(6)	3,5(3)
$C_{(5)}$	418(14)	1147(5)	147(7)	4,0(3)	3995(13)	3018(4)	1870(6)	3,6(3)	-2738(13)	3083(4)	1677(7)	3,9(3)
$C_{(6)}$	1960(17)	735(6)	-154(8)	5,9(4)	5659(15)	3044(5)	2586(7)	4,9(3)	-1027(18)	3444(5)	-2037(8)	5,8(4)
$C_{(7)}$	2490(15)	340(5)	651(8)	5,1(3)	6039(15)	3653(4)	2908(7)	4,5(3)	-640(15)	3249(6)	-3079(7)	5,3(4)
$C_{(8)}$	3145(13)	674(5)	1512(8)	4,7(3)	6420(13)	4050(4)	2046(8)	3,9(3)	-242(13)	2629(5)	-3126(7)	4,4(3)
$C_{(9)}$	1588(12)	1088(4)	1823(6)	3,2(3)	4779(13)	4022(4)	1349(7)	3,4(3)	-1974(13)	2262(5)	-2760(6)	3,8(3)
$C_{(10)}$	1018(12)	1472(4)	1010(6)	3,1(3)	4367(12)	3423(4)	1031(6)	3,0(2)	-2286(12)	2468(4)	-1725(6)	3,2(3)
$C_{(11)}$	243(14)	350(5)	2864(7)	4,3(3)	3281(13)	4838(4)	2189(6)	3,2(3)	-3543(16)	2235(5)	-4348(6)	4,7(3)
$C_{(12)}$	1223(17)	562(6)	3779(7)	5,8(4)	3983(16)	5326(4)	1548(9)	5,7(4)	-2860(20)	1644(6)	-4635(8)	6,9(4)
$C_{(13)}$	-1607(16)	5(5)	2985(8)	5,2(3)	1370(13)	5010(4)	2697(6)	3,0(2)	-5367(14)	2437(4)	-4821(6)	4,0(3)
$C_{(14)}$	-2455(16)	-287(5)	2276(8)	5,7(4)	804(14)	4614(5)	3394(7)	4,4(3)	-3915(14)	3001(5)	-4699(7)	4,5(3)
$C_{(15)}$	-4081(21)	-601(7)	2361(11)	8,1(5)	-926(16)	4769(6)	3900(8)	6,2(4)	-7551(16)	3217(6)	-5143(7)	5,5(4)
$C_{(16)}$	-4879(21)	-639(6)	3219(12)	9,0(6)	-1846(19)	5250(7)	3751(11)	8,7(5)	-8680(16)	2894(7)	-5703(7)	6,8(4)
$C_{(17)}$	-4165(20)	-386(7)	3977(10)	8,4(5)	-1263(17)	5660(5)	3052(9)	6,4(4)	-8167(17)	-2333(7)	-5805(8)	7,5(5)
$C_{(18)}$	-2480(24)	-60(7)	3900(10)	9,0(5)	486(15)	5501(5)	2559(8)	5,3(3)	-6367(17)	2112(5)	-5388(7)	5,7(4)

TABLE 2. Torsion Angles τ (deg) in Structure Va

Angle	Molecule		
	VA	VB	VC
C(9)—N(1)—C(2)—C(3)	59(1)	60(1)	64(1)
C(11)—N(1)—C(2)—C(3)	-173(1)	-169(1)	-168(1)
N(1)-C(2)—C(3)—C(4)	-57(1)	-56(1)	-61(1)
C(2)—C(3)—C(4)—O	179(1)	178(1)	177(1)
C(2)—C(3)—C(4)—C(10)	57(1)	51(1)	61(1)
O—C(4)—C(10)—C(5)	-52(1)	-50(1)	-52(1)
O—C(4)—C(10)—C(9)	180(1)	-177(1)	-175(1)
C(3)—C(4)—C(10)—C(5)	68(1)	75(1)	64(1)
C(3)—C(4)—C(10)—C(9)	-60(1)	-53(1)	-59(1)
C(4)—C(10)—C(5)—C(6)	178(1)	179(1)	178(1)
C(9)—C(10)—C(5)—C(6)	-55(1)	-54(1)	-63(1)
C(10)—C(5)—C(6)—C(7)	55(1)	55(1)	58(1)
C(5)—C(6)—C(7)—C(8)	-57(1)	-55(1)	-57(1)
C(6)—C(7)—C(8)—C(9)	58(1)	56(1)	60(1)
C(7)—C(8)—C(9)—C(10)	-55(1)	-56(1)	-58(1)
C(8)—C(9)—C(10)—C(4)	-176(1)	-177(1)	-175(1)
C(8)—C(9)—C(10)—C(5)	55(1)	55(1)	60(1)
C(5)—C(10)—C(9)—N(1)	-68(1)	-72(1)	-65(1)
C(4)—C(10)—C(9)—N(1)	61(1)	56(1)	61(1)
C(8)—C(9)—N(1)—C(2)	179(1)	174(1)	174(1)
C(10)—C(9)—N(1)—C(2)	-60(1)	-59(1)	-67(1)
C(8)—C(9)—N(1)—C(11)	52(1)	42(1)	51(1)
C(10)—C(9)—N(1)—C(11)	173(1)	169(1)	169(1)
C(2)—N(1)—C(11)—C(12)	-66(1)	-61(1)	-62(1)
C(2)—N(1)—C(11)—C(13)	65(1)	63(1)	68(1)
C(9)—N(1)—C(11)—C(12)	60(1)	68(1)	62(1)
C(9)—N(1)—C(11)—C(13)	-169(1)	-168(1)	-167(1)
N(1)—C(11)—C(13)—C(14)	60(1)	63(1)	59(1)
N(1)—C(11)—C(13)—C(18)	-124(2)	-124(1)	-122(2)
C(12)—C(11)—C(13)—C(14)	-169(2)	-172(1)	-171(2)
C(12)—C(11)—C(13)—C(18)	7(2)	2(1)	8(1)

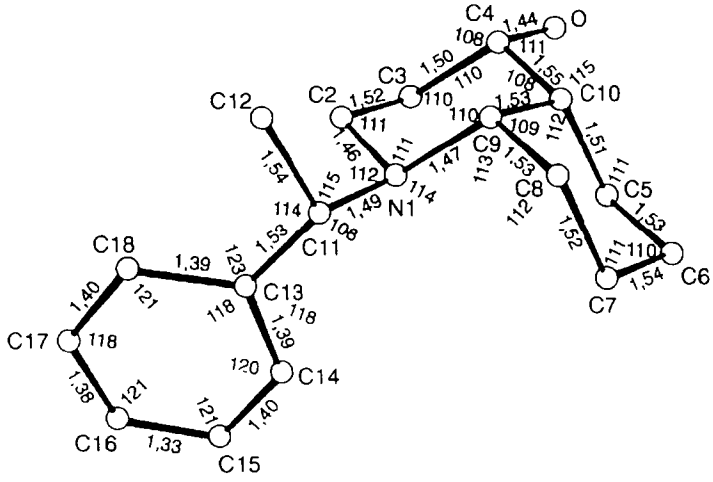


Fig. 1. Structure of molecule VA.

The objective of the present work was to determine the stereochemistry and absolute configuration of the stereoisomers of the decahydroquinolin-4-ols Va and Vb and, coupled with previous results, to estimate the stereochemical rules for obtaining diastereomeric pairs of their bicyclic analog, the cyclopentano- and cycloheptano-2,3-piperidine-4-ols IVa, b and VIa, b.

The diastereomers IVa, b and VIa, b were isolated in pure form via column chromatography or by fractional crystallization of mixtures of isomers.

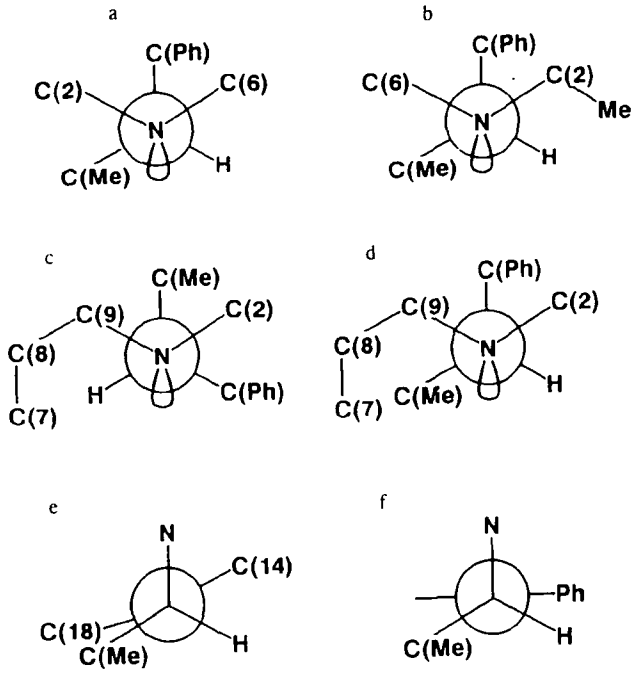


Fig. 2. Newman projections along the exocyclic $N-C_{(13)}$ bond in the structures of X (*a*), XI (*b*), Va(A-C) (*c*), Va(A-C) (*d*, hypothetical model structure) and along the C(MeH)−Ph bond in structures Va(A-C) (*e*) and X, XI (*f*).

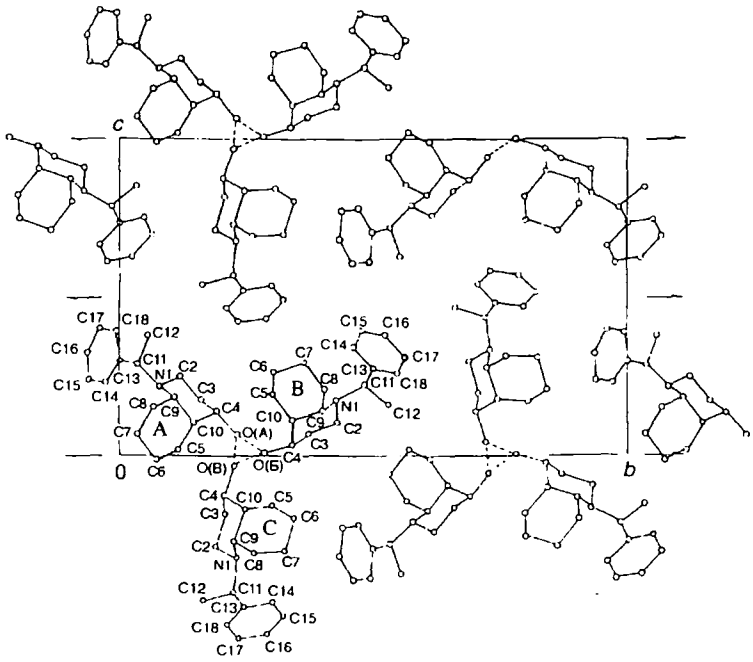


Fig. 3. Projection of the crystal structure of Va on the *yz* plane.

The absolute configuration of the major decahydroquinolin-4-ol Va was established by x-ray crystallography using the 1-(*S*)-phenylethyl groups as the initial chiral marker as had been done in previous structural studies [4, 5].

X-Ray structural analysis showed that the six-membered rings of the three independent molecules VA, VB, and VC (which have uniform structural parameters) are *cis*-fused and have the *chair* conformation (Table 1). The fusion angles, i.e., the interfacial angles between the planes $C_{(2)}C_{(3)}C_{(10)}C_{(9)}$ and $C_{(6)}C_{(7)}C_{(9)}C_{(10)}$, which are planar to 0.01-0.02 Å, are 126°

TABLE 3. Experimental and Calculated ^{13}C Chemical Shifts for *cis*-Va and *trans*-Vb

C Atom number	2	3	4	5	6	7	8	9	10	1'	1''	Solvent
Exp. spectrum of *cis*-Va	43,3	30,0	72,4	21,2	26,1	19,8	29,2	56,1	44,4	52,3	7,8	$CDCl_3$
Calc. spectrum of *cis*-decahydroquinoline with *e*-OH	44,0	31,2	70,6	20,8	25,4	20,5	31,9	52,1	42,5			$CDCl_3$
$\Delta\delta_{exp} - \delta_{calc}$	-0,7	-1,2	1,8	0,4	0,7	-0,7	-1,7	4,0	1,9			
Exp. spectrum of *trans*-Vb	42,9	35,4	74,1	28,1	25,5	25,5	30,1	61,1	50,6	52,8	8,1	$(CD_3)_2CO$
Calc. spectrum of *trans*-decahydroquinoline with *e*-OH	44,8	36,5	73,2	27,7	25,8	25,4	33,5	60,0	50,2			
$\Delta\delta_{exp} - \delta_{calc}$	-1,8	-1,1	0,9	0,4	-0,3	0,1	-3,5	1,1	0,4			

TABLE 4. Relative Intensities of Characteristic Ions in the Mass Spectra of the Isomeric Pairs IVa, b and VIa, b

Isomer	$I\ [M-OH]^+ / I\ [M]^+$	$I\ [M-C_nH_{2n+1}]^+ / I\ [M]^+$	w_M
IVa	0,05	1,7	7,1
IVb	0,07	1,5	3,5
Va	0,04	2,8	5,0
Vb	0,05	2,2	8,0
VIa	0,03	4,5	3,6
VIb	0,03	3,0	4,5

TABLE 5. IR Spectra of the Diastereomeric Pairs IVa, b-VIa, b

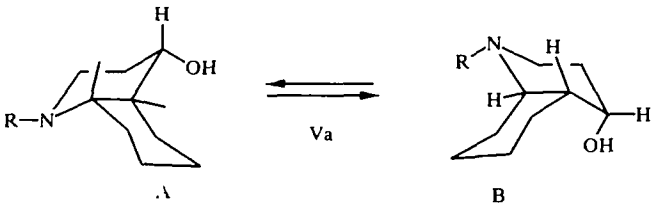
Isomer	ν CO, cm^{-1}			ν_{OH}, cm^{-1},
	strong	medium	weak	in CH_2Cl_2, $c \cdot 10^{-2}$ m
IVa	1065, 1075	970, 1020, 1035	-	3610, 3400
IVb	1035, 1070	945, 975, 1015	1090	3610
cis-Va	1055, 1099	990, 1030, 1040, 1080	-	3610
trans-Vb	1055, 1060	1085	965, 995, 1020, 1030	3610
VIa	1030, 1060, 1085	955, 965, 1020	980	3610
VIb	1045	950, 955, 1030, 1090	1010, 1025,1060	3610

(1A), 127° (1B), and 130° (1C) which are effectively the same as the corresponding angle (128.1°) in the molecule of *cis*-decalin [6]. The 4-hydroxy and 1-phenylethyl groups are in equatorial positions relative to the piperidine ring. The bond lengths and angles in molecules VA-VC are normal and the values in the different molecules are the same within the limits of accuracy of the determination. Differences between the torsion angles (Table 2) are also negligible. The structure of molecule VA is shown in Fig. 1 with bond lengths and angles averaged over molecules VA-VC (error limits are 0.01-0.02 Å and 1-2°). These results show that the major decahydroquinolin-4-ol product VA is the *cis*-isomer and has the (4*S*,9*S*,10*R*) configuration.

As mentioned above the establishment of the absolute configurations of the three new chiral centers — $C_{(9)}$-*S*, $C_{(10)}$-*R* and $C_{(4)}$-*S* — in the molecules VA-VC is based on the known *S* configuration of the 1-phenylethyl substituent on the nitrogen atom, as was the case for the previously studied optically active 1-(*S*-1-phenylethyl)-(3*S*)-(2-cyanoethyl)piperidin-4-one (X) [4] and 1-(*S*-1-phenylethyl)-(2*R*,5*S*)dimethylpiperidin-4-one (XI) [5]. However, there are a series of structural differences in the conformations of molecules VA-VC and the piperidin-4-ones X and XI relative to the exocyclic N–C bonds and the CH(Ph) bonds. In the molecules of the piperidin-4-ones X and XI the 1-phenylethyl substituent is in a restricted (skew) conformation with the phenyl group between the carbon atoms of the piperidine ring (Fig. 2a, b), i.e., the phenyl ring and the unshared pair

of the nitrogen atom are in the *anti*-perpendicular position. In molecules VA-VC the methyl group is in this position which is equivalent to rotation of the phenylethyl group about the exocyclic N−C bond by 120° in comparison with the piperidin-4-ones X and XI (Fig. 2c). This rotation is possibly explained by the steric effect of the methylene group $C_{(7)}H_2$, since the sterically unfavorable juxtaposition of the methyl and methylene groups $C_{(7)}H_2$ would arise if molecule V had a conformation analogous to that observed in the piperidin-4-ones X and XI. In addition, different rotations of the phenyl substituent around the C(HMe)-(Ph) bond are observed in molecules VA-VC and the piperidin-4-ones X and XI. In molecules VA-VC the phenyl group is in the eclipsed position relative to the C-Me bond (Fig. 2e) whereas in the molecules of the piperidin-4-ones X and XI, as already noted the phenyl group is practically perpendicular (in projection) to the C−N bond (Fig. 2f), i.e., it is rotated by 30°. As a consequence of hydrogen bonding of the type O-H...O, in which all three "active" hydrogens participate, the molecules A-C in the structure of molecule V are connected in a spiral chain A...B...C...A with a symmetry close to $[3_1]$, parallel to the *a* axis (Fig. 3): O(A)...O(B) 2.784(9) Å, O(A)...O(C) 2.717 Å, O(B)...O(C) (1 + *x*,*y*) 2.741(9) Å. Thus, a spiral system of cooperative hydrogen bonds with pseudosymmetry $/3_1/$ exists in the structure of V. Similar systems of hydrogen bonds are characteristic of a series of derivatives of phenol [7]. The remaining intermolecular contacts in the structure V are at van der Waals distances.

Conformational analysis of the decahydroquinolin-4-ones Va and Vb in solution was carried out using ^1H and ^{13}C NMR spectra. The equatorial conformation of the hydroxy group at $C_{(4)}$ in the predominant *cis*-isomer Va was established from the size of $^3J_{3a,4a}$ = 11.2 Hz and the *cis*-fusion of the rings follows from the coupling constant $^3J_{4a,10e}$ = 4.4 Hz. The conformational equilibrium is strongly shifted towards conformer A:

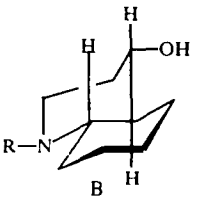


R = (*S*)-CH(CH$_3$)C$_6$H$_5$

Consequently *cis*-decahydroquinolin-4-ol Va exists predominantly in conformation A both in the crystalline state and in solution.

The spatial structure of the minor isomer Vb was established from the excellent agreement of the experimental and calculated values of the ^{13}C chemical shifts for both the major *cis*-Va and minor Vb isomers using increments of the equatorial hydroxy groups for *trans*-decalol-1*e* [8] and ^{13}C data for *trans*-decahydroquinoline [9] (Table 3).

These results show that the minor isomer Vb has *trans*-fusion of the rings and equatorial orientation of the hydroxy group at $C_{(4)}$, i.e., it has conformation B:



R = (*S*)-CH(CH$_3$)C$_6$H$_5$

To determine the absolute configuration of *trans*-decahydroquinolin-4-ol Vb it was first necessary to determine whether the isomers *cis*-Va and *trans*-Vb belong to the same configurational series. To this end we compared the spectroscopic and chiroptical characteristics of the *cis*- and *trans*-isomers Va and Vb with those of optically pure *cis*-(4*S*,9*S*,10*R*)-XII and *trans*-isomers of XIII 1-(*S*-1-phenylethyl)decahydroquinolin-4-ol which were obtained previously by the reduction of *cis*-(9*S*,10*R*)- and *trans*-(9*S*,10*S*)-decahydroquinolin-4-ones of known absolute configuration [10]. The characteristics were practically identical. The major *cis*-isomer Va was identical in melting point, IR spectrum, and chiroptical properties with the *cis*-isomer XII and the analogous data for the minor *trans*-isomer Vb coincided with those for the *trans*-isomer XIII. Consequently the *trans*-isomers Vb and XIII have the same (9*S*,10*S*) configuration. On this basis the isomers *cis*-Va and *trans*-Vb have the same

(*S*) configuration at $C_{(9)}$, but have different *S*-(*trans*) and *R*-(*cis*) configurations at $C_{(10)}$, and belong to the same configurational series.

Thus, complete hydride reduction of the enamine ketone VIII is highly stereoselective and leads to the production of only one stereochemically linked *cis-trans* pair of decahydroquinolin-4-ols with the same (*S*)-configuration at $C_{(9)}$.

The same conclusion is reached from the IR spectra of the *cis-trans* diastereoisomeric pair Va and Vb. The spatial structure in a series of decahydroquinolinols is generally established by application of rules connected with the orientation of the hydroxy group and the type of ring junction and the position of the C−O stretch found for isomers of decalol-1 [10] and steroid alcohols [11] which are extended to decahydroquinolinols. In our case practically the same set of bands was observed in the C−O stretching region for the *cis*- and *trans*-diastereomers: two intense bands in the 1050-1090 cm^{-1} and a medium intensity band at 1085 cm^{-1}. A band for the free OH group was observed at 3610 cm^{-1} in dilute solutions (CH_2Cl_2) of both isomers. In this case it is difficult to describe the C−O stretch as characteristic and it cannot be used to determine the spatial orientation of the OH group or the type of ring junction.

It was not possible to determine the spatial structure of the octahydropyridin-4-ol isomers (cyclopentano-2,3-piperidin-4-ol) IVa, b and the cycloheptano-2,3-piperidin-4-ols VIa, b by ^1H NMR spectroscopy because of their complex conformational behavior. We conducted a comparison of the relative stereochemical behavior of the *cis-trans* pair Va, Vb and the diastereomeric pairs IVa, b and VIa, b using chromatographic mobility, IR spectra, and mass spectral fragmentation. Analysis of the mass spectral fragmentation of *cis*-Va and *trans*-Vb, which have an equatorial OH group at $C_{(4)}$, showed the same probability of loss of OH for both *cis*- and *trans*-isomers. On this basis it can be concluded that the orientation of the hydroxyl groups is also the same, i.e., equatorial, in the isomers IVa and b and VIa and b, since practically identical probability of the loss of the OH group was observed in the pairs of isomeric amino alcohols IVa-VIa and IVb-VIb (Table 4).

On the basis of the probability of formation of the ion $[M - C_nH_{2n+1}]^+$ it may be suggested that the isomers IVa and VIa have *cis*-fusion of the rings, while the isomers IVb and VIb have *trans*-fusion and this is confirmed by the stability of the molecular ions.

Definite regularity in chromatographic mobility was also observed: the major isomers IVa-VIa, which we have assigned to the *cis*-series, have larger R_f values than the *trans*-isomers.

Analogous behavior was observed in the IR spectra of the pairs IVa, b-VIa, b. The IR spectra of dilute solutions of the cycloalkano-2,3-piperidin-4-ols contain a free OH band at 3610 cm^{-1} and C−O stretches in the 1030-1100 cm^{-1} region, corresponding to the C−O stretch of an equatorial OH group (Table 5).

The IR spectrum of the diastereomerically pure octahydropyridin-4-ol IVa has an OH band at 3400 cm^{-1} in addition to the band for the free OH group. Its position did not change in dilute CCl_4 solution up to 70°C. This shows the presence of an intramolecular hydrogen bond in compound IVa which can only occur with *cis*-fusion of the piperidine and cyclopentane rings. This is an additional confirmation that octahydropyridin-4-ol IVa has the *cis*-configuration and that it exists in solution as an equilibrium mixture of conformers A and B in a ratio 2:1 which follows from calculation of the ratio of the integrated intensities of the free OH bands in isomer IVa and the major *trans*-isomer IVb in which there is no intramolecular hydrogen bond.

Thus, a high degree of stereoselectivity of asymmetric reduction is observed in the tetrahydroborate reduction of the enaminoketones VII-IX, during which complete reduction of the enaminoketone groups occurs, to produce one configurationally linked *cis-trans* pair of cycloalkano-2,3-piperidin-4-ol diastereomers IVa, b-VIa, b with the *cis*-isomers IVa-VIa predominating. Stereoselectivity of the asymmetric reduction of the enaminoketones VII-IX increases with increasing size of the carbocycle.

EXPERIMENTAL

Crystals of Va are monoclinic; at 20°C a = 6.9249(5), b = 23.392(2), c = 14.290(1) Å, β = 90.834(7)°, V = 2314.6 $Å^3$, d_{calc} 1.117 g/cm^3, space group $P2_1$, Z = 6, three independent molecules. Unit cell parameters and the intensities of 2458 independent reflections with $F^3 > 3\sigma$ were measured on a Hilger-Watts Y-290 automatic four circle goniometer (λCuK_α, graphite monochromator, $\theta/2\theta$ scanning, $\theta < 66°$). The structure was solved by direct methods using SHELX-86 [12]. Nonhydrogen atoms were refined by full matrix least squares initially in the isotropic approximation and then anisotropically. Hydrogen atoms attached to carbon (except for methyl groups) were placed in geometrically calculated positions. The H atoms of the hydroxy groups appeared during difference syntheses (H atoms of methyl groups were not revealed). All hydrogen atoms were included in the refinement with fixed positions and thermal parameters (B_{iso} = 6 $Å^2$). The final R factor was 0.088 (R_w

= 0.090). Calculations were carried out with an Eclipse S/200 computer using the INEXTL program [13]. Coordinates of the nonhydrogen atoms and their equivalent isotropic factors are given in Table 1.

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