evaporated. The residue was triturated with ethyl acetate and filtered to give 1.7 g (44%) of VId.

The ethyl acetate mother liquor was evaporated, and the residue was triturated with petroleum ether. The precipitate was removed by filtration and recrystallized from heptane to give 0.6 g (16%) of VIIc.

Compounds VIa-c, e, f, and VIIa, b, d, e were similarly obtained (see Table 2).

LITERATURE CITED

1. N. B. Marchenko and V. G. Granik, Khim. Geterotsikl. Soedin., No. 1, 65 (1982).
2. V. G. Granik, E. M. Peresleni, T. D. Kurochkina, A. M. Zhidkova, N. B. Marchenko, R. G. Glushkov, and Yu. N. Sheinker, Khim. Geterotsikl. Soedin., No. 3, 349 (1980).
3. L. N. Yakhontov, D. M. Krasnokut-skaya, E. M. Peresleni, Yu. N. Sheinker, and M. V. Rubtsov, Dokl. Akad. Nauk SSSR, 176, 613 (1967).
4. V. A. Palm, Introduction to Theoretical Organic Chemistry [in Russian], Vysshaya Shkola (1974), p. 242.
5. C. M. Judson and M. Kilpatrick, J. Am. Chem. Soc., 71, 3110 (1949).
6. L. N. Yakhontov, D. M. Krasnokutskaya, E. M. Peresleni, Yu. N. Sheinker, and M. V. Rubtsov, Dokl. Akad. Nauk SSSR, 172, 118 (1967).

STEREOCHEMISTRY OF NITROGEN HETEROCYCLES.

48.* SYNTHESIS, CONFIGURATIONS, AND IR SPECTRA OF 1,2-DIMETHYLDECAHYDROQUINOLINE-5-OL ISOMERS AND THEIR ACETATES

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Three isomers of 1,2-dimethyldecahydroquinolin-5-ol and their acetates with cis and trans fusion of the rings and different orientations of the hydroxy and acyloxy groups were obtained. The correlation of the frequencies of the C—O, O—H, and N^+—H stretching vibrations with the configurations of these compounds was studied. The 1,2-dimethyl-cis-decahydroquinolin-5-ol isomer with a syn orientation of the amino and hydroxy groups exists in a conformation with an intramolecular hydrogen bond, the character of which is determined by the functional state of the amino group (OH...N in the base and N^+—H...O in the hydrochloride). The hydrochloride of the acetate of this alcohol does not form an intramolecular N^+—H...O($COCH_3$) bond.

We have previously obtained four isomeric 2-methyldecahydroquinolin-5-ols by hydrogenation of 2-methyl-5-keto-5,6,7,8-tetrahydroquinoline [2, 3]. Investigations of the chemical transformations and IR spectra made it possible to assign to three of these isomers 2α-methyl-trans-decahydroquinolin-5α-ol[†] (I), 2α-methyl-trans-decahydroquinolin-5β-ol (II), and 2α-methyl-cis-decahydroquinolin-5α-ol (conformation IIIA, with a syn orientation of the amino- and hydroxy groups and an intramolecular hydrogen bond) configurations.

In this paper we describe the synthesis of three isomeric 1,2-dimethyldecahydroquinolin-5-ols (IV-VI) and their acetates (VII-IX) and an investigation of their IR spectra in order to study the correlation between the three-dimensional structures of these compounds and their spectral characteristics.

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[*] See [1] for Communication 47.

[†] The symbols α and β denote trans and cis orientations, respectively, of the substituent relative to 9-H.

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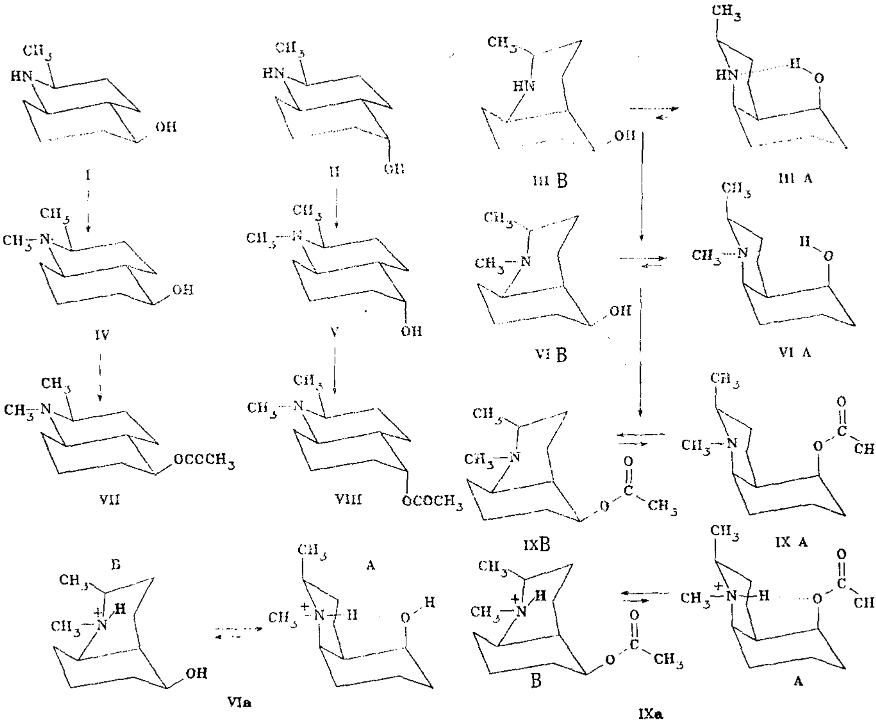
1,2-Dimethyldecahydroquinolin-5-ols IV-VI (Table 1) were obtained in 84-92% yields by Leuckart methylation of isomers I-III. Acetates VII-IX (Table 1) were obtained in almost quantitative yields by acetylation of isomers IV-VI with a mixture of acetyl chloride and acetic anhydride.

The characteristic frequencies of the vibrations of the investigated compounds are presented in Table 2.

All of the 1,2-dimethyldecahydroquinolin-5-ol isomers in the crystalline state (in the liquid state in the case of isomer VI) have an associated hydroxy group that participates in the formation of a hydrogen bond of the OH...N type.

The IR spectra of solutions of the 1,2-dimethyldecahydroquinolin-5-ol bases show that bands of associated hydroxy groups vanish, and, correspondingly, bands of free hydroxy groups appear in the spectra of alcohols IV and V with trans fusion of the rings at solution concentrations of 0.3 mole/liter. The band of an associated hydroxy group does not vanish, and the band of a free hydroxy group does not appear in the spectrum of alcohol VI with cis fusion of the rings when the solution is diluted to 0.005 mole/liter. These results constitute evidence that the hydrogen bond in amino alcohols IV and V is intermolecular and that the hydrogen bond in VI is intramolecular (conformation VIA), as in the corresponding secondary hydroxy amine IIIa [2]. This conformation of amino alcohol VI is stabilized not only by a hydrogen bond but also by an equatorial 2-CH_3 group.



The shift of the frequency of the stretching vibration of the hydroxy group in the formation of a hydrogen bond ($\Delta\nu OH = \Delta OH_{free} - \nu OH_{bond}$) for the first two isomeric alcohols with trans fusion of the rings (IV and V) is considerably greater (452 and 477 cm^{-1}, respectively) than for cis alcohol VI with an intramolecular hydrogen bond ($\sim$340 cm^{-1}). This constitutes evidence for lower strength of the intramolecular hydrogen bond for cis alcohol VI as compared with the intermolecular hydrogen bonds of trans alcohols IV and V and can be explained by taking into account the stereospecificity of the hydrogen bond: In alcohols IV and V the intermolecular OH...N bond is evidently close to linear, since the hydroxy group and the nitrogen atom can occupy an orientation most favorable for the formation of a hydrogen bond and the crystal as a whole (angle O—H...N is 169° in crystals of the 2-methyldecahydroquinol-5-ol isomer with mp 142°C [4]); in the case of cis-amino alcohol VI the three-dimensional structure of conformation VIA, which permits the formation of a hydrogen bond, rigidly localizes in space the oxygen atom and the nitrogen atom with its free electron pair, and the formation of a hydrogen bond can occur only in the chair conformation of the $N{-}C_{(9)}{-}C_{(10)}{-}C_{(5)}{-}O{-}H$ six-membered ring, i.e., angle O—H...N is close to 109°. Significant deviation of the O—H...N atoms from linearity leads to weakening of the hydrogen bond, and this is also manifested in a smaller shift of the νOH_{bond} band for alcohol VI.

TABLE 1. 1,2-Dimethyldecahydroquinolin-5-ol Isomers (IV-VI) and Their Acetates (VII-IX)

Compound	mp, °C [bp, °C (mm)]	R_f	n_D^{20}	d_4^{20}	Found				Yield, %
					C, %	H, %	N, %	MR_D	
IV[a]	84—85[b]	0,57	—	—	72,4	11,6	7,4	—	88
IVa[c]	238—239[d]		—	—			6,4	—	97[e]
V[a]	129—130[b]	0,42	—	—	72,4	11,3	7,8	—	93
Va[c]	202—203[d]		—	—			6,6	—	98[e]
VI[a]	[88—89 (2)]	0,77	1,4975	1,0036	72,0	11,3	7,4	53,58	84
VIa[h]	178—179[d]		—	—			6,3	—	96[e]
VII[f]	[114—115 (3)]	0,88	1,4818	1,0084	69,3	10,6	6,2	63,96	97[g]
VIIa[h]	221—222[d]		—	—			5,2	—	95
VIII[f]	[109—110 (3)]	0,83	1,4821	1,0083	69,0	10,7	6,5	64,00	98[g]
VIIIa[h]	229—230[d]		—	—			5,0	—	94
IX[f]	[118—119 (3)]	0,73	1,4879	1,0319	69,4	10,4	6,0	63,18	98[g]
IXa[h]	224—225[d]		—	—			5,5	—	83

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[a] $C_{11}H_{21}NO$. Calculated: C 72.1; H 11.6; N 7.6%; MR_D 54.06. [b] From ether. [c] $C_{11}H_{21}NO\cdot HCl$. Calculated: N 6.4%. [d] From ethanol—ether. [e] From the base. [f] $C_{13}H_{23}NO_2$. Calculated: C 69.3; H 10.3; N 6.2% MR_D 63.43. [g] From the hydrochloride. [h] $C_{13}H_{23}NO_2\cdot HCl$. Calculated: N 5.4%.

As compared with the equatorial carbocyclic (steroid) alcohols, the axial alcohols have a higher frequency of the O–H stretching vibrations and a lower frequency of the C–O stretching vibrations [5]. In conformity with this rule and previous determinations of the configurations of 2-methyldecahydroquinolin-5-ol isomers [2, 3], the 1,2-dimethyl-trans-decahydroquinolin-5α-ol isomer (IV) with an equatorial hydroxy group has a lower νOH_{free} frequency in solution (3621 cm^{-1} in CH_2Cl_2 and 3637 cm^{-1} in CCl_4) than axial alcohol V (3629 cm^{-1} in CH_2Cl_2 and 3642 cm^{-1} in CCl_4). The correlation for the νC–O absorption bands is hindered by the fact that, whereas this band can be isolated as the most intense band at 1050 cm^{-1} in the 900-1100 cm^{-1} region in the case of equatorial alcohol IV (the most intense band in this region is usually assumed to be the νC–O absorption of secondary cyclic alcohols), in the case of the axial alcohol there are several bands of medium intensity at 990, 1004, and 1080 cm^{-1}. The band at 1045 cm^{-1} is the most intense band in the case of a solution of alcohol VI with an axial hydroxy group (conformation VIA).

For the reliable identification of the νC–O band we recorded the spectra of solutions in CH_2Cl_2 of amino alcohols deuterated at the hydroxy group. This made it possible to confirm that the band at 1050 cm^{-1} for alcohol IV and the band at 1045 cm^{-1} for alcohol VI belong to the νC–O vibrations and to assign the band at 990 cm^{-1} for alcohol V to the νC–O vibration. Thus, the above-indicated correlation between νC–O and the orientation of the OH group (νC–O of the equatorial alcohol is greater than that of the axial alcohol) is also observed for epimeric alcohols IV and V with trans fusion of the rings. It should be noted that the band of medium intensity at 990 cm^{-1} for alcohol V decreases significantly upon deuteration to a greater degree than the intense band at 1050 cm^{-1} for alcohol IV. This constitutes evidence for great specific character of the first band and for complex character of the second with a smaller contribution of the C–O stretching vibration to it.

Although there is no doubt about the axial orientation of the OH group in alcohol VI, the high frequency of the νC–O stretching vibration at 1045 cm^{-1}, which is usually characteristic for equatorial alcohols, seems unexpected at first glance. However, it can be explained by the combined effect of two factors. First, νC–O should have increased as a consequence of the fact that the C–O–H fragment is rigidly localized in a six-membered ring. Second, quantum-mechanical calculations of the interaction of two water molecules show that migration of electrons from the bridged hydrogen atom to the adjacent bond occurs in the formation of a hydrogen bond [6]. In the case of alcohols this sort of migration should lead to a decrease in the electron density on the O–H bond and to an increase in the electron density on the C–O bond, which, in turn, leads to a decrease in the νO–H frequency (which is well known) and should simultaneously lead to an increase in the νC–O frequency, which also occurs in the case of alcohol VI.

The IR spectra of the crystalline hydrochlorides of the 1,2-dimethyldecahydroquiolin-5-ol isomers, like the spectra of the hydrochlorides of 1,2-dimethyldecahydroquinolin-4-ol iso-

TABLE 2. Frequencies of the Characteristics of the Vibrations in the IR Spectra of 1,2-Dimethyldecahydroquinolin-5-ols and Their Acetates

Compound	Aggregate state or solvent	Concn., mole/liter	ν, cm^{-1}				
			C—O	C=O	NH$^+$	OH$_{bond}$	OH$_{free}$
IV	Crystalline		—	—	—	3185	—
	CH_2Cl_2	0,3	1050	—	—	—	3621
	CCl_4	0,01	1052	—	—	—	3637
IVa	Crystalline			—	2570	3316	—
V	Crystalline					3165	—
	CH_2Cl_2	0,3	990	—	—	—	3629
	CCl_4	0,02	991	—	—	—	3642
Va	Crystalline			—	2580	3398	—
VI	Liquid			—	—	3300	—
	CH_2Cl_2	0,3	1045	—	—	3250	—
	CH_2Cl_2	0,03	1045	—	—	3250	—
	CCl_4	0,02	1045	—	—	3300	—
VIa	Crystalline			—	2463	3355	—
	Crystalline*			—	2460	3345	—
	$CHCl_3$	0,1	1040	—	2470	3360	3615 w
	$CHCl_3$	0,01		—		3360	3615 w
VII	Liquid		1244	1734	—	—	—
	CCl_4	0,01	1252	1730	—	—	—
VIIa	Crystalline		1250	1738	2470	—	—
	$CHCl_3$	0,1	1250	1730	2400	—	—
VIII	Liquid		1235, 1249	1735	—	—	—
	CCl_4	0,02	1240, 1257	1735	—	—	—
VIIIa	Crystalline		1248, 1261	1748	2480	—	—
	$CHCl_3$	0,1	1243	1736	2400	—	—
IX	Liquid		1242	1732	—	—	—
	CCl_4	0,01	1256	1733	—	—	—
IXa	Crystalline		1250	1735	2395	—	—
	$CHCl_3$	0,1	1246	1732	2400	—	—

*Suspension in mineral oil.

mers [7], show the existence of a hydrogen bond. The smaller shift of the νOH_{bond} frequency for the hydrochlorides as compared with the bases constitutes evidence for a weaker hydrogen bond. It was therefore assumed that a hydrogen bond of the OH...N type is formed in the bases, whereas a hydrogen bond of the OH...O type is formed in the hydrochlorides [7]. However, a dependence of $\nu O{-}H_{bond}$ on the character of the anion ($\Delta\nu OH$ for hydrochlorides is greater than for hydrobromides) was previously noted in [7], and this provides a basis for the assumption that an O—H...Cl$^-$(Br$^-$) bond rather than an O—H...O bond is realized in crystals of the salts. The N^+—H...anion bonds in crystals of ammonium salts should be similar but even stronger hydrogen bonds. The high intensity of the $\nu N^+{-}H$ stretching vibrations (according to [8], the energy of a hydrogen bond is directly proportional to the square root of the increase in the intensity of the vibration) and their large shift to the long-wave side, which reaches 500-800 cm^{-1}, can be explained precisely by a strong N^+—H...anion hydrogen bond.

The formation of a hydrogen bond of the N^+—H...O type is also formally possible for the hydrochlorides of amino alcohols. The conditions for the formation of this sort of bond are particularly favorable in the case of the hydrochloride of amino alcohol VI with cis fusion of the rings and a syn orientation of the amino and hydroxy groups (conformation VIaA). The high solubility of the hydrochloride of amino alcohol VI in low-polarity solvents such as diethyl ether and chloroform, in which the hydrochlorides of amino alcohols of the trans series are insoluble, also constitutes evidence in favor of this sort of intramolecular hydrogen bond for this compound.

A study of the IR spectra of solutions of cis-alcohol hydrochloride VIa in $CHCl_3$ (0.003-0.1 mole/liter) actually confirmed the existence in it of an intramolecular hydrogen bond (νOH_{bond} 3360 cm^{-1}). In addition to a strong νOH_{bond} band, the spectrum also contained a weak νOH_{free} band at 3615 cm^{-1}; the ratio of these bands remained virtually unchanged when the solution was diluted. This constitutes evidence, on the one hand, for the existence of conformational equilibrium

VIaA $\rightleftarrows$ VIaB (shifted to favor VIaA) and, on the other, for a weaker N^+—H...O bond (νOH_{bond} 3360 cm^{-1}) as compared with the O—H...N bond (νOH_{bond} 3250 cm^{-1}).

Since the formation of a hydrogen bond leads to a decrease in the frequency of the stretching vibrations of the OH and N^+H groups that participate in it, it may be expected that the frequency of the νN^+H stretching vibration for amino alcohol hydrochloride VIa should also be decreased. In fact, a comparison of the νN^+H bands of the amino alcohol hydrochlorides with cis and trans fusion of the rings in the crystalline state (this band is the same for cis-hydrochloride VIa in the crystalline state and in solution) shows that in the case of cis-hydrochloride VIa the frequency of the middle of the νN^+H absorption band is ~100 cm^{-1} smaller than in the case of trans-hydrochlorides IVa and Va, for which the νN^+H frequencies are approximately the same (2570 and 2580 cm^{-1}) as for the hydrochlorides of 1,2-dimethyl-trans-decahydroquinolin-4-ol isomers (2540-2580 cm^{-1}) [7].

A rule, according to which acetates of axial alcohols have a split band of the C—O stretching vibration of the acyloxy radical, whereas acetates of equatorial alcohols have an unsplit band, is known in the steroid series [5]. This rule was applied mechanically to acetates of alcohols of the azocyclic series, although the effects of the introduction of a strongly basic nitrogen atom and a change in the type of crystal lattice from molecular to ionic on passing from the base to the hydrochloride on the form of the νC—O band were not investigated. The effect of the aggregate state of the substrate also was not ascertained.

We have previously studied these problems in series of azocyclic compounds in the case of the acetates of five 1,2-dimethyldecahydroquinolin-4-ol isomers; we showed [9] that the described correlation between the orientation of the acetoxy group and the character of the νC—O band is retained for both the crystalline bases and the hydrochlorides and for solutions of the bases in the case of conformationally rigid alcohols with a trans fusion of the rings. However, in the case of conformationally labile alcohols with a cis fusion of the rings, for which a predominant conformation with an equatorial acetoxy group was expected, the νC—O band was a singlet in the spectra of solutions of the base and the crystalline hydrochloride, whereas it was split in the case of the crystalline base.

A study of the IR spectra of isomeric 1,2-dimethyldecahydroquinol-5-yl acetates (VII-IX) showed that in the case of esters of amino alcohols with a trans fusion of the rings the acetate rule is satisfactorily observed for the liquid base, the crystalline hydrochloride and a solution of the base in chloroform; the acetate (VII) of the equatorial alcohol has a singlet νC—O band, whereas the acetate (VIII) of the axial alcohol has a split band. However, solutions of the acetate hydrochlorides in $CHCl_3$ have an unsplit νC—O band in the case of both the equatorial (VIIa) and axial (VIIIa) alcohols.

Thus a study of the IR spectra of acetates of conformationally rigid alcohols of the azacyclic series that contain an acetoxy group in both the piperidine and cyclohexane rings showed that the acetate rule is applicable for the crystalline and liquid bases and their solutions and crystalline hydrochlorides but possibly inapplicable for solutions of the hydrochlorides.

In the case of the acetate of the cis-amino alcohol the νC—O band is a singlet for base IX and for hydrochloride IXa, as in the case of esters of equatorial alcohols. This result can be interpreted in such a way that replacement of a hydroxy group by an acetoxy group leads to disappearance of the hydrogen bond that stabilizes conformation VIA and that the axial acetoxy group in conformation IXA creates strong steric hindrance, and the molecule is inverted to conformation IXB with an equatorial acetoxy group.

As in the case of 1,2-dimethyl-cis-decahydroquinolin-5-ol hydrochloride (VIa), the formation of an intramolecular hydrogen bond of the N^+—H...O($COCH_3$) type through the proton of the ammonium group is possible in the case of acetate hydrochloride IXa. However, the singlet character of the νC—O band of the acetate hydrochloride constitutes evidence in favor of conformation IXaB rather than IXaA. The fact that the frequency of the νN^+—H band in the spectrum of a solution is the same as in the spectra of acetate hydrochlorides of the trans series VIIa and VIIIa, which do not form intramolecular hydrogen bonds, constitutes evidence for the absence of an intramolecular hydrogen bond in acetate hydrochloride IXa.

The absence of an intramolecular hydrogen bond in acetate hydrochloride IXa, in contrast to the hydrochloride (VIaA) of the alcohol itself, and its primary existence in conformation IXaB are evidently due, on the one hand, to the lower basicity of the ester oxygen atom as compared with the hydroxy oxygen atom and, on the other, to the steric hindrance created by the acetoxy group.

The νC—O absorption band of the acetate bases is symmetrical, whereas it has a shoulder on the low-frequency side in the case of crystalline hydrochlorides VIIa-IXa and solutions of hydrochlorides VIIIa and IXa.

EXPERIMENTAL

The 2α-methyldecahydroquinolin-5-ol isomers I-III, with mp 163-164, 190-191, and 79-80°C, used in this research were obtained as described in [2].

Chromatography was carried out in a thin layer of loose activity II-III alkaline aluminum oxides with elution by dioxane (the isomeric compounds were chromatographed on the same plate).

The IR spectra of the isomeric 1,2-dimethyldecahydroquinolin-5-ols and their acetates were recorded with a UR-20 spectrometer; KBr and KCl pellets, respectively, were used to record the spectra of the crystalline compounds and the hydrochlorides, thin layers between KBr plates were used for the liquid bases, and solutions in CCl_4, $CHCl_3$, or CH_2Cl_2 were used to record the spectra of the bases and (where the solubilities made this possible) hydrochlorides.

1,2-Dimethyldecahydroquinolin-5-ol Isomers IV-VI (Table 1). These compounds were obtained by heating 2-methyldecahydroquinolin-5-ol isomers I-III with formalin and formic acid on a boiling-water bath (10% excesses of formalin and 85% formic acid with respect to the theoretical amounts were used). The reaction mixture was refluxed until carbon dioxide evolution ceased completely (30-40 min), after which it was cooled and treated with potassium carbonate, and crystlline N-methyl derivatives IV and V were separated with a Schott filter, washed with water, dried, and crystallized until they had constant melting points. The aqueous mother liquors were extracted with ether, the extracts were dried, and the solvent was removed to give additional portions of N-methyl derivatives IV and V. The 1,2α-dimethyl-cis-decahydroquinolin-5α-ol isomer (VI), which was an uncrystallized oil, was purified by distillation.

Hydrochlorides IVa-VIa (Table 1) were obtained by neutralization of the bases with a solution of dry hydrogen chloride in anhydrous ethanol with subsequent crystallization.

1,2-Dimethyl-5-acetoxydecahydroquinoline Isomers VII-IX (Table 1). A mixture of 0.1 g of hydrochloride IVa-VIa, 4.5 ml of acetic anhydride, and 4.5 ml of acetyl chloride was refluxed on a water bath for 2 h, after which the precipitate was separated, washed with ether, dried, and recrystallized from ethanol.

For the isolation of bases VII-IX the corresponding hydrochlorides were decomposed in aqueous potassium carbonate solution. The bases were extracted with ether, the extract was dried, the solvent was removed, and the residue was distilled in vacuo.

LITERATURE CITED

1. G. S. Litvinenko, A. A. Espenbetov, and Yu. T. Struchkov, Izv. Akad. Nauk Kazakh. SSR, Ser. Khim., No. 5, 63 (1982).
2. N. Yu. Kuz'mina, Zh. I. Isin, D. V. Sokolov, and G. S. Litvinenko, Izv. Akad. Nauk Kazakh, SSR, Ser. Khim., No. 1, 55 (1981).
3. D. V. Sokolov, N. Yu. Kuz'mina, Zh. I. Isin, and G. S. Litvinenko, Izv. Akad. Nauk Kazakh. SSR, Ser. Khim., No. 4, 59 (1981).
4. A. A. Espenbetov, Yu. T. Struchkov, N. Yu. Kuz'mina, and G. S. Litvinenko, Izv. Akad. Nauk Kazakh. SSR, Ser. Khim., No. 5, 59 (1982).
5. A. R. G. Kohl, in: Elucidation of the Structures of Organic Compounds by Physical and Chemical Methods [Russian translation], A. Weissberger, ed., Vol. 1, Khimiya, Moscow (1967), p. 158.
6. D. Hankins, J. W. Moskowits, and F. Stillinger, J. Chem. Phys., 53, 4544 (1970).
7. O. V. Agashkin, V. I. Artyukhin, G. S. Litvinenko, T. T. Omarov, K. I. Khludneva, D. V. Sokolov, and S. S. Chasnikova, Atlas of IR Spectra of Spatial Isomers of Decahydroquinoline Derivatives [in Russian], Nauka, Alma Ata (1969).
8. A. V. Ioganseh, Dokl. Akad. Nauk SSSR, 164, 610 (1965).
9. K. I. Khludneva, V. V. Sosnova, D. V. Sokolov, and G. S. Litvinenko, Izv. Akad. Nauk Kazakh. SSR, Ser. Khim., No. 2, 43 (1970).