Preparation of Compounds (XX-XXIV). A current of dry HBr was passed for 70-90 min through a solution of 0.5 mmole of the methyl ester of one of compounds (XV-XXIX) for 70-90 min. Then 5 ml of absolute benzene was added and the mixture was evaporated in vacuum at 30°C. The residue was washed with acetone to eliminate traces of anisole and was reprecipitated from ethanol with acetone. The completeness of the elimination of the benzyloxycarbonyl group was checked spectrophotometrically at λ 235-280 nm. The products obtained were passed through a column of silica gel (L 40/100 μ). Elution was performed with butan-1-ol—acetic-acid—pyridine—water (15:3:10:12) buffer solution. The rate of eolution was 12 ml/h. The selected fraction was freeze-dried. The homogeneity of the methyl esters of the TFA derivatives of the peptides obtained was checked by ^{19}F NMR spectroscopy.

SUMMARY

A number of methyl esters of trifluoroacetyl derivatives of peptides forming analogs of the amino acid sequence 33-40 of histone fraction H1 of calf thymus have been synthesized.

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SYNTHESIS AND STUDY OF THE STRUCTURE OF NEW N-SUBSTITUTED 2-METHYL-5-(1-METHYLETHYL)CYCLOHEXYLAMINES

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Original methods of synthesizing N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines by hydroamination reactions of (+)-S-carvone with aliphatic nitriles and the hydroamination of some aldehydes and ketones with (+)-S-carvone oxime have been developed. The optimum conditions for performing these processes has been selected. It has been established by ^{13}C NMR that the reactions studied form a mixture of N-substituted carvo-, isocarvo-, neocarvo-, and neoisocarvomenthylamines in a ratio of 65:20:10:5. As a result of the investigation, 11 secondary amines of the p-menthane series not previously described in the literature have been isolated and characterized. The absolute configurations of the compounds synthesized have been determined.

Amino derivatives of the p-menthane series are compounds with a pronounced pesticidal activity [1, 2]. However, N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines, which are potentially physiologically active substances, have been studied inadequately. This is apparently connected with the fact that the known methods [3, 4] of obtaining these compounds

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TABLE 1. Composition and Yields of the Products of the Hydroamination of Carvone by Nitriles (I-III) and of the Hydroamination of Some Aldehydes and Ketones by Carvone Oxime (IV-XI)

Compound	Composition of the mixture of stereoisomeric amines, %				Total yield of amines, %
	N-substituted carvomenthylamines	N-substituted isocarvomethylamines	N-substituted neocarvomethylamines	N-substituted neoisocarvomenthylamines	
I	61	21	9	9	70,0
II	70	20	8	2	64,2
III	65	20	10	5	61,5
IV	63	19	9	9	64,7
V	64	15	14	7	68,3
VI	68	20	9	3	60,8
VII	65	19	10	6	61,1
VIII	62	18	13	7	58,2
IX	67	18	9	6	69,5
X	65	22	11	2	65,0
XI	60	25	13	2	55,9

are extremely laborious and do not provide the possibility of synthesizing the desired products with any kind of high yield.

As a result of the purposeful study of the possibilities of synthesizing difficultly accessible secondary amines of the p-menthane series from a natural raw material, we have developed a simple and original method of synthesizing N-alkyl-2-methyl-5-(1-methylethyl)-cyclohexylamines [5] which permits the desired products to be obtained in one stage and in preparative yield. For the synthesis of compounds of this type we have used for the first time natural (+)-S-carvone (p-mentha-6,8-diene-2-one) isolated from dill oil by fractional distillation. The essence of the method is as follows: A mixture of carvone and the appropriate nitrile is passed in an apparatus of the flow-through type in a current of hydrogen through a layer of copper-alumina catalyst. The main product of the reaction consists of the corresponding secondary amines. The results of the reaction are given in Table 1.

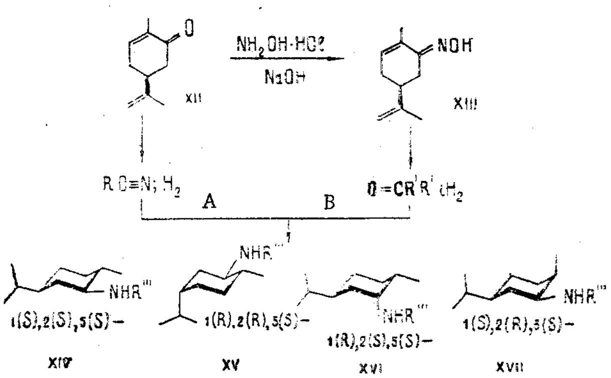
On the basis of literature information on the mechanism of the hydroamination of ketones by nitriles [6, 7], and also the experimental results, it may be assumed that the hydroamination of (+)-S-carvone (XII) with aliphatic nitriles takes place via a stage of the reduction of the nitrile to the primary amine which then condenses with the ketone to form a Schiff's base, latter then being hydrogenated to the corresponding secondary amine (method A). In the course of the reaction, complete reduction of the double carbon—carbon bonds of the carvone takes place and therefore the amines formed as the result of the reaction, having three asymmetric centres (C_1, C_2, and C_5), can exist in the form of four stereoisomeric optically active forms — N-substituted carvo-, isocarvo-, neocarvo-, and neoisocarvomenthylamines (XIV, XV, XVI, and XVII, respectively).

N-Ethyl-, N-propyl-, and N-butyl-2-methyl-5-(1-methylethyl)cyclohexylamines (I, II, and III, respectively) have been synthesized by the method described. However, the wide use of this method of synthesizing secondary amines of the p-menthane series with substituents of different structures on the nitrogen atom is prevented by the limited range of nitriles available industrially.

Experimental results on the reduction of oximes to primary amines under conditions close to the hydroamination reaction [8] have permitted us to use the oxime of (+)-S-carvone (XIII) for the first time as a direct aminating reagent for certain aldehydes and ketones. The reaction was carried out in the following way: A solution of carvone oxime in a carbonyl-containing compound was passed in an apparatus of the flow-through type in a current of hydrogen through a layer of copper-magnesia catalyst.

The oxime reacts with the carbonyl-containing compound by a complex mechanism of concerted hydrogenation and condensation reactions taking place in parallel and in succession on the surface of the catalyst [8, 10]. The main reaction products are N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines.

The results that we have obtained on the hydroamination of carvone oxime by certain aldehydes and ketones are given in Table 1. As can be seen from these results, an increase in the molecular weight and degree of branching of the radicals of the carbonyl compound lead to a fall in the yield of secondary amines.



Method (B) for the synthesis of N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines considerably expands the possibility of obtaining secondary amines of this series, since the wide choice of aldehydes and ketones in the domestic industry permits the synthesis of amines of very different structures. By this method we have synthesized N-(-methylpropyl)-, N-(1-ethylpropyl)-, N-(1-methylbutyl)-, N-(1-methylpentyl)-, N-(1-ethylpentyl)-, N-cyclopentyl-, N-cyclohexyl-, and N-benzyl-2-methyl-5-(1-methylethyl)cyclohexylamines (IV-XI, respectively).

The results of an investigation of the above-described reactions at various temperatures, pressures of hydrogen, and space velocities of passage of the initial mixture have permitted the optimum conditions for their performance to be determined. It can be seen from the results given in Fig. 1 that the maximum yield of secondary amines in the hydroamination of carvone by aliphatic nitriles was achieved in the range of temperatures of 220-230°C, at a hydrogen pressure of 15 atm, and a space velocity of 0.3 h^{-1}.

The optimum conditions for the performance of the reductive amination of certain aldehydes and ketones by carvone oxime are a temperature of 230-250°C, a hydrogen pressure of 15 atm, and a space velocity of 0.2 h^{-1}.

The stereochemical composition of the reaction products was studied by nuclear magnetic resonance on carbon-13 nuclei. It was established that as a result of the reactions described a mixture of four stereoisomeric secondary amines was formed, the ratio of N-substituted carvo-, isocarvo-, neocarvo-, and neoisocarvomethylamines being approximately 65:20:10:5 in all cases. The main reaction products was the N-substituted carvomenthylamine isomer with the triequatorial arrangement of the substituents in the cyclohexane ring, which is the most thermodynamically stable. The least stable is obviously the neoiso isomer, the amount of which in the reaction products was about 5%. From this it may be concluded that the stereochemistry of the hydroamination reaction is controlled by thermodynamic factors.

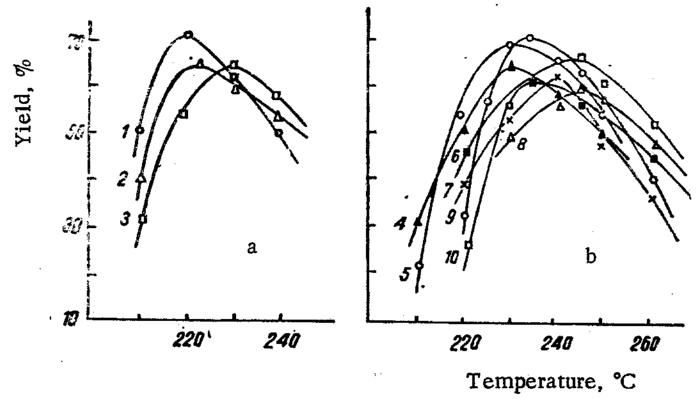


Fig. 1. Dependence of the yield of N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines on the reaction temperature: a) hydrogen pressure 15 atm, space velocity 0.3 h^{-1}, catalyst 15% Cu/Al_2O_3 + 2% LiOH; b) pressure of hydrogen 15 atm, space velocity 0.2 h^{-1}, catalyst 36% Cu/MgO. The numbers on the curves correspond to the numbers of the samples in Table 2.

TABLE 2. Properties of the N-Substituted 1(S),2(S),5(S)-2-Methyl-5-(1-methylethyl)cyclohexylamines

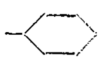
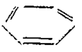
Compound	R	bp, °C (p, mm Hg)	d_4^{20}	n_D^{20}	MR_D		$[\alpha]_D^{20}$, deq	Found, %			Formula	Calculated, %		
					found	calc.		C	H	N		C	H	N
I	$-C_2H_5$	118—120 (15)	0,7869	1,4581	63,58	63,64	+24,3	78,62	13,84	7,24	$C_{12}H_{25}N$	78 61	13,74	7,64
II	$-C_3H_7$	129—131 (15)	0,7426	1,4602	72,82	72,87	+21,2	79,18	13,99	7,30	$C_{13}H_{27}N$	78,11	13,79	7,10
III	$-C_4H_9$	139—141 (15)	0,7071	1,4612	82,06	82,11	+22,9	79,67	13,66	6,80	$C_{14}H_{29}N$	79,54	13,82	6,62
IV	$-CH(CH_3)C_2H_5$	138—140 (15)	0,7898	1,4562	72,78	72,87	+21,3	79,71	13,90	6,85	$C_{14}H_{29}N$	79,54	13,82	6,62
V	$-CH(C_2H_5)_2$	149—151 (15)	0,7515	1,4591	82,01	82,10	+22,0	80,10	13,92	6,41	$C_{15}H_{31}N$	79,92	13,86	6,21
VI	$-CH(CH_3)C_3H_7$	143—145 (15)	0,7496	1,4581	82,06	82,10	+21,2	80,15	13,98	6 37	$C_{15}H_{31}N$	79,92	13,86	6,21
VII	$-CH(CH_3)C_4H_9$	155—157 (15)	0,7188	1,4602	91,26	91,34	+21,4	80,31	13,91	5,93	$C_{16}H_{33}N$	80,26	13,89	5,85
VIII	$-CH(C_2H_5)C_4H_9$	161—163 (15)	0,6941	1,4628	100,53	100,58	+20,3	80,45	13,96	5,75	$C_{17}H_{35}N$	80,55	13 91	5,52
IX		151—152 (15)	0,8946	1,4795	70,64	70,67	+28,3	80,76	13,12	6,42	$C_{15}H_{29}N$	80,64	13,08	6,27
X		162—164 (15)	0,8998	1,4821	75,23	75,29	+24,9	80,87	13,26	6,08	$C_{16}H_{31}N$	80,94	13,15	5,90
XI	$-CH_2-$	159—161 (15)	0,8869	1,5122	83,05	83,12	+20,8	83,41	11,31	6,05	$C_{17}H_{27}N$	83,20	11,09	5,71

TABLE 3. ^{13}C Chemical Shifts of Some N-Substituted 2-Methyl-5-(1-methyl)cyclohexamines

Compound	^{13}C Chemical shifts																
	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}
I. N-Ethylcarvomenthylamine	63,4	38,1	34,8	29,3	43,2	36,3	32,9	19,9	19,7	19,2	41,3	15,9	—	—	—	—	—
N-Ethylisocarvomenthylamine	58,5	34,4	27,9	25,3	38,9	31,0	30,6	20,4	20,4	18,5	41,6	15,8	—	—	—	—	—
N-Ethylneocarvomenthylamine	57,6	36,5	29,2	29,5	36,6	33,5	32,5	19,7	19,9	18,6	42,3	15,8	—	—	—	—	—
N-Ethylneoisocarvomenthylamine	59,4	31,1	30,7	23,5	43,9	32,0	33,0	19,9	20,1	10,9	41,0	15,9	—	—	—	—	—
II. N-Propylcarvomenthylamine	63,4	38,0	34,8	29,3	43,5	36,1	32,9	19,7	19,9	19 2	49,1	23,8	11,9	—	—	—	—
N-Propylisocarvomenthylamine	58,5	34,4	28,0	25,3	39,0	31,0	30,5	20,4	20,4	18,5	49,5	24,2	11,9	—	—	—	—
N-Propylneocarvomenthylamine	57,6	36,5	29,2	29,6	36,6	33,5	32,5	19,7	19,9	18,6	50,1	23,7	11,9	—	—	—	—
N-Propylneoisocarvomenthylamine	59,5	31,1	30,7	23,5	43,9	31,9	33,0	19,9	20,1	10,9	48,9	23,5	11,9	—	—	—	—
III. N-Butylcarvomenthylamine	63,5	38,2	34,8	29,3	43,5	36,3	33,0	19,7	19,9	19,2	47,0	32,9	20,7	14,1	—	—	—
N-Butylisocarvomenthylamine	58,6	34,4	27,9	25,3	38,9	31,0	30,6	20,4	20,4	18,5	47,2	33,0	20,7	14,1	—	—	—
N-Butylneocarvomenthylamine	57,7	36,5	20,9	29,7	36,6	33,5	32,5	19,7	19,9	18,7	47,8	33,0	20,7	14,1	—	—	—
N-Butylneoisocarvomenthylamine	59,6	31,2	30,7	23,5	43,8	31,9	33,0	19,9	20,1	10,9	46,7	33,0	20,7	14,2	—	—	—
V. N-(1-Ethylpropyl)carvomenthylamine	60,7	39,8	35,3	29,5	44,0	38,0	33,1	19,7	20,1	19,6	57,1	26,4	9,1	27,5	10,5	—	—
N-(1-Ethylpropyl)isocarvomenthylamine	55,6	35,4	28,1	25,5	39,1	31,8	31,1	20,3	20,4	18,6	57,4	26,9	9,8	27,5	10,4	—	—
N-(1-Ethylpropyl)neocarvomenthylamine	54,7	37,0	29,5	30 3	37,1	34,2	32,9	19,8	20,1	19,0	57,8	26,4	9,4	27 3	10,8	—	—
N-(1-Ethylpropyl)neoisocarvomenthylamine	56,2	31,4	31,4	23,5	44,2	32,0	33,2	19,9	20,1	11,2	56,1	26,2	9,6	27,4	10,3	—	—
XI. N-Benzylcarvomenthylamine	62,7	38,7	35,0	29,4	43,6	36,5	33,0	19,7	20,0	19,4	51,1	141,9	128,4	128,4	126,6	128,4	128,4
N-Benzylisocarvomenthylamine	58,1	34,3	27,9	25,3	38,7	30,9	30,8	20,3	20,3	18,4	51,7	141,8	128,4	128,4	126,6	128,4	128,4
N-Benzylneocarvomenthylamine	57,0	36,5	29,2	29,9	36,8	33,1	32,6	19,8	19,8	18,8	52,1	141,8	128,4	128,4	126,6	128,4	128,4
N-Benzylneoisocarvomenthylamine *																	

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*The carbon chemical shifts were determined from mixtures of the four isomers. The ^{13}C chemical shifts of N-benzylneoisocarvomenthylamine could not be determined because it was present in the mixture in such a small amount.

From the mixtures of stereoisomeric secondary amines obtained as a result of the reaction we succeeded in isolating the main components of the mixture of N-substituted carvomenthylamines by preparative GLC. The properties of the compounds obtained are given in Table 2.

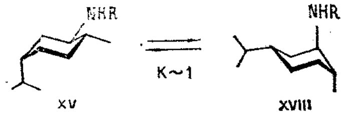
In view of the fact that the isomers have close physicochemical properties and are not separated into individual compounds by the usual methods, it was impossible to isolate the other stereoisomeric amines in the pure form.

The structures of the N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines synthesized were shown by IR, mass, ^{13}C NMR spectroscopy, and also by spectropolarimetry.

The infrared spectra of the secondary amines of the p-menthane series investigated have a characteristic absorption band in the 3300-3400 cm^{-1} region which is due to the stretching vibrations of a >NH group. Bands at 1720 and 1665 cm^{-1} are due to the symmetrical and antisymmetrical deformation vibrations of a secondary amino group, and those at 3000-2800, 1460, 1365, 1260, 1240, 1170, 1120, and 1015 cm^{-1} to the vibrations of the bonds of the structural fragments of the p-menthane carbon skeleton.

The mass spectra of all the compounds obtained included peaks corresponding to the molecular ions M^{+}. Subsequent fragmentation under the action of electron impact took place with the cleavage of the C_2-C_{10} and C_5-C_7 bonds with the formation of the (M − 15) and (M − 43) ions that are characteristic for compounds of the p-menthane series [11].

As a criterion for the determination of the structure and configuration of the N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines from their ^{13}C NMR spectra we used a comparison of the experimental and calculated chemical shifts of the isomeric carvomenthylamines. As model compounds we took the isomeric carvomenthols, the chemical shifts of which have been measured previously [12]. The good agreement of the chemical shifts of the carbon atoms of the ring (with the exception of the position of attachment of the polar substituent and of the carbon atoms connected with it) in the alcohols and the amines permits a conclusion to be drawn concerning the identity of the conformational compositions of individual pairs of isomers. This was in any case to be expected in view of the close conformational energies of the —OH and —NHR groups. On the basis of the chemical shifts of the carvomenthols and the N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines one can only consider the isocarvomenthylamines to be conformationally inhomogeneous, the constant of the equilibrium between the conformers with the equatorial (XVIII) and the axial (XV) positions of the isopropyl groups being 1.



XV XVIII

The measured chemical shifts of the ^{13}C nuclei of the isomeric N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines are given in Table 3. We must mention the fairly large difference in the screening of the carbon atoms in the diastereotopic ethyl groups in N-(1-ethylpropyl)-2-methyl-5-(1-methylethyl)cyclohexylamine (V).

The optical rotatory dispersion curves of the N-substituted carvomenthylamines form smooth positive curves.

The study by ^{13}C NMR of the secondary amines synthesized and also the determination of their optical activities has permitted the conclusion that, as the result of the hydroaminations described, stereoisomeric N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines are formed in which the S configuration at C_5 has been retained. The absolute configurations of the secondary amines of the p-menthane series obtained have been determined on this basis.

EXPERIMENTAL

The hydroamination of (+)-S-carvone with bp 86-88°C (6 mm), d_4^{20} 0.9660, n_D^{20} 1.4953, $[\alpha]_D^{20}$ +69.1° (ethanol) by aliphatic nitriles was carried out as described previously [5].

The hydroamination reaction of certain aldehydes and ketones with (+)-S-carvone oxime having bp 72°C $[\alpha]_D$ +39.2° (ethanol, synthesized by Cassan's method [13]) was carried out in the following way: A solution of the oxime and of the appropriate carbonyl-containing compound was passed in an apparatus of the flow-through type at temperatures of 220-270°C under a pressure of hydrogen of 12-18 atm with a space velocity of 0.15-0.35 h^{-1} through a layer of catalyst which consisted of 36% of copper deposited on magnesia.

The secondary amines were isolated from the reaction mixture by comparative GLC in a chromatograph at a temperature of the column thermostat of 120-130°C in a column (3 m × 12-4 mm) filled with Chromaton N-AW-DMCS (0.2-0.25 mm) which had been washed with a 5% solution of KOH in ethanol and had been impregnated with 20% of Apiezon L.

All the compounds isolated by preparative GLC were subjected to vacuum distillation before study. The purity of the substances obtained was determined by the GLC method on a LKhM-7A chromatograph with programming of the temperature from 90 to 200°C using a column (2 m × 0.5 mm) filled with Chromosorb W (60-80 mesh) and crystalline KOH (9:1) impregnated with Apiezon K (12%).

The infrared spectra of the compounds studied were recorded on a UR-20 spectrometer in the range of frequencies of 400-3800 cm^{-1} with slit program 4 at a rate of scanning of 60 cm^{-1}/min. The compounds were used in the form of liquid films between KBr plates.

Mass spectra were recorded on a Varian MAT-311 instrument at a cathode emission current of 1000 mA and an energy of the ionizing electron of 10 eV. The temperature of evaporation of the samples was 150-200°C, and the temperature of the ion source 200°C.

^{13}C NMR spectra were recorded on a WH-90 spectrometer (Bruker) with a resonance frequency for ^{13}C of 22.62 MHz under conditions of complete decoupling from protons. For interpretation, spectra with external resonance decoupling were recorded. The concentration of the solutions was 1:4 by volume in $CHCl_3$ + $CDCl_3$ [sic]. The deuterated solvent was used for stabilization. The chemical shifts of the ^{13}C nuclei were determined relative to an internal standard — tetramethylsilane. All the spectra were recorded under integrating conditions of detection using a memory volume of 8 K for the real part of the spectrum. The width of the spectrum was 1600-2400 G. The measuring pulse for ^{13}C was 8 μsec (approximately 60°).

The optical rotatory dispersion curves were recorded on a "Jasco J-20" spectropolarimeter in ethanolic solution (concentration 0.1 M) in the range of frequencies of 250-600 nm.

SUMMARY

Methods have been developed for the synthesis of N-substituted 2-methyl-5-(1-methylethyl)cyclohexylamines by the hydroamination of (+)-S-carvone oxime.

It has been established that this forms mixtures of N-substituted carvo-, isocarvo-, neocarvo-, and neoisocarvomenthylamines.

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