

MASS-SPECTROMETRIC STUDY OF SUBSTITUTED 4-ALKYLDECAHYDRO-4-QUINOLOLS AND DECAHYDRO-4- QUINOLONES

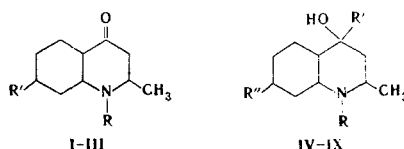
N. S. Vul'fson, V. G. Zaikin,
A. A. Bakaev, A. A. Akhrem,
and L. I. Ukhova

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The primary fraction of the total ion current in the mass spectra of decahydro-4-quinolones and 4-alkyl-2-methyldecahydro-4-quinolols containing various substituents in the 1, 4, and 7 positions is due to fragments with ammonium ion structures.

We have previously shown the applicability of mass-spectrometric methods for the solution of stereochemical problems in trans-fused 4-alkyl-decahydro-4-quinolols by establishment of the configuration of the 2 and 4 centers in the indicated series. However, in none of these papers did we deal in detail with the pathways of fragmentation of the indicated compounds under the influence of electron impact. In the present paper we therefore deemed it expedient to examine in detail the fragmentation of decahydro-4-quinolones and 4-alkyldecahydro-4-quinolols, especially because only one paper [5] has been devoted to a detailed study of decahydroquinolines by means of deuterium labeling, and there are only a few studies [6-8] in which in addition to stereochemical problems, the principal pathways of fragmentation of the stereoisomers of decahydroquinoline and 2-methyl-N-alkyldecahydro-4-quinolols are briefly examined. The subjects of our investigation were substituted 2-methyldecahydro-4-quinolones (I-III) and 4-alkyl-2-methyl-trans-decahydro-4-quinolols (IV-IX), data from the mass spectra of which are presented in Table 1. It should be noted that the ketones (I-III) examined in this paper have a 2e-methyl group and the alcohols (IV-VII) have a 2e-methyl-4e-hydroxy configuration, whereas the stereochemistry of the alcohols (VIII-IX) has not yet been established.

The mass spectra of the investigated compounds are characterized by a relatively small number of intense characteristic peaks.



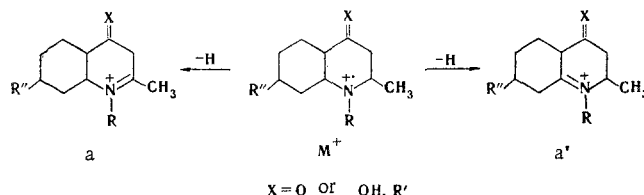
I R=CH₃, R'=H; II R=R'=H; III R=R'=CH₃; IV, VI, VIII, IX R=CH₃; V R=C₂H₅;
VII R=H; IV-VII R''=H; VIII R''=CH₃; IX R''=C₄H₉; IV, V, VIII, IX a R'=
=C=CH; VI, VII a R'=-C≡C-CH=CH₂; IV, V, VIII b R'=CH=CH₂; VI, VII b
R'=n-C₄H₉; IV, V, VIII c, IX b R'=C₂H₅; IV, VIII d R'=COCH₃

The nitrogen-containing fragments, the stability of which is due to the ammonium ion structure, make the principal contribution to the total ion current, whereas the peaks of the hydrocarbon fragments are very small.

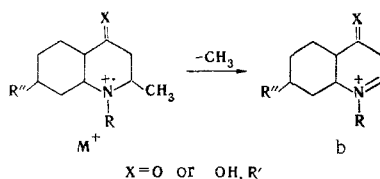
M. M. Shemyakin Institute of Bioorganic Chemistry, Academy of Sciences of the USSR, Moscow. A. V. Topchiev Institute of Petrochemical Synthesis, Academy of Sciences of the USSR, Moscow. Institute of Bioorganic Chemistry, Academy of Sciences of the Belorussian SSR, Minsk. Translated from *Khimiya Geterotsiklicheskikh Soedinenii*, No. 7, pp. 962-968, July, 1975. Original article submitted June 25, 1974.

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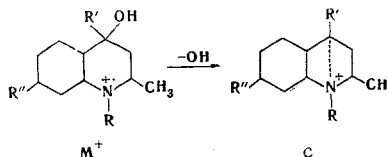
Both in the case of ketones I-III and in the case of alcohols IV-IX, the molecular ion peaks have medium intensities. They are always accompanied by $[M-1]^+$ ion peaks corresponding to the ejection of a hydrogen atom, most likely from C_2 or C_9 (ions a and a'). As shown in [7], in the case of N-trideuteromethyl-2-methyldecahydro-4-quinolol a D atom is not ejected from the M^+ ion, and this makes it possible to also assume the absence of loss of a hydrogen atom from substituent R attached to the nitrogen atom for I-VI, VIII, and IX.



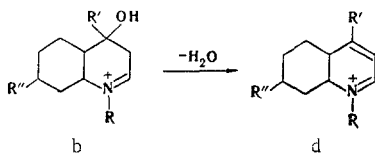
The $[M-15]^+$ Ion (b). This ion is formed as a result of elimination of a 2- CH_3 group. The peaks of these ions are among the maximum peaks in the spectra, and they are sometimes the principal peaks. Judging from the mass spectra of N-ethyl-2-methyl derivatives Va, b, in which the M-29 ion peak is practically completely absent, ejection of an N-methyl group during the formation of an $[M-15]^+$ ion also should not occur in the case of I-IV, VI, VIII, and IX. At the same time, the partial participation of an N-ethyl group in the formation of $[M-15]^+$ ions can be assumed for the N-ethyl derivatives Va, b (see [8]).



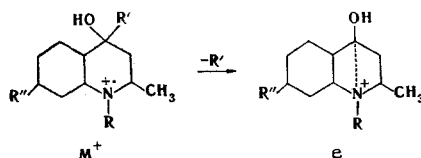
The $[M-17]^+$ Ion (c). In contrast to carbocyclic alcohols, the hydroxyl group of which is, as a rule, split out under the influence of electron impact along with an additional hydrogen atom in the form of water, the fragmentation of the molecular ions of N-heterocyclic alcohols is limited in most cases to ejection of an OH group in the form of a radical. This is associated with the fact that the ejection of an OH radical entails the formation of a stable ammonium ion of the c type. The $[M-17]$ peak is insignificant in the spectra of the 4-ethyl (IVc, Vc, VIIIc, and IXe), 4-n-butyl (VIb, VIIb), and 4-acetyl derivatives (IVd, VIIIId), but it increases in the spectra of the 4-vinyl (IVb, Vb, VIIIb), 4-ethynyl (IVa, Va, VIIIa, and IXa), and 4-vinylethynyl (VIa, VIIa) derivatives and approaches the molecular ion in intensity and sometimes even exceeds it. This is explained by the considerable polarization of the C-O bond in the allyl position with respect to the double or triple bond in substituent R' attached to C_4 .



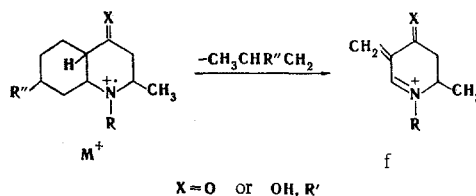
The $[M-15-18]^+$ Ion (d). Whereas the molecular ions of alcohols IV-IX undergo practically no dehydration, the $[M-15]^+$ ions (b) are capable of cleavage of water, evidently because of the possibility of the formation of the conjugated system of double bonds.



The $[M-R']^+$ Ion (e). Because of the difficulties involved in the cleavage of $C_{sp^3}-C_{sp^2}$ and $C_{sp^3}-C_{sp^4}$ bonds, the corresponding ions practically are not formed in the case of vinyl, ethynyl, and vinylethynyl alcohols. However, the ejection of radical R' from the molecular ions of compounds with $R' = C_2H_5$ (IVc, Vc, VIIIc and IXb), $R' = n-C_4H_9$ (VIb, VIIb), or $R' = COCH_3$ (IVd, VIIIId) takes place quite readily, and the peaks of the corresponding ions (e) are frequently the dominant ones in the spectra.



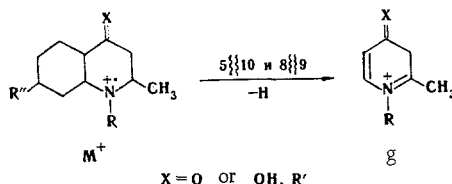
The $[M - 43]^+$ Ions for $R'' = H$, the $[M - 57]^+$ Ion for $R'' = CH_3$, and the $[M - 99]^+$ Ion for $R'' = \text{tert-C}_4H_9$
 (f). The mass number of these ions is independent of the size of substituent R'' , and thus a portion of the hydrocarbon ring, including the C_7 atom, is eliminated during their formation. In the case of N-methyldecahydroquinoline it has been shown by means of deuterium labeling [5] that the corresponding ion no longer contains the C_6 atom but does contain the heterocyclic ring in its entirety. Ion f is most likely formed via a classical mechanism including initial cleavage of the α -C-C bond with respect to the nitrogen atom and migration of the angular 10-H atom to the neutral fragment.



The mass spectrum of the 3,3,10- D_3 analog of III serves as a rather convincing confirmation of the proposed mechanism for the formation of ion f . A doublet of $M - 58$ and $M - 57$ peaks in a ratio of 3:1 is observed in the spectrum of this deuterium derivative. It is unlikely that the D atom leaves the 3 position during the formation of the $M - 58$ ion; it most likely leaves the 10 position, as shown in the scheme. The presence of an $M - 57$ peak in the mass spectrum of the labeled ketone constitutes evidence for partial (less characteristic) formation of ion f under consideration with participation of an H atom from the other positions of the heterocyclic ring.

In the case of ketol IVd, the peak of the $[M - 43]^+$ ion (f) may be superimposed on the peak of the ion with the same m/e value but corresponding to splitting out of a $COCH_3$ radical from the molecular ion (in this case, on the peak of ion e). However, it follows from the mass spectrum of the 4- $COCD_3$ analog of IVd that in the case of this ketol, ion f is practically not formed, whereas the $M - 43$ peak is due to the energetically more advantageous loss of a $COCH_3$ radical (ion e). It should be noted that the preferred character of the competitive detachment of a 4- $COCH_3$ group from M^+ as compared with the formation of rearranged ion f due to fragmentation of the carbocyclic ring is also confirmed by the mass spectrum of ketol VIIIId, in which one of the maximum peaks is the peak of the $M - 43$ ion (ion e), whereas the peak of the $M - 57$ ion (f) is very insignificant.

The $M - 57]^+$ Ion for $R'' = H$, the $[M - 71]^+$ Ion for $R'' = CH_3$, and the $[M - 113]^+$ Ion for $R'' = \text{tert-C}_4H_9$
 (g). The peaks of these ions are of low intensity in all of the spectra. As in the case of ions f , ions g contain atoms of the heterocyclic ring but do not include the C_7 atom. Ion g is most likely formed through cleavage of the 5-10 and 8-9 bonds with migration of a hydrogen atom from the charged fragment.



Ions with m/e 44 ($R = H$), 58 ($R = CH_3$), and 72 ($R = C_2H_5$). Corresponding peaks of medium intensity are present in the mass spectra of all of the investigated substances. The mass numbers of these ions depend on the size of the substituent attached to the nitrogen atom and these ions consequently contain the nitrogen atom, the substituent attached to it, and, most likely, the C_2 atom with the 2-methyl group and have
 $R-NH=CH-CH_3$ structure.

Thus it is apparent that the principal fragments in the mass spectra of the compounds under consideration arise mainly directly from the molecular ions. In this situation, the intensities of the daughter ion

TABLE 1. Mass Spectra of I-IX (intensities in percent of Σ)
50

m/e	I	II	III	IVa	IVb	IVc	IVd	Va	Vb	Vc	VIa	VIb	VIIa	VIIb	VIIIa	VIIIb	VIIIc	IXa	IXb	
53	1.6	3.0	1.6	2.5	1.3	0.8	1.0	0.3		0.3	1.1		1.5	0.2	1.4	0.8	0.6	0.6	0.8	0.5
54	1.9	3.2	3.1	1.1	1.1	0.7	0.7	0.2		0.4	1.2	0.2	1.5	0.5	0.3	0.6	0.3	0.3		
55	3.2	4.2	4.0	1.9	6.8	1.5	1.6	1.2	1.3	1.2	3.1	0.4	2.3	1.6	1.2	6.7	2.0	2.5	1.4	1.6
56	1.6	2.7	1.7	2.4	2.6	1.5	2.0	2.0	0.8	2.3	2.9	0.6	2.1	1.5	2.2	2.8	1.7	1.9	1.8	3.0
57	0.6	0.4	3.4	1.4	1.7	3.5	1.4	0.5	0.3	3.7	2.8	2.5	1.3	2.8	0.7	1.2	7.7	1.3	3.1	5.6
58	0.3			5.8	7.2	6.8	9.0	0.7	0.2	0.6	5.2	5.8	1.0	1.3	7.0	10.0	14.6	6.4	6.1	7.7
59				0.2	0.3	0.4	0.3	0.2			0.3	0.2		0.4			0.7	0.6		
65	0.2	0.6	0.4	0.7	0.5	0.3	0.3			0.2			1.3	0.2			0.3			
66		0.5	0.3	0.4	0.3	0.2	0.2				1.1		0.6				0.2			
67	1.9	3.1	2.0	1.3	1.5	1.3	1.1	0.2	0.6	0.9	1.0	0.3	1.9	1.1	0.8	0.7	0.8	0.9	0.6	0.6
68	2.3	2.9	1.9	1.2	0.9	0.6	0.7			0.4	2.6	0.2	1.4	0.6	0.4	0.5	0.4	0.6	0.2	0.4
69	1.8	2.5	1.3	0.5	0.6	0.7	0.7	1.1		0.8	1.1	0.4	1.2	1.6	0.3	0.6	2.6	0.9	0.7	0.5
70	2.5	3.0	1.0	1.1	1.6	1.4	0.7	0.9	0.5	1.0	1.2	0.6	2.1	1.0	0.8	0.8	1.0	0.7	0.8	1.3
71	0.2	0.2	0.2	0.5	0.5	0.4	0.5	1.1	0.5	0.7	1.3	0.9	0.8	1.3	0.5	0.4	0.2	0.6	0.4	0.5
72			0.3	0.5	0.8	0.7	0.7	2.1	3.6	6.2	1.1	0.5			0.8	0.7	1.0	0.8	0.7	0.5
73									0.2	0.4	0.6	0.3			0.7					
77	1.6	1.0	0.8	1.1	0.8	0.6	0.5	0.2	0.4	0.2	1.7		1.8	0.3	0.7	0.5	0.4	0.3	0.6	
78	0.2	0.2		0.3	0.3	0.2	0.2				0.4		0.6							
79	0.9	1.9	0.9	1.2	1.1	1.0	0.9	0.9		0.6	4.7	0.2	4.3	0.8	0.7	0.6	0.4	0.7	0.5	
80	0.8	1.2	0.2	0.7	0.3	0.4	0.3	0.3		0.2	0.6		0.8	0.3						
81	1.9	4.1	1.3	1.7	1.4	1.2	1.3	1.4	0.4	1.2	1.8	0.4	1.7	1.6	0.8	1.1	0.8	2.2	1.0	0.6
82	2.3	4.8	2.0	1.5	1.7	1.3	1.0	1.3	0.3	0.6	2.2	0.3	2.2	1.5	1.0	1.0	0.6	0.8	1.2	1.2
83	0.8	1.0	0.8	0.5	0.8	0.7	0.5	0.2	0.3	0.8	0.6	0.7	1.2				0.6	0.3	0.3	0.6
84	0.3	0.3	1.9	0.8	1.0	0.7	0.7	0.6	0.5	0.6	1.4	0.3	1.2	0.6	1.2	1.7	1.1	1.6	0.6	0.7
85					0.3	0.3	0.3	0.3	0.2	0.2	0.8	2.2	0.4	2.0		0.2	0.4	0.3		0.4
91	0.3			0.8	0.7	0.5	0.5	0.6	0.2	0.2	1.4		1.7	0.5	0.4	0.3	0.4		0.3	0.3
93	0.4	0.2	0.3	0.2	0.5	0.3	0.3	0.6	0.4	0.3	0.5		0.7	0.4	0.2	0.2	0.3	0.4		0.2
94	1.0	0.8	1.0	0.6	0.7	0.5	0.5	0.3		0.2	2.1	0.2	1.2	0.5	0.3		0.2	0.2	0.4	0.2
95	3.0	0.3	2.6	0.4	0.5	0.4	0.4	1.1	0.3	0.5	0.7	0.3	0.7	0.9	0.7	0.8	0.8	1.1	0.6	0.5
96	3.3	3.0	2.1	1.6	2.5	2.2	1.2	0.9	0.3	0.4	1.9	0.7	1.1	1.2	0.8	0.8	0.5	0.7	0.3	0.2
97	0.8	0.8	0.3		0.4	0.3	0.5	0.4	0.2	0.2	0.5	0.6	1.2	0.3	0.3	0.2	0.3			
98	0.2			0.3	0.7	0.2	0.6	0.5	0.4	0.3	0.6	0.3	0.8	1.0	0.2	0.5	0.3	0.6		
105				0.3	0.3	0.3	0.3	0.4			0.6		0.9	0.2						
106				0.2		0.2					0.3		0.6							
107		0.2		0.4	0.3	0.3	0.4	0.6	0.3	0.2	1.1		0.9	0.4						
108	0.5	0.4	1.0	0.6	0.5	0.5	0.6	0.6	0.2	0.2	0.7	0.2	0.7	0.3	0.2	0.2	0.3		0.2	
109		0.9	0.2	0.4	0.3	0.2	0.4	1.8	0.2	0.3	0.3	0.3	0.6	0.6			0.2			0.2
110	2.3	1.5	3.7	0.8	0.8	0.7	0.9	1.1	0.8	0.9	0.8	0.3	0.8	0.7	0.3	0.5	0.6	0.6	0.5	0.2
111	0.4	0.7	0.3	0.2	0.3	0.2	0.5	0.6	0.3	0.2	0.4	0.4	0.4	0.4						
112	0.2			0.3	0.5	0.4	0.8	0.3	0.4	0.2	0.6	0.4	0.3	0.7	0.2	0.7		0.5		
117				0.2							0.3		0.7							
120				0.2	0.4		0.3				0.3		0.7							
123	0.6		0.8		0.2	0.2	0.2	0.8	0.3	0.1	0.3	0.3	0.5	0.4						0.2
124	7.6	1.3	63.3	0.5	0.8	0.6	0.7	0.4	0.5	0.5	0.7		0.5	1.7	3.2	0.2	0.4	0.2	0.6	0.2
125	8.0	3.4	0.9					0.7	0.2				0.3	0.3	1.1		0.3	0.2		
126	0.9	0.3	0.3	0.2	0.5	1.5	1.2	0.4	0.4	0.4	0.4	0.9	0.6	0.9	0.3	0.4	1.0	1.4		0.7
134				0.9	0.3		0.2	0.2			0.4	0.8	0.8	0.3	0.6					
135				0.3	0.2		0.4	0.4	0.2		0.2	0.2	0.4	0.3						
136	0.2			0.5	0.9	0.6	0.5	0.5	0.2	0.2	0.4	0.2	0.9	0.4	0.2	0.4			0.2	
137				0.3	0.4	0.2	0.3	0.2	0.4	0.2		0.3	0.6	0.5					0.3	
138	4.6	2.5	12.0	1.6	1.5	1.2	9.0	0.9	0.7	0.5	1.2	1.3	0.4	0.4	0.2	0.6	0.6	0.6	2.1	3.0
139	0.7	2.1	1.7	0.2	0.2	0.3	1.8			0.2	0.2	0.4	0.3				0.2	0.4	0.3	0.6
140			0.3	0.2		0.3	0.4	0.4	0.4	1.4	0.4	1.0	0.3				0.2	0.2		
148				0.2	0.4	0.2	0.5	1.3			0.4		0.7	0.2					0.2	
149						0.2	1.4	0.4			0.2	0.2	0.4	0.3						
150		1.0		0.7	0.7	0.8	0.5	0.6	0.4		0.3	0.3	0.5	2.6	0.5				0.4	
152		5.5	3.7	0.2	0.8	0.2	0.4	0.8	1.0	1.2		0.3	1.4	0.6	0.4	1.1	1.0	2.3		
153	1.9	0.6	0.3		0.2			0.2		0.2	0.2		0.3					0.4		
154	0.2							0.1	0.4				0.6	0.2			0.2			
162				0.3	0.4		0.3	0.2			0.4		1.2							0.5
163				0.6	1.6		0.2		0.2				0.2							
164	1.0			15.0	0.8	1.5	3.2	0.9	0.3		0.3	1.0	1.8	16.4	0.5	0.5	0.5	17.4	0.3	
165				2.0	0.8	1.1	0.4	0.2	0.4	0.2		0.3	0.3	0.4	1.6	0.2		1.8		
166	7.5	0.2		0.8	12.6	0.5	0.9		1.0		0.8	0.4		0.6		10.6		0.4		0.5
167	1.0	3.3		0.1	1.7	0.4	0.4		1.3		0.2	0.2				1.0				
168		0.3		0.2	0.9	3.5	0.2				0.4	0.5		18.2		2.2			4.3	
169														2.6		0.3			0.5	
174				0.9								0.4								
176				0.2	0.7			0.3			0.9		4.8							
177					0.2	0.2		0.8	0.6		0.2		0.7	0.2						
178				0.3	1.4		15.3	0.8	0.9				0.3				0.3			
179					0.2	0.4	2.2	0.5	0.2					0.6						
180	0.7		12.2		0.4	0.3	0.5	0.5	19.0	0.4		0.5					0.2	0.9	0.2	
181	6.0		1.3	0.2	0.2	0.7	0.4		2.6	0.3		0.2								
182	0.6			0.2	2.3	22.0	19.0		0.7	5.1		30.0		1.3				0.6		
183				0.3	2.6	2.5				0.7		4.1		0.3				0.3		
186													0.9							
188				0.3				2.3							1.9					
189				0.5				0.3							0.2					
190				3.4	0.2		0.3	0.9		11.6	0.3		0.3			0.6				
191				1.4	0.4		0.2	0.3		1.6	0.2									
192				14.3	2.5		2.5	0.3	0.9	0.2	0.2		0.2	0.8			1.2			
193				2.1	0.5	0.4	0.9	0.3		0.2						0.2	0.2			0.3
194			0.2	0.2	12.0	0.5	0.3		0.3		0.7							0.4		
195		4.3			1.7	0.3		0.4	1.1											
196		0.8			0.7	12.0														

TABLE 1. (continued)

m/e	I	II	III	IVa	IVb	IVc	IVd	Va	Vb	Vc	VIa	VIb	VIIa	VIIb	VIIIa	VIIIb	VIIIc	IXa	IXb
197						1.6			0.3	4.0									
200											1.5	0.3	1.2			0.2	1.4	3.0	
201											0.5		1.7						
202								0.3			0.3		4.2						
203								0.4					0.8						
204								5.8				0.2	8.4						
206				0.9			0.2	26.0	4.4			0.7		0.3	5.0	0.2		3.8	1.4
207				4.0			0.3	2.9	4.8			0.2		0.6	24.6	2.5		0.5	0.4
208				0.5	0.5		1.0	0.4	22.0	0.7				0.8	3.6	0.6			
209					3.1		0.2		3.2	0.8				0.3	0.2	26.5	0.3		
210					0.4	0.6	4.7	0.4	12.0			0.3		11.0		3.2	6.6		1.6
211						2.8	0.7		0.8	3.0		0.2		1.9		0.6	3.4		
212						0.4			0.4	0.2				0.3			0.4		
213									1.2										
216											9.1								
217											1.7								
218											17.0		0.9						
219											2.6		3.8						
220								0.7			0.2		0.8						
221								5.0							0.8	5.3			
222								0.9	0.8			1.3			0.8	0.4		0.6	
223							0.4		7.5			0.2				2.7			
224							0.4		1.2	0.6		14.0		0.8		0.6	0.4	18.2	
225							5.7			4.4		2.4		2.4			3.2	3.1	
226							0.9			0.9		0.2		0.5			0.5	0.5	
230																		1.5	
231																		0.4	
232											0.6								
233											8.0								
234											1.2								0.6
238												0.4						0.6	12.2
239												3.4						5.3	1.5
240												0.5						0.9	
246																			1.7
247																			0.4
248																			19.6
249																			5.2
250																			0.3
252																			26.0
253																			4.2
254																			0.3
262																		0.4	
263																		5.1	
264																		0.7	
266																			0.5
267																			4.0
268																			0.8

and the molecular ion can be considered to be proportional to their concentrations, and the intensity ratio can be considered to be the rate constant of the corresponding monomolecular reaction. The use of precisely these values has previously enabled us to determine the configuration of alcohols IV-VII on the basis of an analysis of the mass spectra [1-4].

EXPERIMENTAL

The mass spectra were obtained with an MKh-1303 spectrometer equipped with a system for direct introduction of the samples into the ion source at vaporization temperatures of 40-50° and an ionizing electron energy of 70 eV.

The 3,3,10-D₃ analog of III and the 4-COCD₃ analog of IVd were obtained by refluxing solutions of 10 mg of III or IVd, respectively, and a small amount of sodium in a mixture of 1 ml of D₂O, 1 ml of EtOD, and 1 ml of tetrahydrofuran for 8 h.

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