THE MASS SPECTRA AND STRUCTURES OF BIQUINOLINYL SYSTEMS

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The mass spectra of methyl-substituted biquinolinyls have been studied and the stabilities of their molecules to electron impact have been determined. It is assumed that the decomposition of the systems studied under the action of electron impact takes place predominantly from the excited molecule, which exists in the form of the trans or the cis isomer, and proceeds through a stage of the formation of rearrangement ions consisting of multiring condensed systems. A difference has been observed in the probability of the formation of doubly charged ions. A scheme of identification is given.

The present work is a continuation of investigations begun previously on the characteristics of the decomposition of bisheterocyclic compounds on interaction with ionizing electrons [1, 2]. A method for the direct hetarylation of organic compounds developed by one of us [3], based on the recombination of heteroaromatic radical anions (in particular, quinoline radical anions) formed in the one-electron reduction of nitrogenous bases by active metals, frequently leads to mixtures of the corresponding biquinolinyl structures, the identification of which with the aid of UV, IR, and PMR spectroscopy is a fairly complex and frequently insoluble task. Thus, the development of a scheme of identification of biquinolinyl systems, and also a number of problems connected with the transfer of conjugation from one aromatic nucleus of a bicyclic system to another, the influence of the mutual position of the heterocycles on the nature of the decomposition under electron impact, and the change of the intensity of the peaks of the doubly charged ions as a function of the position of the nuclei present considerable theoretical and practical interest.

We have considered the mass spectra of the following series of biquinolinyls: 2,2'-biquinolinyl (I), 4,4'-dimethyl-2,2'-biquinolinyl (II), 7,7'-dimethyl-2,2'-biquinolinyl (III), 8,8'-methyl-2,2'-biquinolinyl (IV), 4,4',2,3'-biquinolinyl (V), 6,6'-biquinolinyl (VI), 2-(1-methyltetrahydro-6-quinolinyl)quinoline (VII), 3-(1,2-dihydro-2-quinolinyl)quinoline (VIII), 4-(1,2-dihydro-2-quinolinyl)quinoline (IX), and 2-methyl-4-(2-methyl-1,2-dihydro-4-quinolinyl)quinoline (X).

The mass spectra of the compounds studied (I-X) were taken on an SN-6 mass spectrometer with a system for the direct introduction of the samples into the ion source at an energy of the ionizing electrons of 70 eV and a temperature of the ionization chamber of 180°C. The purity of the substances synthesized was checked chromatographically and was $\geq 98.5\%$.

The stability to electron impact (W_M)* of systems (I-X) is determined by the following factors: 1) the coplanarity of the molecule as a whole; 2) the mutual positions of the quinoline nuclei relative to one another; and 3) the positions of the methyl substituents in the rings of the quinoline nucleus. The greatest role is probably played by the first factor, since in such systems, where the internuclear bond is not a

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*W_M is the stability of a molecule to electron impact, represented by the ratio of the intensity of the polyisotopic peak of the molecular ion to the total ion current.

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TABLE 1. Mass Spectra of Biquinolinyl Systems at an Energy of the Ionizing Electrons of 70 eV (in percentages of the maximum peak in the spectrum)*

Compound I

29 (3,8), 39 (3,9), 41 (8,4), 42 (2,9), 43 (7,8), 51 (3,0), 54 (2,1), 55 (8,2), 56 (3,0), 57 (5,0), 60 (3,1), 63 (2,0), 67 (3,3), 68 (2,1), 69 (4,3), 70 (2,5), 71 (2,7), 73 (2,9), 75 (4,6), 76 (3,9), 77 (5,4), 81 (2,8), 82 (2,0), 83 (2,3), 84 (2,0), 85 (2,0), 91 (2,2), 95 (2,9), 97 (2,7), 98 (2,9), 100 (2,4), 101 (9,3), 102 (6,0), 113,5 (1,2), 114 (2,9), 114,5 (1,0), 127 (4,3), 127,5 (2,8), 128 (20,5), 128,5 (2,3), 129 (4,3), 154 (2,3), 155 (2,9), 227 (3,5), 228 (3,7), 229 (2,4), 253 (4,0), 255 (64,7), 256 (100,0), 257 (19,3), 258 (2,3), $W_M = 31,3$.

Compound II

89 (2,1), 115 (6,5), 116 (3,2), 127 (1,2), 128 (1,9), 133,5 (1,1), 134 (1,7), 134,5 (3,6), 135 (1,3), 140 (3,5), 141 (3,3), 141,5 (2,0), 142 (8,4), 142,5 (1,7), 258 (2,2), 267 (2,2), 268 (5,7), 269 (22,4), 270 (7,8), 281 (3,2), 282 (4,4), 283 (19,0), 284 (100,0), 285 (22,4), 286 (2,6), $W_M = 34,0$.

Compound III

29 (2,1), 31 (6,3), 39 (8,4), 41 (4,2), 43 (4,7), 45 (2,1), 51 (3,7), 56 (3,2), 57 (3,7), 63 (4,7), 65 (3,7), 71 (2,1), 75 (2,1), 76 (2,1), 77 (2,4), 88 (2,1), 89 (7,9), 91 (2,4), 101 (2,1), 102 (2,1), 114 (3,2), 115 (7,4), 116 (18,4), 117 (8,9), 127 (2,4), 128 (5,8), 129 (5,3), 130 (2,9), 135 (4,2), 140 (3,2), 141 (10,0), 141,5 (3,6), 142 (16,3), 142,5 (3,3), 143 (34,2), 144 (3,7), 168 (2,4), 169 (3,2), 170 (3,4), 215 (2,1), 228 (2,1), 240 (2,1), 241 (3,7), 242 (2,61), 253 (3,4), 254 (3,7), 255 (3,4), 256 (3,2), 258 (2,4), 266 (2,9), 267 (5,5), 268 (7,9), 270 (7,4), 279 (2,1), 280 (2,6), 281 (6,8), 282 (12,1), 283 (52,6), 284 (100,0), 285 (27,4), 286 (5,8), $W_M = 18,1$.

Compound IV

31 (2,0), 39 (2,5), 41 (2,4), 43 (2,4), 56 (3,7), 57 (2,2), 63 (2,4), 75 (2,0), 77 (2,0), 89 (4,4), 101 (2,5), 102 (2,3), 114 (3,4), 115 (12,3), 116 (4,9), 120,5 (1,5), 121 (1,7), 121,5 (1,3), 126 (1,2), 127 (2,4), 127,5 (1,5), 128 (4,9), 128,5 (1,6), 129 (2,4), 133,5 (1,9), 134 (3,5), 134,5 (3,7), 135 (4,8), 135,5 (1,2), 140 (5,6), 140,5 (1,5), 141 (7,4), 141,5 (3,9), 142 (16,9), 142,5 (2,3), 143 (2,3), 169 (2,3), 241 (2,1), 254 (2,2), 255 (5,3), 256 (3,2), 267 (3,0), 268 (5,5), 269 (24,2), 270 (36,8), 271 (7,2), 281 (2,4), 282 (5,7), 283 (24,5), 284 (100,0), 285 (21,7), 286 (2,8), $W_M = 23,2$.

Compound V

38 (12,2), 40 (2,0), 43 (2,2), 49 (4,7), 50 (7,8), 51 (2,3), 61 (3,6), 62 (10,4), 63 (3,3), 64 (5,2), 74 (2,9), 75 (6,7), 76 (3,9), 77 (6,0), 87 (2,7), 88 (3,6), 89 (10,4), 90 (2,6), 91 (2,4), 101 (3,2), 102 (2,7), 113 (4,1), 114 (6,1), 114,5 (1,1), 115 (24,3), 116 (13,1), 117 (2,4), 120,5 (3,4), 121 (2,0), 121,5 (1,1), 126,5 (1,3), 127 (4,9), 127,5 (2,4); 128 (7,3), 128,5 (2,0), 133,5 (3,9), 134 (17,8), 134,5 (14,8), 135 (3,6), 139 (2,4), 140 (13,0), 140,5 (3,2), 141 (10,4), 141,5 (7,0), 142 (28,7), 142,5 (5,5), 143 (3,1), 167 (2,1), 168 (2,1), 169 (4,7), 241 (3,2), 242 (2,2), 254 (4,7), 255 (4,5), 256 (3,5), 258 (2,5), 267 (5,2), 268 (15,7), 269 (70,5), 270 (16,5), 271 (2,3), 279 (2,1), 280 (3,2), 281 (8,7), 282 (11,3), 283 (50,4), 284 (100,0), 285 (26,5), 286 (3,0), $W_M = 13,6$.

Compound VI

29 (8,5), 38 (2,2), 39 (8,1), 40 (2,6), 41 (14,4), 42 (6,3), 43 (13,3), 45 (2,6), 50 (2,8), 51 (4,1), 52 (2,6), 53 (3,1), 54 (3,2), 55 (12,2), 56 (5,4), 57 (7,8), 60 (4,3), 63 (3,5), 65 (2,6), 67 (6,5), 68 (4,3), 69 (6,6), 71 (4,4), 73 (4,4), 75 (4,3), 76 (3,4), 77 (4,4), 78 (3,4), 79 (3,9), 83 (5,4), 84 (4,1), 88 (3,7), 88,5 (1,8), 89 (2,5), 91 (4,8), 95 (4,0), 97 (5,0), 98 (5,0), 99 (5,0), 101 (7,1), 101,5 (2,2), 113,5 (2,8), 114 (6,9), 114,5 (2,6), 115 (3,1), 127 (3,5), 127,5 (3,1), 128 (12,6), 128,5 (2,9), 129 (4,0), 143 (2,2), 220 (4,6), 226 (2,9), 227 (6,3), 228 (6,2), 229 (3,5), 253 (5,0), 254 (7,9), 255 (33,3), 256 (100,0), 257 (20,9), 258 (2,9), $W_M = 11,8$.

Compound VII

29 (26,5), 38 (7,6), 39 (28,2), 41 (3,3), 42 (19,4), 43 (10,4), 50 (9,4), 51 (11,8), 52 (11,2), 53 (11,8), 54 (13,5), 55 (44,7), 56 (20,6), 57 (21,8), 63 (10,6), 64 (8,2), 65 (10,6), 67 (21,8), 68 (15,9), 69 (26,5), 70 (16,5), 71 (20,0), 73 (17,6), 75 (10,0), 76 (7,6), 77 (14,7), 78 (11,8), 79 (15,3), 83 (21,8), 91 (17,6), 92 (8,8), 93 (10,0), 95 (15,3), 97 (17,1), 114,5 (6,5), 115 (10,6), 115,5 (4,1), 116 (6,5), 117 (7,6), 128 (52,9), 129 (17,6), 130 (8,8), 131 (7,1), 135 (8,8), 136 (11,8), 137 (12,3), 155 (10,6), 156 (14,7), 157 (9,4), 183 (7,6), 184 (10,6), 255 (8,8), 256 (4,1), 263 (7,6), 264 (9,4), 265 (27,0), 266 (61,9), 267 (31,2), 268 (10,6), 269 (13,5), 270 (7,1), 271 (10,6), 272 (15,3), 273 (56,2), 274 (100,0), 275 (23,5), 276 (5,9), $W_M = 4,5$.

Compound VIII

29 (2,9), 31 (5,1), 39 (5,2), 41 (4,4), 42 (2,9), 43 (3,6), 50 (5,7), 51 (10,2), 52 (4,4), 55 (2,2), 56 (5,7), 63 (5,8), 64 (2,2), 65 (2,2), 74 (3,6), 75 (9,5), 76 (10,2), 77 (24,0), 78 (6,6), 87 (4,4), 87,5 (2,9), 88 (3,6), 88,5 (2,2), 89 (5,7), 90 (2,2), 91 (3,6), 101 (22,6), 103 (8,0), 113 (2,2), 113,5 (5,7), 114 (10,9), 114,5 (5,8), 115 (5,8), 115,5 (1,5), 126 (4,4), 127 (9,5), 127,5 (5,1), 128 (68,9), 129 (40,8), 129,5 (4,4), 130 (56,3), 131 (8,0), 154 (13,8), 155 (4,4), 226 (2,2), 227 (5,8), 228 (7,3), 229 (5,1), 230 (14,4), 243 (2,2), 244 (2,9), 252 (3,6), 253 (2,9), 254 (30,6), 255 (56,3), 256 (100,0), 257 (87,6), 258 (20,4), 259 (4,4), $W_M = 2,2$.

TABLE 1 (continued)

Compound IX

29 (14,2), 31 (16,6), 39 (8,3), 40 (2,4), 41 (15,9), 42 (7,4), 43 (8,1), 45 (19,6), 51 (5,0), 52 (2,0), 53 (3,7), 55 (17,6), 56 (6,3), 57 (21,6), 60 (13,5), 67 (6,8), 68 (3,7), 69 (13,1), 70 (5,0), 71 (13,1). 73 (7,4), 77 (6,8), 78 (2,8), 79 (2,6), 81 (8,5), 85 (11,1), 87 (3,5), 88 (4,8), 89 (5,2), 91 (2,8), 94 (2,2), 95 (5,7), 101,5 (2,4), 102 (7,4), 102,5 (2,2), 107 (2,4), 107,5 (3,1), 108 (2,4), 108,5 (4,6), 113 (4,1), 113,5 (2,8), 114 (16,6), 114,5 (6,1), 115 (15,3), 155,5 (6,1), 116 (5,0), 127 (4,4), 127,5 (2,0), 128 (38,8), 128,5 (4,8), 129 (16,3), 129,5 (4,1), 130 (11,5), 142 (3,7), 157 (2,0), 215 (3,1), 216 (3,9), 224 (2,4), 228 (8,5), 229 (5,0), 230 (49,2), 231 (3,5), 241 (4,6), 242 (9,6), 243 (12,6), 244 (14,2), 245 (2,8), 255 (8,9), 256 (49,6), 257 (41,1), 258 (100,0), 259 (25,5), $W_M = 10{,}6$.

Compound X

31 (3,7), 36 (12,6), 38 (4,4), 39 (3,7), 41 (2,6), 42 (2,2), 43 (2,2), 51 (2,6), 56 (3,0), 63 (3,0), 77 (3,7), 89 (3,0), 101 (3,7), 102 (2,6), 103 (2,0), 114 (4,1), 114,5 (2,6), 115 (5,2), 115,5 (1,1), 120,5 (3,3), 121 (2,6), 121,5 (2,6), 122 (1,9), 122,5 (1,9), 127 (3,7), 127,5 (1,1), 128 (5,6), 128,5 (1,1), 129 (1,5), 130 (4,8), 133,5 (1,9), 134 (2,6), 134,5 (3,0), 135 (9,3), 135,5 (4,1), 139 (2,6), 141 (2,0), 141,5 (1,5), 142 (5,6), 142,5 (2,2), 143 (14,1), 143,5 (2,6), 144 (13,6), 193 (3,3), 194 (6,3), 214 (2,6), 215 (2,6), 216 (2,0), 217 (3,0), 226 (3,3), 227 (3,7), 228 (4,4), 229 (3,0), 230 (4,1), 231 (2,0), 240 (3,0), 241 (4,8), 242 (4,4), 243 (7,4), 244 (7,4), 245 (5,9), 255 (9,6), 256 (13,3), 257 (5,9), 268 (6,3), 269 (12,2), 270 (15,9), 271 (73,3), 272 (16,3), 273 (3,3), 283 (3,3), 284 (12,6), 285 (18,5), 286 (100,0), 287 (24,8), 288 (3,0), $W_M = 13{,}7$

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* With the exception of some peaks important for discussion, only peaks with an intensity greater than 2% have been considered.

donor–acceptor bond, because of the redistribution of the π-electron density the internuclear bond acquires the nature of a π bond, provided that the arrangement of the rings is close to coplanar [4]. Thus, a disturbance of coplanarity for the bi compounds must lead to a marked fall in the value of W_M, since a weakening of the internuclear bond leads to the appearance of an additional pathway of decomposition on electron impact.

On the basis of the fairly high values of W_M for the series of compounds (I–IV) (Table 1) which are, respectively, 31.3 (I), 34.0 (II), 18.1 (III), and 23.2 (IV), it can be said that in the excited state the arrangement of the aromatic rings of the bicyclic system is very close to coplanar (value of W_M for quinoline 34.5) [5]. Partial hydrogenation at the position of linkage of one of the rings of the system (compounds VIII–X) naturally leads to a marked fall in the stability of the molecule [W_M for compounds (VIII–X) 2.2, 10.6, and 13.6, respectively.]

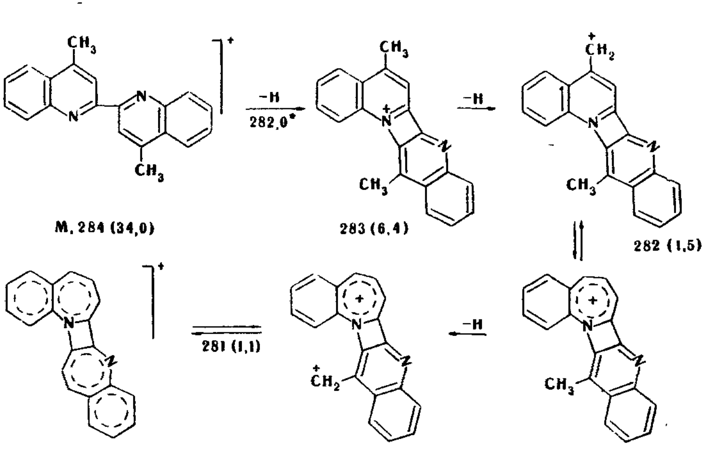
The mutual arrangement of the nuclei in the biquinolinyl systems must have a considerable influence on stability to electron impact. It is known that for the bipyridinyls the degree of double-bondedness decreases in the sequence 4,4′ > 2,2′ > 3,3′. The decrease in W_M for compounds (V) and (VI) (13.6) agrees well with an analogous relationship for the biquinolinyls. These values are three times smaller than the corresponding values of W_M in the compounds (I) and (III) isomeric with them (Table 1).

The variations in the values of W_M in the series of compounds (II–V) and (VII–X) are due to the position of the methyl group in the rings of the biquinolinyl nucleus. These changes agree well with the nature of the fluctuations of the value of W_M for methyl-substituted quinolines [5, 6].

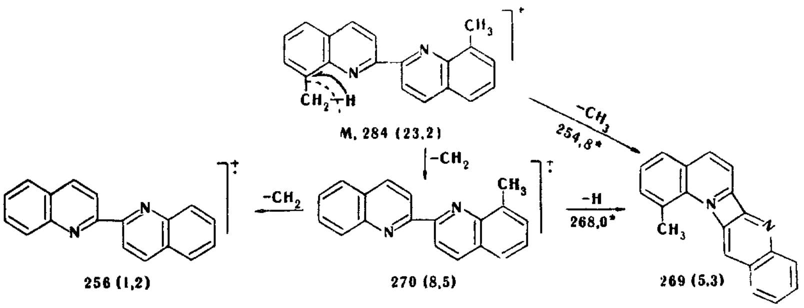
It can be seen from an analysis of the mass spectra (I–VI) obtained that, as was to be expected, the decomposition of the bicyclic structures under the action of electron impact takes place in a similar manner to the decomposition of the phenylpyridines [7], the bipyridinyls [1], and the imidazolylquinolines [8]: the loss by the molecular ion of one hydrogen atom leads to the formation of the most energetically favorable structure, which arises in the formation of a four-membered ring through a nitrogen atom. Then the ion that has arisen $(M-H)^+$ loses hydrogen atoms successively, the maximum number of H atoms eliminated being determined by the possibility of the retention by the ions of an aromatic cyclic structure. The condensed systems formed promote the appearance in the spectra of a series of doubly charged ions. The cleavage of the internuclear C–C bond becomes possible only when conjugation between the rings of the system is absent (compounds VIII–X). The presence of methyl substituents is responsible for additional decomposition pathways.

The first stage of the process (dehydrogenation) takes place for compounds (I–V) monotypically. The π-electronic nature of the bond between the aromatic nuclei and the successive elimination of hydrogen from the molecular ion permit the conclusion that in the excited state the molecular ion in biquinolinyl systems exists as s-trans and s-cis forms, according to the position of the internuclear bond. Thus, for compounds (I–IV), which have the ring linkage in the 2,2′ position, the s-trans configuration of the molecular ion is preferred, thus ensuring predominant cyclization with the formation of quadruply bound nitrogen, which leads to the appearance of a multiring system with condensed nuclei. This hypothesis is confirmed indirectly by the high value of the ratio of the intensities of the peaks of the fragmentary ions $(M-1)^+/(M-2)^+$ for compound (I) (Table 2). A similar process of ring closure may take place exclusively from the s-cis form of the molecular ion in the case of compounds (V) and (VII).

The dehydrogenation process thus leads to a condensed system including two aza ions (compounds II and V) or two benzotropylium ions (compounds III and IV), depending on the position of the methyl group in the quinoline nucleus [9]. In the compounds (I-VI) studied, the dehydrogenation process corresponds to 9-24% of the total ion current.†



The presence of methyl substituents in both quinoline nuclei (compounds II-V) leads to the appearance in the mass spectra of these compounds of the peak of a fragmentary ion $(M-15)^+$, the intensity of which amounts to 5.0-9.5% of the total ion current. The elimination of a methyl substituent is accompanied by a rearrangement process connected with the migration of a hydrogen atom from the β position of the quinoline ring to the position of the splitting off of a methyl group or by a redistribution of the hydrogen atoms in the aromatic ring. The closeness of the methyl group to the nitrogen atom of the heterocycle (compound IV) explains the anomalous splitting off of a methylene group from the molecular ion [5], which leads to the formation of a pseudomolecular ion with mass 270 having the structure of 2-(8-methyl-2-quinolinyl)quinoline.



In an investigation of the mass spectra of the bipyridinyls it was shown [1] that heterocyclic systems linked through a C—C bond present in the α position to the heteroatom form doubly charged molecular ions with a high probability. A similar phenomenon has been observed in the mass spectra of the benzoquinolines, where the formation and intensities of the M^{2+} ions were connected with the size, aromaticity, and stability of the systems considered [10]. The presence of a number of doubly charged ions with m/e values of 142, 141.5, 141, 140.5, 134.5, 134, 128, and 127.5 in compounds (I-VI) is an analytical characteristic of bisheterocyclic compounds. A calculation of intensities for M^{2+} that has been performed shows that, as a rule, cleavage at the bond connecting the two heterocycles does not take place in compounds (I-VI), since the ions with m/e values of 142 and 128 exist to the extent of 80-85% in the M^{2+} form. The total proportion of the doubly charged ions formed amounts to 7.5-10% of the total ion current.

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†Here and below, a figure under a formula corresponds to the value of the mass number for the given ion, a figure in parentheses to the intensity of the peak in % of the total ion current, and a figure with an asterisk to the apparent mass of a metastable transition.

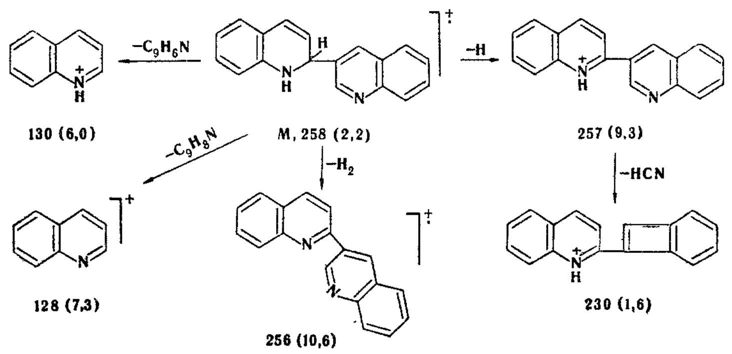
TABLE 2. Scheme of the Identification of Biquinolinyl Structures (intensities in % of the total current)

Compound	Characteristic ions and their ratios				
	M	$(M-1)^+/(M-2)^+$	$(M-3)^+$	$(M-14)^+/(M-15)^+$	$(M-28)^+$
I	31,3	8,3	1,3	—	—
II	34,0	4,2	1,1	2,8	—
III	18,1	4,3	1,2	4,1	—
IV	23,2	4,4	—	0,6	1,2
V	13,6	4,6	—	4,4	—
VI	13,6	3,9	—	—	—
VII	4,5	2,5	1,1	—	—
VIII	2,2	0,9	—	—	1,6
IX	10,6	0,9	—	—	5,0
X	13,6	1,4	—	—	4,6

In its first stages, the decomposition of the partially hydrogenated biquinolinyl (VII) is similar to the decomposition of 1,2,3,4-tetrahydroquinoline [6]. Initially the aromatization of the ring takes place with the loss of three hydrogen atoms, the greatest contribution to the dehydrogenation process being made by the loss of one hydrogen atom from the α position of the hydrogenated pyridine ring of the biquinolinyl, with the subsequent process of the elimination of a methyl group (metastable ion with an apparent mass of 241.8). The combination of the processes described leads to the formation of a pseudomolecular ion with the structure of 2,6'-biquinolinyl, the further decomposition of which takes place in a similar manner to the decomposition of the structures of compounds (I and VI).

The decomposition of the dihydrogenated bicyclic quinoline compounds (VIII-X) is similar to the dissociation of 1,2-dihydroquinoline derivatives under the action of electron impact [11, 12]. The detachment of the α-hydrogen [or of a methyl group in the case of compound (X)] from the molecular ion that is observed takes place in the general case with the formation of a stable cation with the quinolinylquinolinium structure. The presence of a hydrogen atom on the nitrogen of the heterocyclic ring makes it impossible for the nuclei of the bicyclic system to link up with the formation of a four-membered cyclobutadiene ring including the nitrogen atom in the ring. Consequently, in the case of compounds (VIII and IX) an energetically favorable ejection of the neutral particle HCN from the fragmentary ion $(M-H)^+$ is possible, which leads to the appearance in the spectrum of an ion with a mass of 228, the intensity of the peak of which differs greatly according to the initial structure of the isomer [1.7% of the total current for (VIII) and 5.0% for (IX)].

The pseudomolecular ions $(M-2)^+$ with masses of 256 (compounds VIII and IX) and 284 (compound X) formed as a result of the dehydrogenation process have the structures of the corresponding biquinolinyls and undergo further dissociation similar in nature to that of the biquinolinyl structures discussed. The unusually high intensities of the fragmentary ions with masses of 128 and 142 in the case of compounds (VIII-X) (7.3, 4.2, and 5.6% of the total ion current, respectively) are the result of the cleavage of the σ bond connecting the two rings and, to a far smaller extent, of the formation of doubly charged ions.



The correlations found between the molecular structures of the various biquinolinyl structures and the main directions of their dissociative ionization make it possible to use a small number of ions (Table 2) for the identification of biquinolinyl systems.

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