# HETEROCYCLIC ANALOGS OF PLEIADIENE.

# 49.* MASS SPECTRA OF SOME N-SUBSTITUTED PERIMIDINES AND 2,3-DIHYDROPERIMIDINES

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The mass spectra of nine compounds of the perimidine series, as well as 1,3-dimethyl derivatives of 2,3-dihydroperimidine, perimidone, and thioperimidone, were obtained and are discussed. The perimidines are distinguished by their high resistance to electron impact. The initial fragmentation of their molecular ions is characterized by detachment of the substituent attached to nitrogen, as well as by detachment of a hydrogen atom from the N-methyl group. It is proposed that the intensities of the doubly charged $M^{2+}$ ions be used to estimate the π-donor properties of heterocyclic compounds.

Very little study has been devoted to the mass spectra of perimidines, despite the fact that this class of compounds is of considerable interest in view of the peculiarity of their physicochemical properties [2]. Uncoordinated data on the mass spectra of individual 2-substituted perimidines [3, 4] and 2,3-dihydroperimidines [4, 5] are available. Only a table of mass numbers of the ions has been presented for perimidine itself [3]. In the present research we have for the first time obtained the mass spectra of a number of simple N-substituted perimidines (I-IX) and 2,3-dihydroperimidine structures X-XII in order to ascertain the analytical criteria that link the structural peculiarities of this class of compounds with the character of the fragmentation of the molecular ions ($M^+$); these spectra are also discussed in this paper. The results are summarized in Tables 1 and 2.

Perimidines, like other electron-surplus heterocycles (indoles [6], carbazoles [6], and benzimidazoles [7]), are distinguished by their high resistance to electron impact. Thus, the $W_M$ values for the compounds that we studied range from 30.6 for V to 43.1 for XI. Compounds VII-X, for which the $W_M$ values are lower (from 20.5 to 22.5), constitute exceptions. The decrease for the first two compounds is due to the significant contribution of the $F_1$ ion (in this case its appearance is due to the facile elimination of $CH_3CO$ and $CH_3O$ particles from $M^+$) to the total ion current. The relatively low stability of IX and X is explained by the presence in their structures of an incompletely aromatized fragment (the piperidine ring for IX and the dihydroperimidine ring for X), which is readily aromatized under the influence of ionizing electrons.

Another important analytical characteristic of the investigated compounds is the formation of an intense $M^{2+}$ peak; this, in our opinion, is associated with the high degree of π-donor character of perimidines [8] and 2,3-dihydroperimidines [9]. In fact, the $J_{M^{2+}}$ values for N-methyl derivatives of pyrrole, indole, carbazole, and perimidine, which are 6.0, 6.6, 8.3, and 10.1%, respectively [10, 11], change strictly symbatically with an increase in the π-donor character [12]. The $J_{M^{2+}}$ values for π-amphoteric and π-deficient heterocycles are substantially lower: for example, they are 4 and 1% for 1-methylbenzimidazole and pyridine, respectively [10, 11]. This observation constitutes evidence that the intensities of the doubly charged ions can be used as good criteria for the π-donor character of heterocycles. The $M^{2+}$ peaks are absent only in the case of VI-VIII and X, which have either reduced π-donor character because of the presence of an electron-acceptor group ($CH_2OCH_3$ in VII) or form a relatively unstable molecular ion (VI, VIII, and X).

*See [1] for communication 48.

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TABLE 1. Mass Spectra of I-XII

| Compound | Mass spectra, m/e (the peaks of ions with intensities > 3% of the maximum peak are presented) |
|---|---|
| I | 63 (5,6), 77 (4,5), 91 (10,1) — $M^{+2}$, 113 (4,5) — $F_6$, 127 (5,6) — $F_4$, 140 (48,3) — $F_5$, 141 (3,4), 154 (3,4) — $F_3$, 167 (46,1) — F, 168 (6,7), 181 (4,5) — $F_1$, 182 (100,0) — $M^+$, 183 (12,4); $W_M$=37,3 |
| II | 57 (3,8), 63 (6,3), 77 (5,1), 98 (8,6) — $M^{+2}$, 113 (5,1) — $F_6$, 126 (6,3), 127 (17,7) — $F_4$, 140 (17,7) — $F_5$, 153 (3,8), 154 (12,7) — $F_3$, 155 (3,8), 167 (3,2), 181 (58,2) — F, 182 (11,4), 195 (3,8) — $F_1$, 196 (100,0) — $M^+$, 197 (13,4); $W_M$=33,0 |
| III | 77 (7,7), 113 (3,8) — $F_6$, 127 (9,6) — $F_4$, 128 (7,7), 128,5 (11,5) — $F_1^{+2}$, 129 (6,8) — $M^{+2}$, 140 (9,6) — $F_5$, 154 (5,8) — $F_3$, 242 (9,6), 243 (28,8) — Ф, 244 (5,8), 256(5,5) — $(M-2H)^+$, 257 (30,8) — $F_1$, 258 (100,0) — $M^+$, 259 (19,2); $W_M$=37,1 |
| IV | 63 (3,3), 77 (4,3), 108,5 (3,2), 113 (3,2) — $F_6$, 122 (5,0) — $M^{+2}$, 140 (13,8) — $F_5$, 166 (3,2), 167 (11,7) — F, 190 (31,9), 216 (3,7) — $F_3$, 217 (31,2), 218 (3,7), 242 (7,4) — $(M-2H)^+$, 243 (16,0) — $F_1$, 244 (100,0) — $M^+$, 245 (18,4); $W_M$=34,1 |
| V | 63 (4,3), 77 (3,3), 113 (4,3) — $F_6$, 125 (7,6) — $M^{+2}$, 126 (3,3), 127 (5,4) — $F_4$, 140 (30,4) — $F_5$, 141 (3,3), 153 (3,2), 166 (4,3), 167 (3,3), 181 (7,6), 197 (13,0), 230 (3,3), 235 (70,7) — F, 236 (8,7), 250 (100,0) — $M^+$, 251 (13,0); $W_M$=30,6 |
| VI | 57 (3,4), 63 (5,7), 91 (100,0), 92 (6,4), 113 (6,7) — $F_6$, 140 (16,2) — $F_5$, 167 (18,9) — F, 168 (3,4), 258 (68,3) — $M^+$, 259 (9,8); $W_M$=34,5 |
| VII | 55 (5,7), 63 (12,6), 77 (9,7), 113 (19,3) — $F_6$, 114 (5,3), 126 (6,1), 127 (19,3) — $F_4$, 140 (41,9) — $F_5$, 141 (5,8), 154 (8,9) — $F_3$, 167 (38,7) — F, 168 (25,8), 181 (27,1) — $F_1$, 182 (32,2), 212 (100,0) — $M^+$, 213 (19,3); $W_M$=22,4 |
| VIII | 43 (14,2), 57 (10,0), 63 (5,6), 77 (10,8), 113 (4,2) — $F_6$, 126 (8,2), 127 (42,8) — $F_4$, 128 (5,0), 140 (17,8) — $F_5$, 153 (6,6), 154 (41,4) — $F_3$, 167 (4,6) — F, 168 (5,7), 181 (100,0) — $F_1$, 182 (16,0), 224 (63,4) — $M^+$, 225 (10,0); $W_M$=20,5 |
| IX | 51 (4,6), 63 (7,8), 74 (3,9), 75 (7,5), 76 (9,8), 77 (6,5), 89 (5,9), 89,5 (5,6), 90 (4,9), 90,5 (4,9), 103 (17,0), 103,5 (6,9), 104 (14,7) — $M^{+2}$, 125 (3,3), 126 (8,2), 127 (6,9), 141 (4,2), 152 (6,2), 153 (13,1) — $F_8$, 154 (11,4) — $F_9$, 155 (4,2), 166 (3,3), 167 (3,9), 177 (3,3), 178 (5,9), 179 (20.6), 180 (32,7) — $F_7$, 181 (13,0), 192 (18,0), 193 (10,8), 205 (11,8), 206 (4,9), 207 (32,3), 208 (100,0) — $M^+$, 209 (15,3); $W_M$=21,4 |
| X | 63 (5,2), 77 (5,3), 89,5 (10,5), 90 (7,0), 91 (8,8), 113 (3,5) — $F_6$, 126 (3,4), 127 (10,7) — $F_4$, 128 (3,5), 140 (10,3) — $F_5$, 141 (3,4), 154 (5,3) — $F_3$, 167 (19,3) — F, 168 (10,9), 181 (3,5) — $F_1$, 182 (64,9), 183 (8,8), 197 (100,0) — A, 198 (89,5) — $M^+$, 199 (10,5); $W_M$=22,5 |
| XI | 57 (4,1), 63 (4,4), 77 (9,2), 106 (5,2) — $M^{+2}$, 127 (9,9) — $F_4$, 128 (3,0), 140 (7,0) — $F_5$, 141 (3,0), 154 (5,6) — $F_3$, 167 (5,0), 168 (6,6), 169 (10,2), 182 (8,5), 183 (4,3), 197 (8,7) — F, 211 (7,3) — $F_1$, 212 (100,0) — $M^+$, 213 (14,1); $W_M$=43,1 |
| XII | 45 (9,6), 63 (3,6), 77 (4,8), 83,5 (3,0), 114 (7,1) — $M^{+2}$, 126 (7,2), 127 (14,5) — $F_4$, 140 (9,6) — $F_5$, 153 (4,7), 154 (15,7) — $F_3$, 166 (3,6), 167 (12,0), 168 (13,3), 182 (26,5), 183 (3,7), 195 (7,2), 213 (12,0) — F, 227 (3,9) — $F_1$, 228 (100,0) — $M^+$, 229 (13,3), 230 (4,8); $W_M$=31,9 |

It is apparent from Table 1 that the $M^+$ peak is the most intense peak in the mass spectra for all of the compounds except VI, VIII, and X. The common pathway of its initial fragmentation for all I-VIII consists in detachment of the substituent attached to nitrogen to give ion F. Judging from the intensity of the F ion, this process dominates for I, II, and V-VII. The N-benzyl group is detached with particular ease, as evidenced by the fact that the most intense peak in the mass spectrum of VI is the peak with m/e 91, which undoubtedly belongs to a benzyl cation that has undergone rearrangement to a tropylium ion. In this connection, it is clear why a second rather general pathway for fragmentation of the $M^+$ ion, which consists in the formation of rearranged ion $F_1$, is not characteristic for VI. The $F_1$ ion arises as a result of detachment of a hydrogen atom from the N-methyl group of I-III or as a result of detachment of methoxy and acetyl groups in the case of VII and VIII. This second pathway for the fragmentation of the $M^+$ ion predominates in the case of 1-methyl-2-phenyl- and 1-acetonylperimidines (compare also the ease of detachment of the benzoyl group from the $M^+$ ion of 1-phenacylperimidine [13]). However, in addition to VI, it is also not realized in the case of 1-methyl-2-trifluoromethylperimidine (V).

The formation of extremely intense $(M-1)^+$ and $(M-2)^+$ ions is characteristic for perimidines IV and III with phenyl substituents in the 1 or 2 positions. It is interesting (for example, see [14]) that the peaks of similar ions are also present in other systems with aryl and hetaryl rings connected by a single bond (biphenyl, dipyridyls, diquinolyls, etc.). Their origin is usually explained by intramolecular crosslinking of the rings with the formation of structures of the diphenylene, fluorene, and other types. In the case of

TABLE 2. High-Resolution Mass Spectra (M/ΔM = 15000 with Polyphosphoric Acid as the Standard)

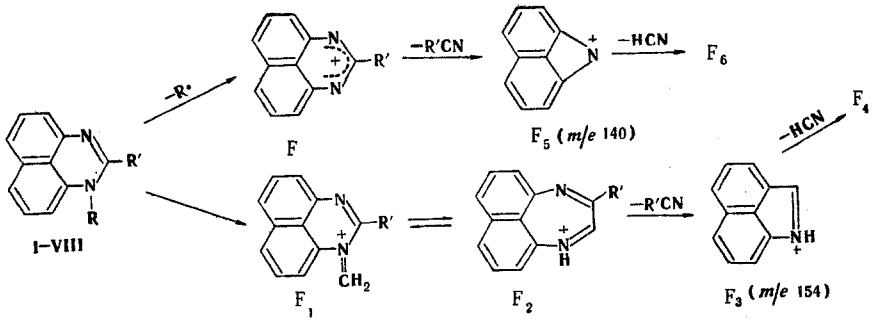
| Compound | Precise mass | | Empirical formula of the ion | Arbitrary designation of the ion |
|---|---|---|---|---|
| | determined | calculated | | |
| I | 182,0888 | 182,0843 | $C_{12}H_{10}N_2$ | M+ |
| | 167,0603 | 167,0609 | $C_{11}H_7N_2$ | F |
| | 154,0673 | 154,0656 | $C_{11}H_8N$ | $F_3$ |
| | 140,0530 | 140,0500 | $C_{10}H_6N$ | $F_5$ |
| | 127,0514 | 127,0547 | $C_{10}H_7$ | $F_4$ |
| | 113,0366 | 113,0391 | $C_9H_5$ | $F_6$ |
| V | 250,0681 | 250,0717 | $C_{13}H_9N_2F_3$ | M+ |
| | 235,0466 | 235,0483 | $C_{12}H_6N_2F_3$ | F |
| | 230,0615 | 230,0655 | $C_{13}H_8N_2F_2$ | |
| | 197,0501 | 197,0516 | $C_{12}H_6N_2F$ | |
| | 181,0748 | 181,0765 | $C_{12}H_9N_2$ | |
| | 166,0566 | 166,0530 | $C_{11}H_6N_2$ | |
| | 140,0525 | 140,0500 | $C_{10}H_6N$ | $F_5$ |
| IX | 208,1034 | 208,1000 | $C_{14}H_{12}N_2$ | M+ |
| | 207,0946 | 207,0922 | $C_{14}H_{11}N_2$ | |
| | 192,0687 | 192,0687 | $C_{13}H_8N_2$ | |
| | 180,0711 | 180,0687 | $C_{12}H_8N_2$ | $F_7$ |
| | 179,0611 | 179,0609 | $C_{12}H_7N_2$ | |
| | 154,0528 | 154,0530 | $C_{10}H_6N_2$ | $F_9$ |
| | 153,0558 | 153,0578 | $C_{11}H_7N$ | $F_8$ |
| XII | 213,0447 | 213,0486 | $C_{12}H_9N_2S$ | F |
| | 195,0929 | 195,0922 | $C_{13}H_{11}N_2$ | |
| | 182,0865 | 182,0843 | $C_{12}H_{10}N_2$ | |
| | 168,0692 | 168,0687 | $C_{11}H_8N_2$ | |
| | 167,0622 | 167,0656 | $C_{11}H_7N_2$ | |
| | 154,0638 | 154,0656 | $C_{11}H_8N$ | $F_3$ |
| | 140,0505 | 140,0500 | $C_{10}H_6N$ | $F_5$ |
| | 127,0552 | 127,0547 | $C_{10}H_7$ | $F_4$ |

III this makes it possible, in particular, to explain the high intensities of the ions with m/e 128 and 128.5. Another pathway for the fragmentation of the $M^+$ ion for 1-phenylperimidine is the successive elimination of two HCN particles (m/e 217 and 190).

Specific fragmentation pathways also are observed for some other compounds. Thus, in the case of V they consist in the elimination of HF (m/e 230) and $CF_3$ (m/e 181) particles from the $M^+$ ion. The formation of these ions is confirmed by data from high-resolution mass spectrometry (Table 2). A peak of a pseudomolecular ion with m/e 182 is present in the mass spectrum of VII. Its formation is evidently associated with migration of hydrogen from the methyl group to the site of cleavage of the C—O bond.

The overall picture of the fragmentation of I-VIII can be represented by the following scheme (the sequence of the fragmentation processes was determined by a study of the spectra of the metastable ions obtained by the DADI method [15]).

The F ion formed after detachment of the substituent attached to nitrogen ejects an R'CN particle and is converted to an ion with m/e 140. A peak with the same mass number is also present in the mass spectrum of perimidine itself [3] and in the mass spectra of virtually all of its 1- and 2-substituted derivatives [3] (Table 1). As a rule, it is distinguished by its high intensity, and its appearance can be regarded as a characteristic feature of the perimidine structure (under the condition that substituents are absent in

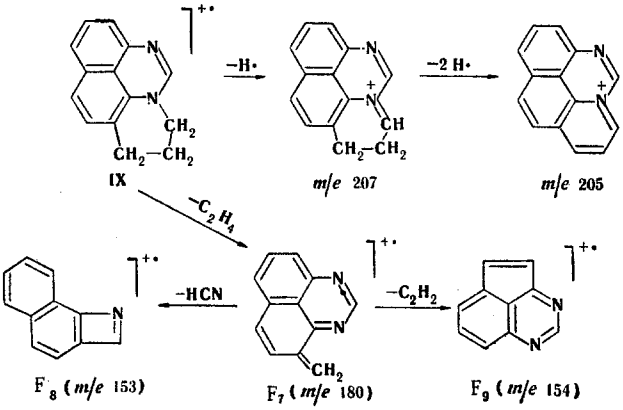



I R=$CH_3$; R'=H; II R=R'=$CH_3$; III R=$CH_3$; R'=$C_6H_5$; IV R=$C_6H_5$; R'=H; V R=$CH_3$; R'=$CF_3$; VI R=$C_6H_5CH_2$; R'=H; VII R=$CH_3OCH_2$; R'=H; VIII R=$CH_3COCH_2$; R'=H

the naphthalene ring). In analogy with the mass spectra of benzimidazoles [7], we assigned the $F_5$ structure to the ion with m/e 140. It must be emphasized that heterocycles with a four-membered ring condensed in the peri positions of naphthalene were recently synthesized [16]. The subsequent fragmentation of the $F_5$ ion involves ejection of an HCN particle and the formation of the $F_6$ ion (m/e 113).

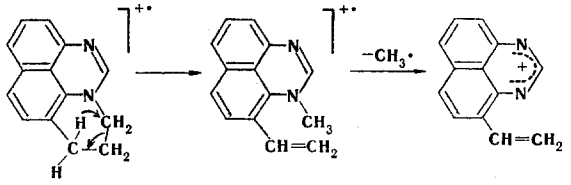
The subsequent fragmentation of the $F_1$ ion is accompanied by ejection of an R'CN fragment and the formation of an ion with m/e 154, which probably has the structure of the benz-[c,d]indole cation ($F_3$). Derivatives of this heterocycle are also known [17]. In analogy with the hypothetical conversion of the $(M - 1)^+$ ion for 1-methylbenzimidazole [7, 18] to a quinoxalinium cation, one may assume rearrangement of the $F_1$ ion to the 1,4-diazapleiadienium cation ($F_2$). The rather high intensity of the peak of the corresponding ion could be evidence in favor of the existence of the latter. In fact, the intensity of this peak is 30.8% for III. However, it does not exceed 4.2% (on the average) for I and II. In addition, the fact that elimination of an HCN particle, which should have led to 2-substituted $F_3$ cations, does not occur in the case of ring expansion to $F_2$, does not occur is surprising. This problem regarding the formation of an ion with the $F_2$ structure obviously requires a special study.

The mass spectrum of IX, which can be regarded as a perimidine that is substituted simultaneously at the nitrogen atom and the $C_9$ atom of the naphthalene ring, is distinguished by its considerable complexity. Judging from the set of $(M - H)^+$, $(M - C_2H_4)^+$, $(M - CH_3)^+$, and $(M - HCN)^+$ peaks, the fragmentation of its $M^+$ ion proceeds via many pathways. The first fragmentation pathway, viz., the successive detachment of three hydrogen atoms from $M^+$, leads to complete aromatization of the piperidine ring (peaks with m/e 207, 206, and 205). This is monitored by the corresponding doubly charged ions.




The formation of a fragment with mass 180 can be conceived of as being the result of splitting out of a $CH_2{=}CH_2$ molecule from the molecular ion of IX in the manner of retrodiene fragmentation. Taking into account the above-noted ease of cleavage of the nitrogen–substituent bond in perimidines and the stability of 4H-isoperimidine structures [19], quinomethide structure $F_7$ should be assigned to the corresponding ion in this case. Its fragmentation with the elimination of HCN and $C_2H_2$ particles gives ionpeaks with m/e 153 and 154, the structures of which can be represented as $F_8$ and $F_9$. These data are confirmed by recording of the high-resolution spectra (Table 2).

The elimination of a methyl group from $M^+$ precedes its rearrangement to an ion of 1-methyl-9-vinylperimidine via the scheme

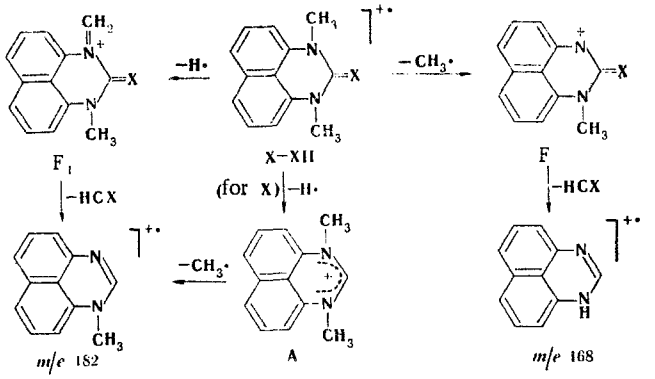



A similar process takes place in the dissociative ionization of pyrrolidine, piperidine, and piperidinium.

The fragmentation of the molecular ion of X in many respects resembles the pathways of fragmentation of I. This is explained by the fact that two successive processes, viz.,

aromatization to the 1,3-dimethylperimidinium cation (ion A) with the detachment of a hydrogen atom [the $(M - 1)^+$ peak is the most intense peak in the spectrum of X] and demethylation, which leads to the pseudomolecular ion of I, occur initially for X with high selectivity.

The initial dissociation of the $M^+$ ions of 1,3-dimethyl derivatives of perimidone (XI) and thioperimidone (XII) is in principle analogous to that for 1-substituted perimidines; it also includes two principal processes that entail detachment of a hydrogen atom from the $CH_3$ group and demethylation. The resulting F and $F_1$ ions split out an HCX particle and give perimidine (m/e 168) and 1-methylperimidine (m/e 182) ions. The formation of the latter, in particular, is proved satisfactorily by the presence in the mass spectra of XI and XII of peaks at m/e 154, 140, and 127, which are characteristic for the mass spectra of typical perimidine structures. A substantial difference between XII and its oxygen analog is the splitting out of an HS particle from $M^+$ (Table 2). These data make it possible to give the following overall picture of the initial fragmentation for the $M^+$ ions of X-XII samples:




X X=$CH_2$; XI X=O; XII X=S

In contrast to X, the mass spectra of XI and XII contain the peaks of a doubly charged $M^{2+}$ ion, the intensities of which are, respectively, 5.2 and 7.1%. These values constitute evidence for a smaller degree of π-donor character for perimidone XI and thioperimidone XII as compared with 1-methylperimidine (I), which is in agreement with the data obtained by other methods [8, 9]. However, there are deviations in the estimates of the π-donor character of XI and XII themselves. Whereas data on charge transfer complexes [9] provide evidence for somewhat greater π-donor character of perimidone, the values presented above lead to the opposite conclusion. It is interesting to note that similar deviations between various methods in the estimation of the π-donor character are also characteristic for other oxygen- and sulfur-containing heterocycles (for example, furan and thiophene, and phenoxazine and phenothiazine) [12].

Thus, our study showed that perimidines in which α cleavage relative to the hetaryl ring predominates resemble π-surplus heterocycles with a pyrrole nitrogen atom (indole, carbazole, and benzimidazole) to a greater extent with respect to their behavior toward electron impact [6, 7] than azines, which are characterized by β cleavage. Moreover, perimidines also have specific features. Thus, whereas the $M^+$ ions of N-alkylcarbazoles tend to split out an N-alkyl substituent but do not split out a hydrogen atom, while N-alkylbenzimidazoles, on the other hand, eliminate hydrogen but do not eliminate a substituent, both of these processes are characteristic for N-alkylperimidines.

## EXPERIMENTAL

The mass spectra of all of the compounds were obtained with a JEOL JMS-01-JC-2 spectrometer with direct introduction of the samples into the ion source. The ionizing voltage was 75 eV, the cathode emission current was 300 μA, the accelerating voltage was 8 kV, and the ionization chamber temperature ranged from 120-130°C. The high-resolution mass spectra were recorded under the same recording conditions with $M/\Delta M = 15000$ on a type Q photoplate and were interpreted with a JMD-2M and JEC-6 microphotometer–computer system.

The investigated compounds were synthesized by the following methods: I, II, VI, and VII by the method in [22]; III by the method in [23]; IV by the method in [24]; V by the

method in [25]; VIII by the method in [13]; X and XI by the method in [26]; and XII by the method in [27]. Compounds VI-IX were synthesized and made available to us by V. V. Dal'nikovskaya, for which we express our gratitude.

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