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Dedicated to Full Member of the Russian Academy of Sciences N.S. Zefirov on His 70th Anniversary

Mass Spectra of New Functionally Substituted Heterocycles: V.* First Example of McLafferty Rearrangement in the Series of 5-(1-Ethoxyethoxy)-2,3-dihydropyridines and 3-(1-Ethoxyethoxy)pyridines Derived from α-Lithiated 1-(1-Ethoxyethoxy)allene and Methoxymethyl Isothiocyanate

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Abstract—Previously unknown 5-(1-ethoxyethoxy)-2-methoxy-6-methylsulfanyl-2,3-dihydropyridine, 3-(1-ethoxyethoxy)-2-methylsulfanylpyridine, and 2-methylsulfanylpyridin-3-ol were synthesized from α-lithiated 1-(1-ethoxyethoxy)allene, methoxymethyl isothiocyanate, and methyl iodide, and their fragmentation under electron impact was studied. At ionizing electron energies of 12 and 60 eV in the temperature range from 50 to 250°C, the molecular ions derived from 5-(1-ethoxyethoxy)-2-methoxy-6-methylsulfanyl-2,3-dihydropyridine and 3-(1-ethoxyethoxy)-2-methylsulfanylpyridine decompose along two main pathways: rupture of the C–O bond in the acetal moiety to give oxonium ions with *m*/*z* 73 (in the two cases) and 168 (pyridine derivative) and unexpectedly facile McLafferty rearrangement with elimination of ethoxyethene molecule to produce fragment ions with *m*/*z* 173 and 141, respectively. The latter pathway was not observed previously for dihydropyridines and acetals, and it predominates in the fragmentation of 3-(1-ethoxyethoxy)-2-methylsulfanylpyridine. Expulsion of methanol molecule from the molecular ion of 5-(1-ethoxyethoxy)-2-methoxy-6-methylsulfanyl-2,3-dihydropyridine occurred only above 170°C.

Recently reported reactions of metalated alkynes and dienes with heterocumulenes turned out to be a powerful tool in modern syntheses of the most important carbo- and heterocycles [2], including alkylsulfanyl-substituted pyridines [2–5] and still poorly studied 2,3-dihydropyridines [2–6].

In continuation of these studies, by reaction of α-lithiated 1-(1-ethoxyethoxy)allene with methoxymethyl isothiocyanate we have synthesized with high selectivity previously unknown and inaccessible by other methods 5-(1-ethoxyethoxy)-2-methoxy-6-methylsulfanyl-2,3-dihydropyridine (**I**). This compound showed an unexpectedly strong tendency to aromatization, leading to 3-(1-ethoxyethoxy)-2-methylsulfanylpyridine (**II**) [5] which was not described previously as well (Scheme 1). Interestingly, the aromatization process occurred even on storage of dihydropyridine **I** in a refrigerator, and elimination of ethanol was accelerated not only on heating (~120°C, 1 h) but also in the presence of acid catalysts (HCl, *p*-toluenesulfonic acid, ~30°C, 1 h). Mild hydrolysis (hydrochloric acid, 20°C, ~10 min) of pyridine **II** smoothly afforded 2-methylsulfanylpyridin-3-ol (**III**) [5] which was identified previously (by mass spectrometry) as one of the most important aromatizing components in smoked meat and smoking fluids [7].

The presence of labile substituents, 2-methoxy and 5(3)-(1-ethoxyethoxy) groups, in molecules **I** and **II** makes these compounds interesting models for both chemical and mass spectrometric studies. It should be emphasized that the known studies on the mass spectra of pyridines were focused mainly on the simplest alkylpyridines [8], while mass spectra of 2,3-dihydropyridines were not reported at all. Moreover, the title

* For communication IV, see [1].

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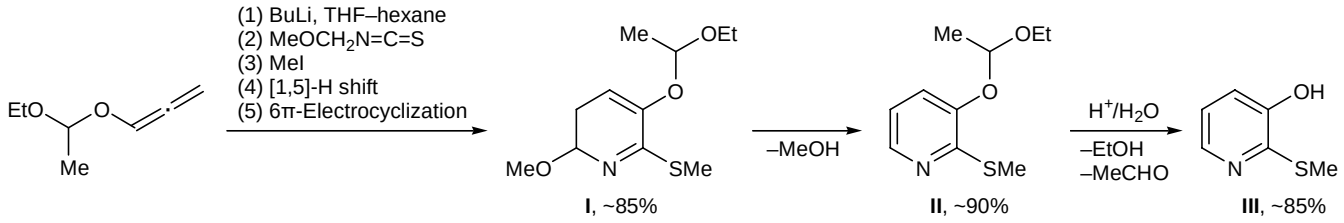
Scheme 1.



compounds may equally be regarded as functionally substituted pyridines (dihydropyridines) and mixed acetals derived from acetaldehyde, ethanol, and 2-methylsulfanylpyridin-3-ol (**III**) or 6-methoxy-2-methylsulfanyl-5,6-dihydropyridin-3-ol (**IV**). Therefore, their decomposition under electron impact may follow either pathways typical of compounds belonging to both these classes or a quite different pathway which could not be predicted *a priori*. In the preceding communications of this series [1, 9], we were the first to report the mass spectra of five-membered nitrogen- and sulfur-containing heterocycles, pyrroles, thiophenes, and dihydrothiophenes (which are also readily available from isothiocyanates and polyunsaturated carbanions [2]) having alkyl, cycloalkyl, alkylsulfanyl, imino, and mono- or disubstituted amino groups attached to ring carbon atoms.

With the goal of studying the mechanism of fragmentation under electron impact of molecular ions derived from functionally substituted aza heterocycles and elucidating the temperature conditions ensuring elimination of methanol, we examined the mass spectra of dihydropyridine **I** and pyridines **II** and **III** in the temperature range from 50 to 250°C with chromatographic and direct sample admission into the ion source.

The chromatograms of dihydropyridine **I** and pyridine **II** (the injector, oven, and ion source temperatures were 250°C) contained three and two peaks, respectively. The mass spectra were matched with those collected in the standard NBS 75K library. Among thermolysis products of dihydropyridine **I**, we identified methanol ($[M]^{+\cdot}$, *m*/*z* 32), ethoxyethene ($[M]^{+\cdot}$, *m*/*z* 72), and 2-methylsulfanylpyridin-3-ol (**III**) ($[M]^{+\cdot}$, *m*/*z* 141). The mass spectra of the thermolysis product from pyridine **II** matched the spectra of ethoxyethene and hydroxypyridine (**III**). The match probability was 89 to 92%. These data, in combination with the absence of peaks corresponding to compounds **I** and **II**, indicate that under the above conditions (250°C) they undergo complete thermal decomposition, though the fragmentation pattern of dihydropyridine **I** remains unclear (Scheme 2). A question arises so as to whether the fragmentation of **I** follows pathway *a* with initial elimination methanol, followed by expulsion of ethoxyethene, or vice versa (pathway *b*), or these processes are concurrent?

We examined the effect of the temperature of both inlet system and ion source on the mode and sequence of thermal transformations of dihydropyridine **I** in the range from 50 to 250°C. Table 1 contains the mass spectra of compound **I**–**III** recorded at an energy of ionizing electrons of 12 eV. The total mass spectra of **I** obtained at 60 eV (70–250°C) and relative ion peak intensities are collected in Table 2. Schemes 3 and 4 illustrate general fragmentation patterns of compounds **I** and **II**, respectively, under electron impact (60 eV). As follows from the data in Table 2, the mass spectra

Scheme 2.

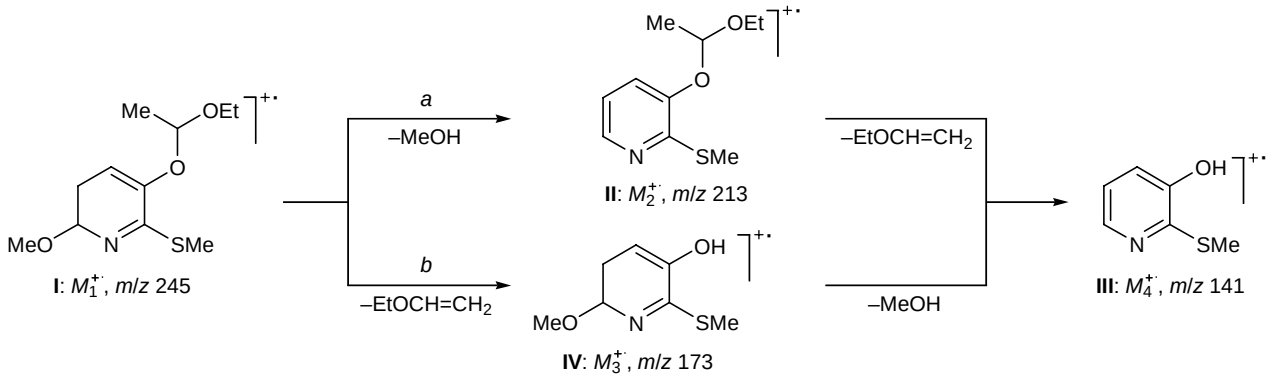


Table 1. Mass spectra of compounds **I**–**III** (electron impact, 12 eV)

Ion	*m*/*z* (I_{rel}, %)		
	I (60, 60)[a]	**II** (40, 50)[a]	**III** (110, 250)[a]
$[M]^{+\cdot}$	245 (20)	213 (40)	141 (100)
$[M - EtOCH{=}CH_2]^{+\cdot}$	173 (80)	141 (100)	–
$EtO^+{=}CHMe$	73 (100)	73 (20)	–

[a] In parentheses are given the temperatures of the direct inlet probe and ion source.

of compound **I**, obtained in the temperature range from 70 to 150°C, are almost identical. They contain the molecular ion peak $[M_1]^{+\cdot}$ with *m*/*z* 245 (in contrast to aliphatic acetals which usually give rise to unstable molecular ions; the corresponding peaks are often absent in the mass spectra [10]) and a limited number of fragment ion peaks. The main fragment ions are cations with *m*/*z* 73 (100%, **A**), 45 (84–87%, **B**), and radical cation with *m*/*z* 173 (69–71%, $[M_3]^{+\cdot}$). No ions expected from expulsion of methanol ($[M_2]^{+\cdot}$, *m*/*z* 213), methoxyl radical (*m*/*z* 214), or methyl radical (*m*/*z* 230) from the molecular ion of **I** were detected. By contrast, decomposition of the molecular ions derived from 5- and 3-methoxy analogs of **I** and **II**, 2,5-dimethoxy-6-methylsulfanyl-2,3-dihydropyridine ($[M]^{+\cdot}$, 72%) and 3-methoxy-2-methylsulfanylpyridine ($[M]^{+\cdot}$, 100%), begins with elimination of methyl radical; the resulting ions are the most abundant in the mass spectra (100 and 75%, respectively) [4]. In addition, the mass spectra of both analogs contain ion peaks due to elimination of MeS and MeO radicals and MeOH molecule from the molecular ion or $[M - Me]^+$.

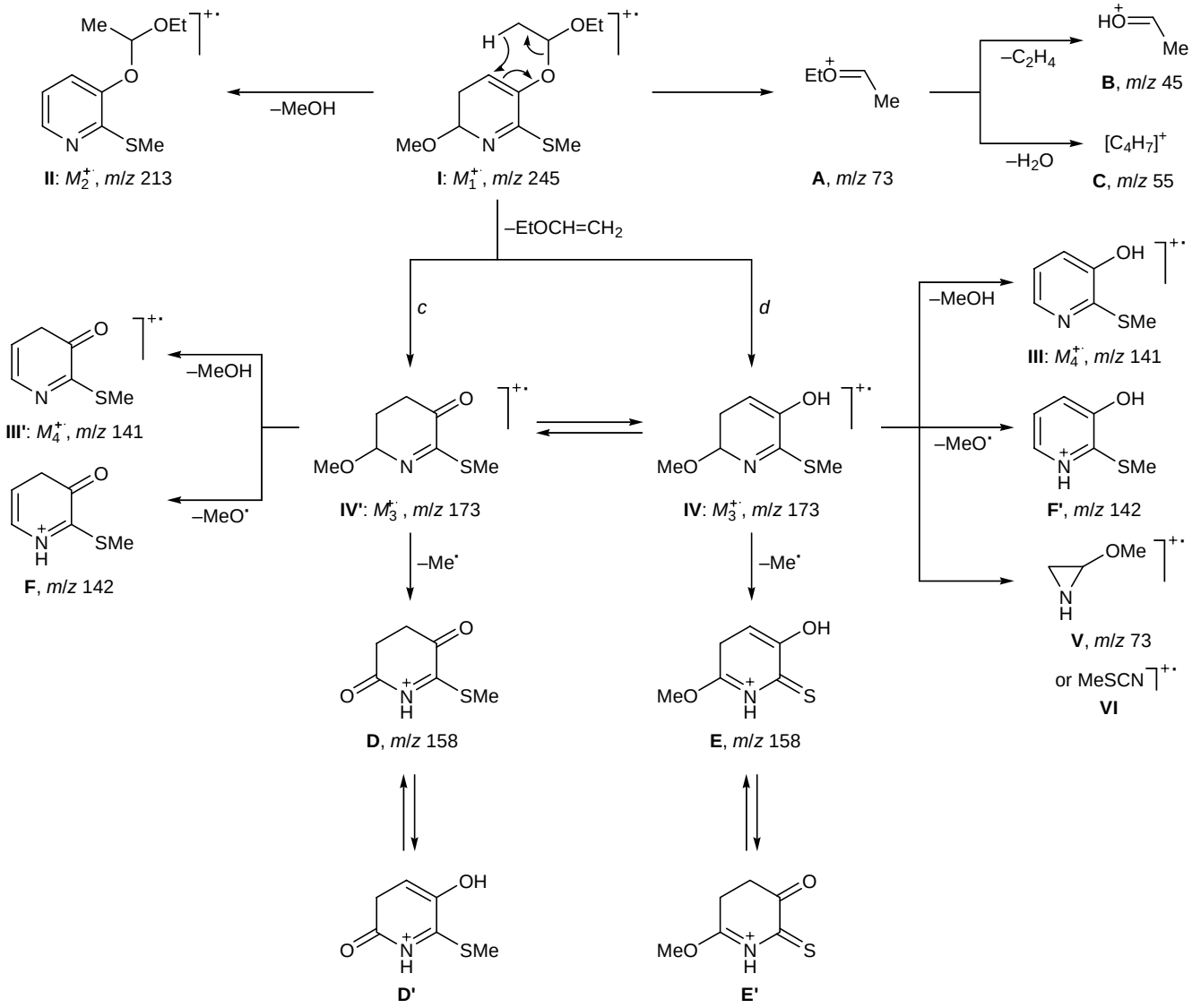
The mass spectra of dihydropyridine **I**, obtained at both 60 eV (Table 2) and 12 eV (Table 1), show two concurrent fragmentation pathways involving cleavage of the C–O bond in the 5-(1-ethoxyethoxy) group (Scheme 3). On the one hand, such decomposition is typical of acetals [10]: elimination of 6-methoxy-2-methylsulfanyl-5,6-dihydropyridin-3-yloxyl (*m*/*z* 172) from the molecular ion gives ethyl(ethylidene)oxonium ion **A** with *m*/*z* 73, which is the most abundant (100%) in both cases. On the other hand, the observed pattern may be interpreted as facile McLafferty-type rearrangement [11] which is characteristic of 2-substituted pyridines (mostly 2-alkyl derivatives) [8]. Such rearrangement was not reported previously for dihydropyridines and acetals; it includes hydrogen migration from the γ-position to the double-bonded carbon atom via six-membered cyclic transition state with decomposition of the β-C–O bond (Scheme 3, pathway *c*).

Table 2. Mass spectra of dihydropyridine **I** (electron impact, 60 eV, direct sample admission into the ion source)[a]

Comp. no.	Ion	*m*/*z*	Relative intensity I_{rel}, %					
			70–80°C[b]	130–150°C	170–180°C	190–200°C	200–210°C	250°C
I	$[M_1]^{+\cdot}$	245	11	16	11	1	0	0
II	$[M_2]^{+\cdot} = [M_1 - MeOH]^{+\cdot}$	213	0	0	6	13	19	0
IV, IV'	$[M_3]^{+\cdot} = [M_1 - EtOCH{=}CH_2]^{+\cdot}$	173	69	71	64	38	22	10
III, III'	$[M_4]^{+\cdot} = [M_2 - EtOCH{=}CH_2]^{+\cdot}$, or $[M_3 - MeOH]^{+\cdot}$, or $[\mathbf{G} - C_2H_3]^{+\cdot}$	141	0	38[c]	45[d]	82[d]	100[d]	40[d]
A	$EtO^+{=}CHMe$	73[e]	100	100	100	100	90	100
B	$[\mathbf{A} - C_2H_4]^+$	45	84	87	93	96	95	76
C	$[\mathbf{A} - H_2O]^+$	55	8	13	11	0	0	0
D, D', E, E'	$[M_3 - Me]^+$	158	26	29	29	11	6	2
F, F'	$[M_3 - MeO]^+$	142	4	4	11	8	1	0
G	$[M_2 - EtO]^+$	168	0	0	6	13	20	1
H	$[M_4 - SH]^+$	108	0	0	13	40	51	18
I	$[C_2H_3S]^+$	59	0	0	5	15	38	2

[a] Equal temperatures of the direct inlet probe and ion source.
[b] The spectra recorded at 50–70°C were essentially similar.
[c] $[M_3 - MeOH]^{+\cdot}$.
[d] Ion peak woth *m*/*z* 141 corresponds to $[M_2 - EtOCH{=}CH_2]^{+\cdot}$, $[M_3 - MeOH]^{+\cdot}$, and $[\mathbf{G} - C_2H_3]^{+\cdot}$.
[e] Coincides with *m*/*z* values for 2-methoxyaziridine (**V**) and methyl thiocyanate (**VI**) (Scheme 3).

Scheme 3.



The peaks of ion formed by the McLafferty rearrangement (**IV'**, *m*/*z* 173) have a strong intensity (80 and 69–71%, see Tables 1 and 2, respectively), but their intensity is lower than the intensity of ion peaks resulting from alternative simple cleavage of the C–O bond (100%). However, the ion with *m*/*z* 173 may be assigned to ionized 6-methoxy-2-methylsulfanyl-5,6-dihydropyridin-3-ol (**IV**) formed by nonspecific hydrogen transfer to the acetal oxygen atom [10] (Scheme 3, pathway *d*). In any case, it is difficult to distinguish (without special studies using labeled molecules) whether ions with similar *m*/*z* values (*m*/*z* 173) appear from dihydropyridine **I** along pathway *c* or *d*.

Thus, fast decomposition of dihydropyridine **I** in an ionization chamber on heating to 50–150°C in vacuo is not accompanied by elimination of methanol. This means that under these conditions dissociation of the C–O bond is faster than elimination of methanol to give pyridine **II**. Among other things, such an "anomalous" result (as compared to preparative experiments) may be regarded as an additional proof for the assumption [5] that the temperature is not determining or, at least, the only factor responsible for facile aromatization of dihydropyridine **I**.

It should also be noted that, among the three most characteristic decomposition pathways of known acetals [10], only one is operative in the case of mixed acetal **I**, namely the pathway leading to oxonium ion **A** (*m*/*z* 73). Oxonium ions with *m*/*z* 244 and 230, which might be expected to be formed along the two other

possible pathways (elimination of hydrogen atom and Me radical from the CHMe fragment) were not detected. Also, we did not identified peak from one more theoretically possible oxonium ion, ethylidene(6-methoxy-2-methylsulfanyl-5,6-dihydropyridin-3-yl)oxonium (*m*/*z* 200), which could result from elimination of ethoxyl radical from the molecular ion.

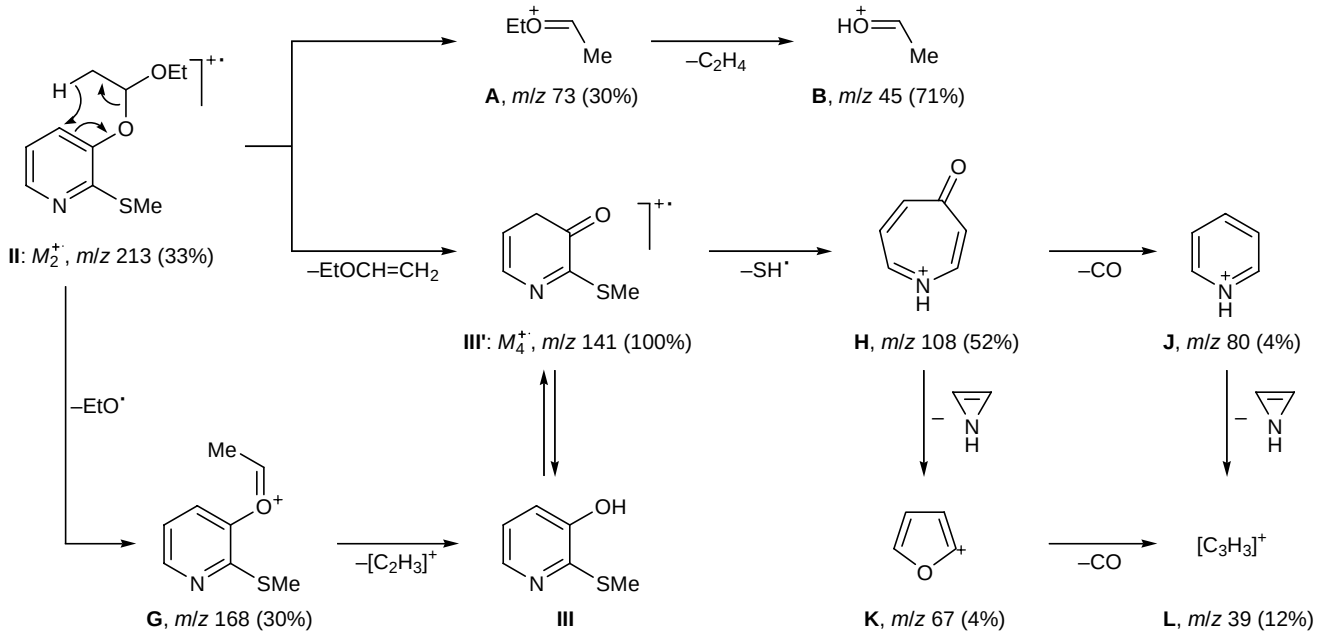
The presence of an ion peak with *m*/*z* 141 (38%, $[M_4]^{+\cdot} = [M_3 - \text{MeOH}]$) in the mass spectra recorded at 130–150°C (this peak was not detected at 70–80°C) indicates that elimination of methanol from dihydropyridine molecular ion is strongly facilitated provided that concurrent reaction center (acetal fragment) is lacking. In the mass spectrum of 2,5-dimethoxy-6-methylsulfanyl-2,3-dihydropyridine, the [*M* – MeOH] peak has a relative intensity of 10% [4].

The mass spectra of **I** recorded at 170–210°C (Table 2), in addition to ions shown in Scheme 3, contained ion peaks with *m*/*z* 213 (6–19%, **II**, $[M_2]^{+\cdot}$), 168 (**G**), 108 (**H**), and 32 (MeOH). In this case, ions with *m*/*z* 141 (**III**, **III'**, $[M_4]^{+\cdot}$) could originate from the molecular ions of **IV** and **IV'** via expulsion of methanol, from the molecular ion of pyridine **II** via McLafferty rearrangement or transfer of the β-hydrogen atom to oxygen, and from oxonium ion **G** via elimination of $[C_2H_3]^+$. Moreover, raising the temperature to 210°C appreciably increases the intensity of ion peaks with *m*/*z* 213, 168, 141, and 108, while ions **IV**, **IV'**, and **D**–**F** resulting from fragmentation of molecular ion $[M_1]^{+\cdot}$ strongly decrease in intensity (Table 2). No molecular ion peak of dihydropyridine **I** (*m*/*z* 245) was detected at that temperature. The presence of ion peak with *m*/*z* 59 (**I**) is likely to result from thermal transformations of the molecular ion of pyridine **II** and/or its derivatives, and/or the corresponding fragment ions. At lower temperature (70–150°C), ion peak with *m*/*z* 59 was not detected.

We can conclude that dihydropyridine **I** at 170–210°C loses methanol molecule (thermal process), yielding pyridine **II**. However, further raising the temperature (to 250°C) leads to disappearance of the molecular ion of **II** (*m*/*z* 213) from the mass spectrum. It should be emphasized that in the spectra obtained at the same temperature but at lower ionization energy (12 eV) the intensity of ion peaks with *m*/*z* 73 (100%) and 72 (40%) sharply increases. These ions are likely to be formed from 5,6-dihydropyridin-3-ol (**IV**) or 5,6-dihydropyridin-3(4*H*)-one (**IV'**), for both dihydropyridine **I** and pyridine **II** have already lost the 1-ethoxyethoxy fragment as source of ion **A** (*m*/*z* 73). Therefore, ions with *m*/*z* 73 and 72 may be assigned to 2-methoxyaziridine (**V**) or methyl thiocyanate (**VI**) as possible products of decomposition of compounds **IV** and/or **IV'** (Scheme 3).

For comparison, we examined the mass spectrum of pure pyridine **II** at 60 eV, the ion source and direct inlet probe temperatures being 50°C (Scheme 4). The spectrum was identical to the mass spectrum of di-

Scheme 4.



hydropyridine **I** recorded at 210°C. Like 3-alkylpyridines with branched substituents [8], for which the contribution of McLafferty rearrangement is comparable to that of cleavage of the C–C bond, the decomposition channel involving McLafferty rearrangement prevailed for pyridine **II**. Elimination of ethoxyethene molecule from the molecular ion of **II** (*m*/*z* 213) gives ion $[M_4]^{+\cdot}$ [*m*/*z* 141 (100%)] which has the structure of 2-methylsulfanylpyridin-3(4*H*)-one (**III'**) or tautomeric 2-methylsulfanylpyridin-3-ol (**III**). The presence in the mass spectrum of peaks from ions **H** and **J**–**L**, corresponding to decomposition of $[M_4]^{+\cdot}$, also counts in favor of the above fragmentation channel. Expulsion of SH radical from the molecular ion $[M_4]^{+\cdot}$ gives ion with *m*/*z* 108 which may be identified as 4-oxo-4*H*-azepinium ion **H** [8]. The formation of azepinium ion via elimination of SH radical was also observed in the fragmentation of 3-methoxy-2-methylsulfanylpyridine [4]. The subsequent fragmentation of ion **H** involves loss of CO molecule or 1*H*-azirine, leading to ions **J** and **K**, respectively. The decomposition pattern of ion $[M_4]^{+\cdot}$ (*m*/*z* 141) (formed by fragmentation of the molecular ions of **I** and **II**) is identical to that observed for an authentic sample of 2-methylsulfanylpyridin-3-ol (**III**), both synthesized by us [5] and described previously [7].

On the other hand, the fragmentation pathway typical of acetals (initial dissociation of the C–O bond with formation of oxonium ions **A** and **G** and elimination of RO radical from the molecular ion) is not preferred for compound **II**; this follows from the intensity of peaks from fragment ions **A** and **G**, *m*/*z* 73 (70%) and 168 (30%), respectively.

Our results lead us to conclude that fragmentation at 12 and 60 eV of molecular ions derived from six-membered nitrogen-containing heterocycles having a 1-ethoxyethoxy substituent (acetal moiety) at C^5 (**I**) or C^3 (**II**) follows two main pathways: (1) simple dissociation of the C–O bond in the acetal fragment to give oxonium ions **A** (*m*/*z* 73, from **I** and **II**) and **G** (*m*/*z* 168, from **II**); and (2) facile McLafferty rearrangement leading to ions with *m*/*z* 173 (**IV**, **IV'**) and 141 (**III**, **III'**), respectively. We were the first to observe McLafferty rearrangement in the series of dihydropyridines and acetals. No ion peak resulting from elimination of methanol molecule from the molecular ion of dihydropyridine **I** ($[M_1]^{+\cdot}$) was detected at 50–170°C; i.e., dihydropyridine **I** does not lose methanol below 170°C in a vacuum. The mass spectrum of dihydropyridine **I** recorded at 210°C is identical to the spectrum of pyridine **II**.

EXPERIMENTAL

The mass spectra of compounds **I**–**III** were recorded on an LKB-2091 GC–MS system at energies of ionizing electrons of 12 and 60 eV; samples were admitted into the ion source either through a chromatographic column (SE-54 capillary column, 38 m× 0.25 mm, film thickness 0.25 μm; carrier gas helium; injector temperature 250°C; interface temperature 250°C; oven temperature programming from 50 to 250°C at a rate of 10 deg/min) or through a direct inlet probe. The ion source and direct inlet probe temperatures were varied from 40 do 250°C; accelerating voltage 2.3 kV.

5-(1-Ethoxyethoxy)-2-methoxy-6-methylsulfanyl-2,3-dihydropyridine (**I**), 3-(1-ethoxyethoxy)-2-methylsulfanylpyridine (**II**), and 2-methylsulfanylpyridin-3-ol (**III**) were synthesized as described in [5].

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