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Uncommon Reaction of 2-Bromomethyl-1,3-thiaselenole with Pyridine and Its Derivatives

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Abstract—Regioselective reaction of 2-bromomethyl-1,3-thiaselenole with pyridine and its derivatives is followed by rearrangement with ring expansion and the formation of a bond between a nitrogen atom and a carbon in the position *2*. A set of derivatives of 2,3-dihydro-1,4-thiaselenine was obtained, substituted in the position *2* by a pyridinium residue functionalized by pharmacophoric groups.

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N- and Se-heterocycles possess a wide spectrum of biological activity [1–3]. Compounds, where are simultaneously present selenium and nitrogen heterocycles, are described in [4–15]. Some representatives of such systems possess various types of bioactivity [7–10]. Combining useful properties of N- and Se-heterocycles in one molecule may result in creation of compounds with important biological properties, so the search for methods of synthesis of such heterocyclic systems is an actual task.

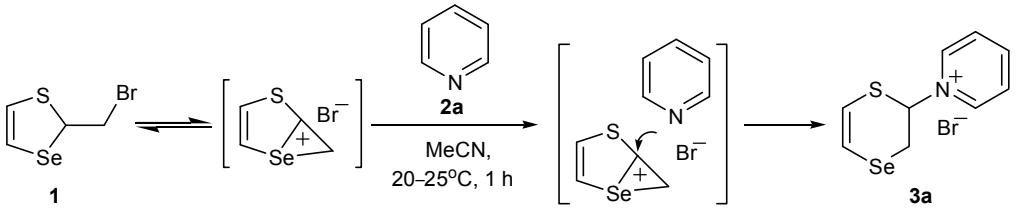
We developed a one-pot method for the preparation of 2-bromomethyl-1,3-thiaselenole **1** based on the reaction of selenium dibromide with divinyl sulfide [16, 17]. Compound **1** shows unusual reactivity in reactions of nucleophilic substitution with nucleophiles of diverse origin. The reactions proceed regioselectively at several centers of intermediate seleniranium cation [17–21].

We found a reaction of nucleophile substitution to occur in 2-bromomethyl-1,3-thiaselenole with pyridine and its derivatives in acetonitrile at room temperature which is followed by rearrangement with a ring expansion. The reaction with pyridine **2a** results in the formation of 1-(2,3-dihydro-1,4-thiaselenin-2-yl)pyridinium bromide **3a** in 85% yield. The reaction product is a yellow powder easily soluble in water and alcohol, sparingly soluble in chloroform.

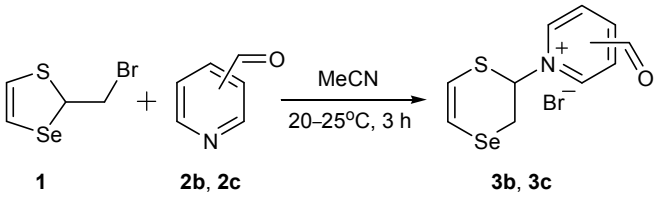
Reaction proceeds regioselectively with the formation of a bond of the nitrogen atom not with the carbon atom of CH_2Br group, but with the carbon atom in position *2* of the generated 2,3-dihydro-1,4-thiaselenine ring. A product of reaction with the preservation of the five-membered ring is not formed even in traces. It may be assumed that in the reaction of nucleophilic substitution in 2-bromomethyl-1,3-thiaselenole with pyridine in bipolar aprotic solvent (acetonitrile) a seleniranium cation intermediately forms with subsequent nucleophilic attack on the nodal carbon atom of seleniranium cation. As a result a ring expansion occurs with the formation of 2,3-dihydro-1,4-thiaselenine substituted in position *2* (Scheme 1).

Similar reaction with pyridine-3- and -4-carbaldehydes **2b** and **2c** (Scheme 2) after 3 h led to

Scheme 1.



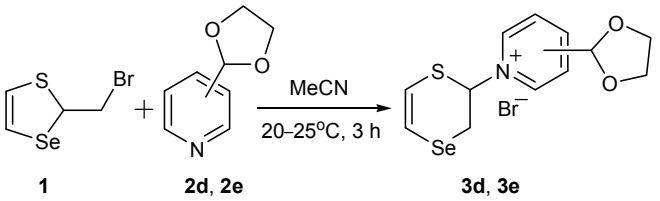
Scheme 2.



the formation of bromides 1-(2,3-dihydro-1,4-thiaselenin-2-yl)-3-formylpyridinium and -4-formylpyridinium **3b** and **3c** in yields 97 and 96% respectively. The reaction with pyridine-2-carbaldehyde in these conditions does not occur, evidently, as a result of steric hindrances and deactivation of pyridine nitrogen atom by the electron-acceptor aldehyde group.

The reaction of 2-bromomethyl-1,3-thiaselenole with 3- and 4-(1,3-dioxolan-2-yl)pyridines **2d** and **2e** proceeds in MeCN at room temperature during 3 h (Scheme 3). The reaction with compound **2d** occurs regioselectively with the formation of 1-(2,3-dihydro-1,4-thiaselenin-2-yl)-3-(1,3-dioxolan-2-yl)pyridinium bromide **3d** in 84% yield. From compound **2e** 1-(2,3-dihydro-1,4-thiaselenin-2-yl)-4-(1,3-dioxolan-2-yl)-pyridinium bromide **3e** was obtained in 94% yield.

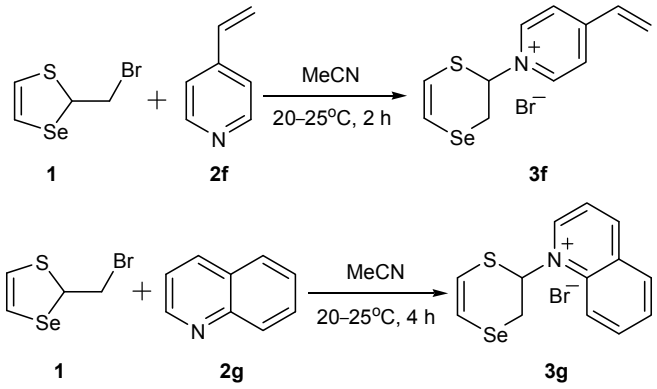
Scheme 3.



In reaction of compound **1** with 4-ethenylpyridine **2f** 1-(2,3-dihydro-1,4-thiaselenin-2-yl)-4-ethenylpyridinium bromide **3f** was obtained in 92% yield. Compound **3f** is interesting as a potential monomer. The reaction with quinoline **2g** occurs in acetonitrile at room temperature and results in 1-(2,3-dihydro-1,4-thiaselenin-2-yl)quinolinium bromide **3g** in 92% yield (Scheme 4).

The structure of compounds **3a–3g** was confirmed by multinuclear NMR spectroscopy (^{1}H, ^{13}C, ^{15}N, ^{77}Se), the composition was confirmed by the data of elemental analysis. In ^{1}H NMR spectra of compounds **3a–3g** signals of protons of CH_2Se groups were observed as two doublets at 3.22–3.99 ppm. Signals of protons of SCH group of 2,3-dihydro-1,4-thiaselenine

Scheme 4.



cycle are shifted downfield (6.52–8.60 ppm) due to the presence of a positive charge on the nitrogen atom. The formation of six-membered ring is confirmed by the direct spin-spin coupling constant between the atoms of selenium and carbon of the CH_2 group in the fine structure ^{13}C and ^{77}Se NMR spectra. The values of coupling constants 71.1–72.3 Hz are in the range typical of direct constants $^1J_{Se,C}$ [22].

Compounds **3b** and **3c** are poorly soluble in water and DMSO. At registration of NMR spectra in D_2O compounds **3b** and **3c** fully transform into dihydroxymethyl derivatives as a result of hydration of the aldehyde group. However in the IR spectrum of compounds **3b** and **3c** typical bands of absorption of carbonyl group at 1706–1710 cm^{-1} are observed.

Hence applying a new regioselective reaction of 2-bromomethyl-1,3-thiaselenole with pyridine and its derivatives we obtained in high yields a set of previously unknown derivatives of 2,3-dihydro-1,4-thiaselenine, functionalized with pharmacophoric groups containing a pyridine ring. Compounds **3a–3g** probably possess bioactivity.

EXPERIMENTAL

IR spectra were registered on a spectrophotometer Bruker Vertex 70 from pellets with KBr. ^{1}H, ^{13}C, ^{15}N, and ^{77}Se NMR spectra were obtained on a spectrometer Bruker DPX-400 (400.13, 100.61, 40.56, and 76.30 MHz respectively). Elemental analysis was carried out on a CHNS-analyzer Thermo Scientific Flash 2000. 2-Bromomethyl-1,3-thiaselenole **1** was obtained by method [17].

3-(1,3-Dioxolan-2-yl)pyridine (2d). To a solution of 2.14 g (0.02 mol) of pyridine-3-carbaldehyde cooled

to 0°C and 2.3 g of ethylene glycol in 30 mL of toluene was added dropwise while stirring 1 mL of concentrated sulfur acid. The mixture was heated for 2 h to boiling, water was distilled with toluene from the reactive flask. Then the mixture was cooled to 0°C and 10% solution of NaOH was added dropwise to neutralize sulfur acid till weak alkaline reaction. The reaction products were extracted with ethyl acetate, the organic solution was dried with Na_2SO_4, the solvent was distilled off on a rotary evaporator. Yield 2.36 g (78%).

4-(1,3-Dioxolan-2-yl)pyridine (2e) was obtained similary in 80% yield.

Compounds 3a–3g. General procedure. To a solution of 0.488 g (2.00 mmol) of 2-bromomethyl-1,3-thiaselenole **1** in 5 mL of acetonitrile was added 2.00 mmol of compound **2a–2g**, and the mixture was intensively stirred at room temperature for 1–4 h. The precipitate was filtered off, washed with acetonitrile, and dried in a vacuum. Liquid products of reaction **3c** and **3e** were dried in a vacuum after distillation of acetonitrile.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)pyridinium bromide (3a). Reaction time 1 h. Yield 0.549 g (85%). Yellow-brown powder, mp 135–137°C (with decomposition). ^{1}H NMR spectrum (CD_3OD), δ, ppm: 3.50 d.d (1H, $SeCH_2$, ^2J 13.6, ^3J 5.3 Hz), 3.68 d.d (1H, $SeCH_2$, ^2J 13.6, ^3J 2.2 Hz), 6.86 d.d (1H, $SCHN^+$, ^3J 5.3, ^3J 2.2 Hz), 6.78 d (1H, SC<u>H</u>=CHSe, ^3J 10.0, $^3J_{H,Se}$ 21.0 Hz), 6.78 d (1H, SCH=C<u>H</u>Se, ^3J 10.0, $^2J_{=CH-Se}$ 52.0 Hz), 8.21 d.d (2H, $H^{3,5}$, C_5H_5N, ^3J 7.5, ^3J 6.9 Hz), 8.71 t.t (1H, H^4, C_5H_5N, ^3J 7.5, ^4J 1.2 Hz), 9.18 d.d (2H, $H^{2,6}$, C_5H_5N, ^3J 6.9, ^4J 1.2 Hz). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 25.67 (CH_2Se), 67.35 ($SCHN^+$), 112.62 (=CHSe, $^1J_{C-Se}$ 114.6 Hz), 117.72 (=CHS), 129.00 ($C^{3,5}$, C_5H_5N), 144.89 ($C^{2,6}$, C_5H_5N), 148.11 (C^4, C_5H_5N). ^{15}N NMR spectrum (CD_3OD), δ, ppm: –169.7. ^{77}Se NMR spectrum (CD_3OD), δ, ppm: 85.1. Found, %: C 33.42; H 3.11; N 4.21; S 9.91. $C_9H_{10}BrNSSe$. Calculated, %: C 33.46; H 3.12; N 4.33; S 9.92.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)-3-formylpyridinium bromide (3b). Reaction time 3 h. Yield 0.681 g (97%), brown powder, mp 129–130°C. IR spectrum, ν, cm^{-1}: 1706 (C=O). In D_2O it is present as 1-(2,3-dihydro-1,4-thiaselenin-2-yl)-3-(dihydroxymethyl)pyridinium bromide. ^{1}H NMR spectrum (D_2O), δ, ppm: 3.22 d.d (1H, $SeCH_2$, ^2J 13.3, ^3J 5.1 Hz), 3.60 d.d (1H, $SeCH_2$, ^2J 13.3, ^3J 2.1 Hz), 6.15 s (1H, OCHO), 6.53 d.d (1H, $SCHN^+$, ^3J 5.1, ^3J 2.1 Hz), 6.61 d (1H, SCH=, ^3J 10.4 Hz), 6.64 d (1H, SeCH=, ^3J 10.4, $^2J_{Se,H}$ 47.2 Hz), 8.02 d.d (1H, H^5, C_5H_4N, ^3J 6.1, ^3J 8.1 Hz), 8.58 d (1H, H^4, C_5H_4N, ^3J 8.1 Hz), 8.91 d (1H, H^6, C_5H_4N, ^3J 6.1 Hz), 8.99 s (1H, H^2, C_5H_4N). ^{13}C NMR spectrum (D_2O), δ, ppm: 24.13 (CH_2Se), 65.81 ($SCHN^+$), 86.54 (OCHO), 111.15 (=CHSe), 116.23 (=CHS), 127.33 (C^5, C_5H_4N), 140.95 (C^2, C_5H_4N), 142.35 (C^3, C_5H_4N), 142.84 (C^6, C_5H_4N), 144.31 (C^4, C_5H_4N). ^{15}N NMR spectrum (D_2O), δ, ppm: –156.0. ^{77}Se NMR spectrum (D_2O), δ, ppm: 81.1. Found, %: C 33.94; H 2.80; N 4.21. $C_{10}H_{10}BrNOSSe$. Calculated, %: C 34.21; H 2.87; N 3.99.

1-(2,3-Dihydro-1,4-thiaselenin)-4-formylpyridinium bromide (3c). Reaction time 3 h. Yield 0.674 g (96%). Brown oily substance. IR spectrum, ν, cm^{-1}: 1710 (C=O). In D_2O it is present as 1-(2,3-dihydro-1,4-thiaselenin)-4-(dihydroxymethyl)pyridinium bromide. ^{1}H NMR spectrum (D_2O), δ, ppm: 3.22 d.d (1H, $SeCH_2$, ^2J 13.5, ^3J 5.3 Hz), 3.57 d.d (1H, $SeCH_2$, ^2J 13.5, ^3J 2.1 Hz), 6.11 s (1H, OCHO, J 17.7, 10.8 Hz), 6.52 br.d.d (1H, $SCHN^+$, ^3J 5.3, ^3J 2.1 Hz), 6.59 d (1H, SC<u>H</u>=CHSe, ^3J 10.4 Hz), 6.61 d (1H, SCH=C<u>H</u>Se, ^3J 10.4, $^2J_{=CH-Se}$ 47.0 Hz), 8.06 d (2H, $H^{3,5}$, C_5H_4N, ^3J 6.7 Hz), 8.92 d (2H, $H^{2,6}$, C_5H_4N, ^3J 6.7 Hz). ^{13}C NMR spectrum (D_2O), δ, ppm: 25.40 (CH_2Se), 66.62 ($SCHN^+$), 88.58 (OCHO), 112.47 (=CHSe), 117.48 (=CHS), 125.90 ($C^{3,5}$, C_5H_4N), 144.59 ($C^{2,6}$, C_5H_4N), 162.00 (C^4, C_5H_4N). ^{15}N NMR spectrum (D_2O), δ, ppm: –156.6. ^{77}Se NMR spectrum (D_2O), δ, ppm: 82.3. Found, %: C 33.94; H 2.80; N 4.21. $C_{10}H_{10}BrNOSSe$. Calculated, %: C 34.21; H 2.87; N 3.99.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)-3-(1,3-dioxolan-2-yl)pyridinium bromide (3d). Reaction time 3 h. Yield 0.664 g (84%). Red-orange powder, mp 138–139°C. ^{1}H NMR spectrum (D_2O), δ, ppm: 3.56 narrow m (2H, $SeCH_2$), 4.05 m (4H, OCH_2CH_2O), 6.15 s (1H, OCHO), 6.96 t (1H, $SCHN^+$, ^3J 3.0 Hz), 6.84 d (2H, SeCH=, ^3J 10.0, $^2J_{SeH}$ 47.0 Hz), 6.88 d (2H, SCH=, ^3J 10.0 Hz), 8.30 d.d (1H, H^5, C_5H_4N, ^3J 7.8, ^3J 6.1 Hz), 8.72 d (1H, H^4, C_5H_4N, ^3J 7.8 Hz), 9.12 d (1H, H^6, C_5H_4N, ^3J 6.1 Hz), 9.14 m (1H, H^2, C_5H_4N). ^{13}C NMR spectrum (D_2O), δ, ppm: 24.64 (CH_2Se, $^1J_{SeC}$ 71.1 Hz), 65.27 ($SCHN^+$), 65.45 (OCH_2), 99.12 (OCHO), 111.93 (=CHSe, $^1J_{SeC}$ 113.8 Hz), 117.35 (=CHS), 127.90 (C^5, C_5H_4N), 138.79 (C^3, C_5H_4N), 141.46 (C^2, C_5H_4N), 144.12 (C^4, C_5H_4N), 144.89 (C^6, C_5H_4N). ^{15}N NMR spectrum (D_2O), δ, ppm: –153.2. ^{77}Se NMR spectrum (D_2O), δ, ppm: 85.3. Found, %: C 36.23; H 3.89; N 3.82. $C_{12}H_{14}BrNO_2SSe$. Calculated, %: C 36.47; H 3.57; N 3.54.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)-4-(1,3-dioxolan-2-yl)pyridinium bromide (3e). Reaction time 3 h. Yield 0.743 g (94%). Brown oily substance. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.66 d.d (1H, $SeCH_2$, ^2J 13.9, ^3J 4.4 Hz), 3.71 d.d (1H, $SeCH_2$, ^2J 13.9, ^3J 2.6 Hz), 4.07–4.11 m (2H, OCH_2CH_2O), 6.03 s (1H, OCHO), 6.64 s (2H, SeCH=CHS), 7.78 s (1H, $SCHN^+$), 8.10 d (2H, $H^{3,5}$, C_5H_4N, ^3J 6.5 Hz), 9.72 d (2H, $H^{2,6}$, C_5H_4N, ^3J 6.5 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 25.74 (CH_2Se, $^1J_{C–Se}$ 72.3 Hz), 64.63 ($SCHN^+$), 65.85 (OCH_2), 99.95 (OCHO), 111.16 (=CHSe), 116.68 (=CHS), 124.70 ($C^{3,5}$, C_5H_4N), 144.19 ($C^{2,6}$, C_5H_4N), 157.85 (C^4, C_5H_4N). ^{15}N NMR spectrum ($CDCl_3$), δ, ppm: –153.8. ^{77}Se NMR spectrum ($CDCl_3$), δ, ppm: 77.9. Found, %: C 36.33; H 3.58; N 3.32. $C_{12}H_{14}BrNO_2SSe$. Calculated, %: C 36.47; H 3.57; N 3.54.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)-4-(vinyl)pyridinium bromide (3f). Reaction time 2 h. Yield 0.643 g (92%). Yellow powder, mp 146–147°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.52 d.d (1H, $SeCH_2$, ^2J 13.5, ^3J 5.4 Hz), 3.56 d.d (1H, $SeCH_2$, ^2J 13.5, ^3J 2.5 Hz), 6.05 d (1H, $=CH_2$, $^3J_{cis}$ 10.8 Hz), 6.64 d (1H, $=CH_2$, $^3J_{trans}$ 17.7 Hz), 6.77 br.d.d (1H, SCH N^+, ^3J 5.4, ^3J 2.5 Hz), 6.82 d (1H, SeCH=, ^3J 10.3 Hz), 6.86 d (1H, SCH=, ^3J 10.3 Hz), 7.05 d.d (1H, C<u>H</u>=CH_2, J_{trans} 17.7, J_{cis} 10.8 Hz), 8.28 d (2H, $H^{3,5}$, C_5H_4N, ^3J 7.2 Hz), 8.98 d (2H, $H^{2,6}$, C_5H_4N, ^3J 7.2 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 24.46 (CH_2Se), 64.57 ($SCHN^+$), 111.83 (=CHSe), 117.44 (=CHS), 123.98 ($C^{3,5}$, C_5H_4N), 128.63 (CH=<u>C</u>H_2), 132.43 (<u>C</u>H=CH_2), 143.55 ($C^{2,6}$, C_5H_4N), 153.77 (C^4, C_5H_4N). ^{15}N NMR spectrum (DMSO-d_6), δ, ppm: –161.1. ^{77}Se NMR spectrum (DMSO-d_6), δ, ppm: 91.7. Found, %: C 38.03; H 3.44; N 3.84. $C_{11}H_{12}BrNSSe$. Calculated, %: C 37.84; H 3.46; N 4.01.

1-(2,3-Dihydro-1,4-thiaselenin-2-yl)quinolinium bromide (3g). Reaction time 4 h. Yield 0.687 g (92%). Yellow-brown powder, mp 135–136°C (with decomposition). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.30 d.d (1H, $SeCH_2$, ^2J 13.8, ^3J 4.7 Hz), 3.99 d.d (1H, $SeCH_2$, ^2J 13.8, ^3J 1.7 Hz), 6.61 d (1H, =CHSe, ^3J 10.4, $^2J_{=CH–Se}$ 49.2 Hz), 6.74 d (1H, =CHS, ^3J 10.4 Hz), 7.97 t (1H, H^6, C_9H_7N, ^3J 7.6 Hz), 8.15 d.d (1H, H^3, C_9H_7N, ^3J 8.2, ^3J 6.1 Hz), 8.35 d.d (1H, H^7, C_9H_7N, ^3J 7.6, ^3J 9.4 Hz), 8.36 d (1H, H^5, C_9H_7N, ^3J 7.6 Hz), 8.60 br.d (1H, $SCHN^+$, ^3J 4.7 Hz), 9.14 d (1H, H^4, C_9H_7N, ^3J 8.2 Hz), 9.26 d (1H, H^2, C_9H_7N, ^3J 6.1 Hz), 9.64 d (1H, H^8, C_9H_7N, ^3J 9.4 Hz). ^{13}C NMR spectrum, δ, ppm: 24.67 (CH_2Se), 62.52 (SCHN), 109.88 (=CHSe), 117.52 (=CHS), 121.08 (C^3, C_9H_7N), 122.56 (C^{8a}, C_9H_7N), 129.5 (C^{4a}, C_9H_7N), 130.99 (C^6, C_9H_7N), 131.12 (C^7, C_9H_7N), 138.06 (C^5, C_9H_7N), 146.93 (C^2, C_9H_7N), 148.16 (C^4, C_9H_7N), 137.65 (C^{8a}, C_9H_7N). ^{77}Se NMR spectrum, δ, ppm: 78.2. Found, %: C 41.42; H 3.11; N 4.02. $C_{13}H_{12}BrNSSe$. Calculated, %: C 41.84; H 3.24; N 3.75.

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