Reactions of Chloromethyloxirane and Dihalopropanols with Chalcogens in Basic Reducing Systems

A. V. Elaev, V. A. Grabel'nykh, N. V. Russavskaya, E. P. Levanova, E. N. Sukhomazova, E. R. Zhanchipova, L. V. Klyba, A. I. Albanov, and N. A. Korchevin

> Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia e-mail: venk@irioch.irk.ru

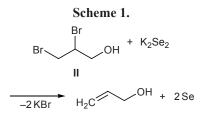
> > Received September 5, 2006

Abstract—Chloromethyloxirane and 2,3-dibromopropan-1-ol reacted with a solution of selenium or tellurium in the system hydrazin hydrate–potassium hydroxide (K₂Se₂, K₂Te₂) to give allyl alcohol; the reaction was accompanied by regeneration of the initial free chalcogen. 1,3-Dichloropropan-2-ol reacted with selenium in the same system to give oligomeric product having a 2-hydroxypropane-1,3-diyldiseleno monomeric unit, while the reaction with tellurium led to the formation of allyl alcohol and almost complete regeneration of initial tellurium. Probable reaction mechanisms are discussed. Polyselenide oligomers containing a hydroxy group in a monomeric unit were formed in reactions of chloromethyloxirane and 1,3-dichloropropan-2-ol with selenium in the system hydrazine hydrate–2-aminoethanol. Under analogous conditions 2,3-dibromopropan-1-ol was converted into allyl alcohol with regeneration of elemental selenium. Reductive cleavage of polyselenide oligomers gave Se-methyl derivatives of 2-hydroxypropane-1,3-diselenol.

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Chloromethyloxirane (I), 2,3-dibromopropan-1-ol (II), and 1,3-dichloropropan-2-ol (III) were reported to react with sulfur in the system hydrazine hydrate–alkali–water, yielding polysulfide oligomers with hydroxy groups in their macromolecules [1]. The resulting oligomers underwent reductive cleavage at the S–S bonds by the action of an alkaline solution of hydrazine hydrate with no other solvent. These transformations underlay simple preparative procedures for the synthesis of dithioglycerols and their S-methyl derivatives. In the reactions with any compound I–III, mixtures of isomeric dithioglycerols were formed, i.e., the process was accompanied by migration of the hydroxy group [1].

With a view to reveal other relations holding in reactions of polyelectrophilic chloromethyloxirane and dihalopropanols with chalcogenide, in the present



work we examined reactions of compounds I–III with selenium and tellurium in hydrazine-based basic reducing systems. We anticipated that these reactions will lead to the formation of oligomeric polychalcogenides having hydroxy groups, as well as seleniumand tellurium-containing glycerol analogs which can be regarded as valuable multidentate ligands for complex formation and reagents for organic synthesis.

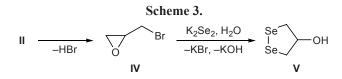
However, 2,3-dibromopropan-1-ol (II) reacted with selenium activated in the systems hydrazine hydratepotassium hydroxide-water and hydrazine hydrate-2-aminoethanol (in both cases, diselenide ions were generated [2]) to give allyl alcohol (vield 50–74%) with regeneration of elemental selenium (yield 66-76%) (Scheme 1). The reaction follows a scheme analogous to that reported for the reaction of 1,2-dichloroethane with potassium diselenide [3]. Separation of elemental selenium was observed after addition of a required amount dibromide II to a solution of selenium in the above systems, but the mixture remains intensely colored, indicating the presence of polyselenides. It is known [4] that polyselenides can be formed from elemental selenium and hydrazine (Scheme 2); i.e., a reaction reverse to the dissolution of selenium

Scheme 2.

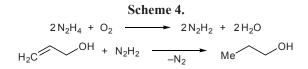
$$2xSe + 5N_2H_4 \cdot H_2O \longrightarrow 2(N_2H_5)_2Se_x + N_2 + 5H_2O$$

occurs. Presumably, this is the reason why regeneration of elemental selenium in the reaction shown in Scheme 1 is not complete.

Allyl alcohol was isolated by extraction of the reaction mixture with diethyl ether and was identified by ¹H NMR spectroscopy. Apart from allyl alcohol, the extract contained (according to the GC–MS data) initial dibromide II (its amount corresponded to a conversion of ~92%), bromomethyloxirane (IV) (~3%, calculated on the initial dibromide II), and 4-hydroxy-1,2-diselenolane (V) (yield 5%). Compounds IV and V were likely to be formed according to Scheme 3. The mass spectrum of bromomethyloxirane (IV) contained the molecular ion peak as a doublet typical of the presence of one bromine atom in the molecule (m/z 136/138). The base peak in the spectrum had an m/z value of 57; it originated from elimination of bromine atom from the molecular ion.



When the reaction of II with selenium was performed for a longer time, the reaction mixture also contained propan-1-ol which was formed via hydrogenation of allyl alcohol with the system N_2H_4 – KOH–O₂. It is known that the latter generates diimide [5] (Scheme 4). The yield of propyl alcohol attains 38% when the reaction time is more than 3 h, and it can even exceed the yield of allyl alcohol. The hydrogenation of allyl alcohol is favored by its good solubility in the system hydrazine hydrate–alkali.

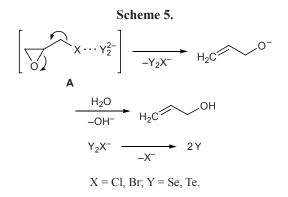


In the mass spectrum of 1,2-diselenolane V the molecular ion gives rise to a low-intensity peak cluster $(m/z \ 218 \ \text{for}^{80}\text{Se})$. The base peak belongs to the fragment ion with $m/z \ 58$, which is formed as a result of elimination of two selenium atoms from the molecular ion. In addition, 1,2-diselenolane V was identified by ¹H NMR spectroscopy (see Experimental).

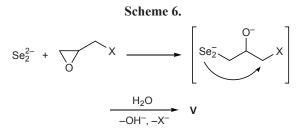
Chloromethyloxirane (I) reacted with selenium in the system hydrazine hydrate–alkali in a way similar to

dibromide II. The reaction resulted in regeneration of elemental selenium (60%) and formation of allyl alcohol (52%) and a small amount (2–3%) of 4-hydroxy-1,2-diselenolane V. Likewise, in the reactions of all compounds I–III with tellurium in the system hydrazine hydrate–KOH (K_2Te_2 [6]), tellurium was recovered from the reaction mixtures almost quantitatively, and the yield of allyl alcohol ranged from 58 to 62%. No 4-hydroxy-1,2-ditellurolane was detected among the products.

We believe that in all cases the process involves the corresponding halomethyloxirane [elimination of hydrogen bromide from 2,3-dibromopropan-1-ol (II) in basic medium gives 2-bromomethyloxirane]. Halomethyloxiranes are capable of reacting with diselenide and ditelluride ions along two pathways. The first of these is common S_N2 nucleophilic replacement of halogen, and the second is halophilic attack by Y_2^{2-} ion on the halogen atom [3]. Taking into account higher electronegativity of the oxygen atom as compared to chlorine or bromine [7], halophilic attack by dichalcogenide ion on halomethyloxirane through intermediate state **A** (Scheme 5) should occur more readily than its attack on 1,2-dihaloethane [3].

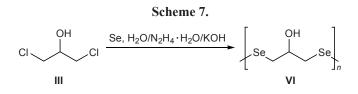


The other reaction direction implies nucleophilic attack by Y_2^{2-} on the methylene carbon atom in the oxirane ring; our results showed that this reaction pathway is observed only for selenium (Scheme 6). Oligomeric products could be formed according to a similar scheme. However, we detected no oligomeric products



in the reactions of compounds I and II with selenium and tellurium in the system hydrazine hydrate–alkali.

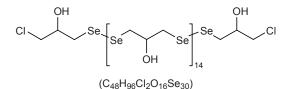
Unlike compounds I and II, 1,3-dichloropropan-2ol (III) reacted with selenium in the system hydrazine hydrate–potassium hydroxide–water to give oligomer VI (Scheme 7).



Compounds structurally related to VI can also be obtained from chloromethyloxirane (I) and 1,3-dichloropropan-2-ol (III) by reaction with selenium in the system hydrazine hydrate–2-aminoethanol at a molar ratio of 10:1 (Scheme 8).

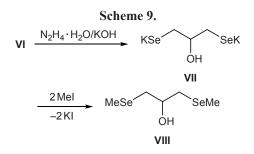
Scheme 8.
I. III Se,
$$N_2H_4 \cdot H_2O/H_2NCH_2CH_2OH \rightarrow VI$$

In all cases, oligomers VI were formed in nearly quantitative yield (97–99%) as viscous orange–red substances; they contained about 70% of selenium and residual chlorine (see Experimental). Assuming that the residual chlorine atoms are located at the ends of the macromolecule, the molecular weight of the oligomeric product was estimated at 3330 a.m.u. The data of elemental analysis are very consistent with the general formula $C_{48}H_{96}Cl_2O_{16}Se_{30}$.

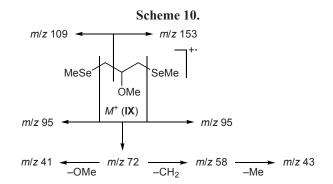


By extraction of the crude oligomeric product with acetone or other organic solvents we isolated 4-hydroxy-1,2-diselenolane (V) which was likely to be formed concurrently with the polycondensation process. The yield of V, calculated on the initial selenium, was about 5%. Despite poor yield, we succeeded in isolating compound V as individual substance.

Like other diselenide oligomers [2], oligomer VI underwent cleavage at the Se–Se bonds in the system hydrazine hydrate–KOH; the reaction was accompanied by complete dissolution, and the resulting diselenolate VII was treated *in situ* with methyl iodide. We thus isolated 48–52% of 1,3-bis(methylselanyl)-



propan-2-ol (VIII) (Scheme 9). The structure of compound VIII was confirmed by the ¹H and ¹³C NMR and mass spectra. Our results showed that only one isomer of bis(methylselanyl)propanol VIII is formed with 1,3-arrangement of the selenium atoms. No 2,3-bis-(methylselanyl)propan-1-ol was detected (cf. [1]). Among the reaction products we also identified by GC–MS 2-methoxy-1,3-bis(methylselanyl)propane (IX, yield ~2%). Its fragmentation pattern under electron impact is illustrated by Scheme 10; it unambiguously indicates that the methoxy group is attached to the C² atom.



Taking into account the direction of reactions of chloromethyloxirane derivatives with selenium and tellurium in the systems hydrazine hydrate–base, the absence of products with vicinal arrangement of the selenium atoms (in the system hydrazine hydrate– 2-aminoethanol), and previously reported data on the reactions of 1,2-dihaloethanes with selenium and tellurium [3], we can conclude that the examined reactions of 1,2-dielectrophiles with selenium- and tellurium-containing anions are fairly general. The driving force of these processes is polarization of the dielectrophile molecule by the action of chalcogenide ion.

$$x - c - c - x' \cdots Y_n^{2-}$$

On the other hand, the polarizing effect of Y_n^{2-} is determined by its electron-donating power, i.e., it decreases in the series Te > Se > S [7], which is very consistent with our experimental data.

Thus, the systems hydrazine hydrate–alkali–water and hydrazine hydrate–2-aminoethanol make it possible to obtain oligomeric poliselenides containing hydroxy groups from selenium and 1,3-dichloropropan-2-ol. From chloromethyloxirane, such oligomers can be obtained only in the system hydrazine hydrate– 2-aminoethanol. Reductive cleavage of polyselenide oligomers provides a synthetic route to 2-hydroxypropane-1,3-diselenol derivatives.

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EXPERIMENTAL

The progress of reactions was monitored, and the liquid products were analyzed, by gas-liquid chromatography on LKhM 80-MD-2 (2000×3-mm column packed with 5% of DC-550 on Chromaton N-AW-HMDS; linear oven temperature programming at a rate of 12 deg/min; carrier gas helium) and Tsvet-500 instruments (2000×5-mm steel column packed with 5% of XE-60 on Chromaton N-AW-HMDS; linear oven temperature programming from 30 to 230°C at a rate of 12 deg/min: carrier gas helium). The IR spectra were measured on Specord 75IR and Bruker IFS-25 spectrometers from samples prepared as thin films. The ¹H and ¹³C NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.1 and 100.6 MHz, respectively, using CDCl₃ as solvent and hexamethyldisiloxane as internal reference. The mass spectra were obtained on a Shimadzu GCMS-QP5050A instrument (SPB-5 column, 60000×0.25 mm, film thickness 0.25 µm; injector temperature 250°C; carrier gas helium, flow rate 0.7 ml/min; oven temperature programming from 60 to 260°C at a rate of 15 deg/min; detector temperature 250°C; quadrupole mass analyzer, electron impact, 70 eV; ion source temperature 200°C; a.m.u. range 34-650).

Reaction of 2-chloromethyloxirane (I) with selenium in the system hydrazine hydrate–KOH– H_2O . A solution of potassium diselenide was prepared from 5.6 g (0.1 mol) of potassium hydroxide, 5 g (0.1 mol) of hydrazine hydrate, 30 ml of water, and 7.9 g (0.1 mol) of powdered selenium. The mixture was heated for 2 h at 80–85°C and cooled to 40°C, 4.6 g (0.05 mol) of chloromethyloxirane (I) was slowly added, and the mixture was stirred for 3 h at 60–70°C. During the process, a black solid separated and was filtered off, washed, dried under reduced pressure, and identified as elemental selenium (4.74 g, mp 217– 222°C). The filtrate was extracted with diethyl ether, the extract was evaporated, and the residue was analyzed by GLC, ¹H NMR, and GC–MS. Vacuum distillation gave 1.5 g of allyl alcohol, bp 47–54°C (115 mm). ¹H NMR spectrum, δ , ppm: 4.10 d (CH₂O), 5.12 d.d, 5.24 d.d (CH=), 5.95 m (=CH–), 3.23 br.s (OH); $J_{cis} = 10.5$, $J_{trans} = 17.3$, ³J = 5.1, ²J = 1.65, ⁴J = 1.6 Hz (*ABCX*₂ spin system). Ppropan-1-ol was identified by ¹H NMR as an impurity (yield 5%). When the reaction time was increased to 10 h, the yield of propan-1-ol reached 38%, while the yield of allyl alcohol fell down to 33%. 4-Hydroxy-1,2-diselenolane (**V**) was identified among the products by GC–MS; its ¹H NMR spectrum is given below.

Reaction of 2,3-dibromopropan-1-ol (II) with selenium in the system hydrazine hydrate-KOH- H_2O . A solution of potassium diselenide was prepared from 2.8 g (0.05 mol) of potassium hydroxide, 2.5 g (0.05 mol) of hydrazine hydrate, 15 ml of water, and 4.1 g (0.052 mol) of powdered selenium. The mixture was heated for 2 h at 80–85°C and cooled to 45°C, 8.2 g (0.038 mol) of 2,3-dibromopropan-1-ol (II) was slowly added, and the mixture was stirred for 7 h at 80-85°C. During the process, a black solid separated and was filtered off, washed, dried under reduced pressure, and identified as elemental selenium (2.7 g, mp 210–215°C). The filtrate was extracted with diethyl ether, the extract was dried over MgSO₄, the solvent was removed, the residue was cooled, and 0.31 g of a crystalline product was separated. According to the GLC, GC-MS, and ¹H NMR data, it was identified as 4-hydroxy-1,2-diselenolane (V). ¹H NMR spectrum of V [in a mixture with allyl alcohol, propan-1-ol, and 2,3-dibromopropan-1-ol (II)], δ, ppm: 5.35 m (CHO), 3.27 d and 3.40 d (CH₂Se), 3.80 s (OH). Protons in the CH₂ group of compound V are differently oriented with respect to the CHO proton; therefore, three spinspin couplings were detected with the following constants: $J_{AB} = 10.48$, $J_{AX} = 2.42$, $J_{BX} = 2.96$ Hz.



The liquid part of the residue (after separation of crystals) was distilled under reduced pressure. A fraction with bp $51-53^{\circ}$ C (115 mm), 1.27 g, was identified by GLC and ¹H NMR as allyl alcohol. Its spectral parameters are given above.

Reaction of 1,3-dichloropropan-2-ol (III) with selenium in the system hydrazine hydrate–KOH– $H_2O.$ 1,3-Dichloropropan-2-ol (III), 4.86 g (0.038 mol), was added dropwise to a solution of 3.95 g (0.05 mol) of selenium in a mixture of 2.5 g (0.05 mol) of hydra-

zine hydrate, 2.8 g (0.05 mol) of potassium hydroxide, and 15 ml of water, prepared as described above, and the mixture was stirred for 1 h at 60–70°C. An orange– red solid separated and was filtered off, washed with water and organic solvents, and dried under reduced pressure. We thus isolated 5.2 g of oligomer **VI** with mp 60–110°C (decomp.). IR spectrum, v, cm⁻¹: 3432, 2915, 2840, 1410, 1393, 1276, 1160, 740, 625, 435. Found, %: Cl 2.05; Se 71.04. $C_{48}H_{96}Cl_2O_{16}Se_{30}$. Calculated, %: Cl 2.11; Se 70.35. The filtrate was extracted with diethyl ether, the extract was dried over MgSO₄, and the solvent was removed. The residue, 1.13 g, was analyzed by GC–MS, It was identified as 4-hydroxy-1,2-diselenolane (**V**).

Reaction of 2-chloromethyloxirane (I) with tellurium in the system hydrazine hydrate-KOH. A solution of potassium ditelluride was prepared from 1.4 g (0.025 mol) of potassium hydroxide, 10 g (0.2 mol) of hydrazine hydrate, and 3.19 g (0.025 mol) of powdered tellurium. The mixture was heated for 2 h at 80-85°C and cooled to 45°C, 1.15 g (0.0125 mol) of 2-chloromethyloxirane (I) was added, and the mixture was stirred for 2.5 h at 50–52°C. During the process, a black powder-like solid separated and was filtered off, washed, dried under reduced pressure, and identified as elemental tellurium (3 g) (by elemental analysis). The filtrate was extracted with diethyl ether, the extract was dried over MgSO₄, and the solvent was removed to obtain a liquid residue which was distilled under reduced pressure. A fraction with bp 68-70°C (140 mm), 0.43 g, was identified as allyl alcohol on the basis of the GLC and ¹H NMR data. Neither initial chloromethyloxirane (I) nor 4-hydroxy-1,2-ditellurolane was detected.

Reaction of 2,3-dibromopropan-1-ol (II) with tellurium in the system hydrazine hydrate–KOH. A solution of potassium ditelluride was prepared as described above from 2.8 g (0.05 mol) of potassium hydroxide, 20 g (0.4 mol) of hydrazine hydrate, and 6.38 g (0.05 mol) of powdered tellurium. The mixture was cooled to 45°C, 5.45 g (0.025 mol) of 2,3-dibromopropan-1-ol (II) was added, and the mixture was stirred for 3 h at 60–70°C. Elemental tellurium, 6.2 g, separated from the mixture. The filtrate was treated as described above to isolate 0.84 g of allyl alcohol. Neither initial 2,3-dibromopropan-1-ol (II) nor 4-hydroxy-1,2-ditellurolane was detected.

Reaction of 1,3-dichloropropan-2-ol (III) with tellurium in the system hydrazine hydrate–KOH. A solution of potassium ditelluride was prepared as described above from 2.8 g (0.05 mol) of potassium hydroxide, 20 g (0.4 mol) of hydrazine hydrate, and 6.38 g (0.05 mol) of powdered tellurium. The mixture was cooled to 45°C, 3.2 g (0.025 mol) of 1,3-dichloropropan-2-ol (III) was slowly added, and the mixture was stirred for 2 h at 60–65°C. Elemental tellurium, 6.1 g, separated from the mixture. The filtrate was treated as described above to isolate 0.89 g of allyl alcohol. Neither initial 1,3-dichloropropan-2-ol (III) nor 4-hydroxy-1,2-ditellurolane was detected..

Reaction of 2,3-dibromopropan-1-ol (II) with selenium in the system hydrazine hydrat-2-aminoethanol. A solution of potassium diselenide was prepared from 7.5 g (0.15 mol) of hydrazine hydrate, 0.92 g (0.015 mol) of 2-aminoethanol, and 3.95 g (0.05 mol) of powdered selenium. The mixture was heated for 2 h at 70–75°C and cooled to 50°C, 8.16 g (0.037 mol) of 2,3-dibromopropan-1-ol (II) was added, and the mixture was stirred for 1.5 h at 65-70°C. During the process, a black solid separated and was filtered off, washed, dried under reduced pressure, and identified as elemental selenium (3 g, mp 210–216°C). The filtrate was extracted with diethyl ether, the extract was dried over MgSO₄, and removal of the solvent left a liquid residue which was subjected to vacuum distillation to isolate 1.61 g of a fraction with bp 50–54°C (115 mm). The product was identified as allyl alcohol on the basis of the GLC and ¹H NMR data.

Oligomer VI. a. 2-Chloromethyloxirane (I), 4.6 g (0.05 mol), was added dropwise to a solution of 7.9 g (0.1 mol) of selenium in a mixture of 15 g (0.3 mol) of hydrazine hydrate and 1.84 g (0.03 mol) of 2-aminoethanol, prepared as described above. The mixture was heated for 9 h at 65-70°C, and the orange-red solid was filtered off, washed with water and organic solvents, and dried under reduced pressure. Yield 10.7 g, mp 85–120°C (decomp.). IR spectrum, v, cm⁻¹: 3430, 2920, 2840, 1418, 1395, 1280, 1160, 740, 618, 436. Found, %: C 17.07; H 2.88; Cl 2.13; Se 69.95. C₄₈H₉₆Cl₂O₁₆Se₃₀. Calculated, %: C 17.09; H 2.85; Cl 2.11; Se 70.35. A 3.5-g (0.016-mol) portion of oligomer VI was treated with a mixture of 23.7 g (0.4 mol) of acetone and 1 g (0.009 mol) of o-xylene (standard for GC), and the mixture was left to stand for 1 h. The extract was analyzed by GC-MS, and 4-hydroxy-1,2-diselenolane (V) was identified; yield 5%.

b. 1,3-Dichloropropan-2-ol (III), 12.8 g (0.1 mol), was added to a solution of 7.9 g (0.1 mol) of selenium in a mixture of 15 g (0.3 mol) of hydrazine hydrate and 1.84 g (0.03 mol) of 2-aminoethanol, prepared as

described above. The mixture was stirred for 1.5 h at 70°C, and the orange–red solid was filtered off, washed with water and organic solvents, and dried under reduced pressure. Yield 10.7 g, mp 85–120°C (decomp.). IR spectrum, v, cm⁻¹: 3425, 2910, 2835, 1415, 1390, 1270, 1158, 740, 620, 435. Found, %: C 17.11; H 3.05; Cl 2.07; Se 71.44. C₄₈H₉₆Cl₂O₁₆Se₃₀. Calculated, %: C 17.09; H 2.85; Cl 2.11; Se 70.35. A small amount of oligomer **VI** was treated with CDCl₃, the mixture was kept for 17 h at room temperature, and the solution was analyzed by ¹H NMR to identify 4-hydroxy-1,2-diselenolane (**V**).

1.3-Bis(methylselanyl)propan-2-ol (VIII). Oligomer VI, 3 g (0.014 mol), prepared as described above in a, was dissolved at 70°C in a mixture of 17 g (0.034 mol) of hydrazine hydrate and 3.9 g (0.07 mol) of potassium hydroxide (2 h). The mixture was cooled, 3.9 g (0.028 mol) of methyl iodide was slowly added, the mixture was extracted with diethyl ether, and the extract was dried over CaCl₂ and evaporated. According to the GLC and GC-MS data, the residue, 1.76 g, was almost pure 1,3-bis(methylselanyl)propan-2-ol (VIII). ¹H NMR spectrum, δ , ppm: 2.03 s (CH₃Se), 2.74 d.d (CH₂Se), 2.83 br.s (OH), 3.85 m (CH). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 5.02 (CH₃, ¹ $J_{\rm Se,C}$ = 61.92 Hz), 32.61 (CH₂Se, ${}^{1}J_{Se,C} = 64.87$ Hz), 69.22 (CH). Mass spectrum, m/z for ⁸⁰Se (I, % of the total ion current): 248 (6) $[M]^+$, 233 (17) $[M - CH_3]^+$, 175 (9) [CH₃SeSe]⁺, 139 (5) [CH₃SeCH₂CHON]⁺, 137 (5) $[CH_3SeCH_2SO]^+$, 109 (17) $[CH_3SeCH_2]^+$, 95 (9) $[CH_3Se]^+$, 93 (7) $[CHSe]^+$, 80 (3) $[Se]^+$, 58 (4) $[CH_2CHONCH_2]^+$, 57 (4) $[CH_2SONCH_2]^+$, 43 (6) $[CH_2CHO]^+$. Found, %: C 24.57; H 4.80; Se 64.38. C₅H₁₂Se₂O. Calculated, %: C 24.39; H 4.88; Se 64.23. Reductive cleavage and methylation of oligomers **VI** obtained in the other systems were carried out in a similar way.

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