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# Regioselective syntheses of bis-(2-haloalkyl) selenides and dihalo[bis-(2-haloalkyl)]-λ⁴-selanes from selenium dihalides and 1-alkenes, and the methoxyselenenylation reaction

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**Dedicated to Prof. Oleg A. Rakitin on the occasion of his 65th birthday**

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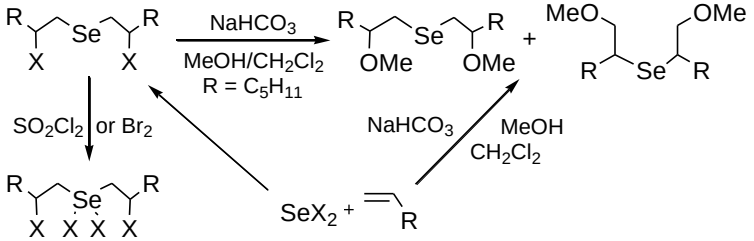
**Abstract**

Regioselective syntheses of bis-(2-haloalkyl) selenides in excellent yields were developed based on selenium dihalides and terminal alkenes (1-hexene, 1-heptene and 1-octene). The addition of selenium dichloride and dibromide to the alkenes occurred *via* the intermediate formation of kinetic products, anti-Markovnikov bis-(1-haloalk-2-yl) selenides, which were further transformed into thermodynamically stable Markovnikov products presumably via seleniranium intermediates. Preparations of dihalo[bis-(2-haloalkyl)]-λ⁴-selanes in 95-99% yields were accomplished by halogenation of bis-(2-haloalkyl) selenides. The system MeOH / $NaHCO_3$ / $CH_2Cl_2$ was developed for methoxyselenenylation of the alkenes using selenium dibromide leading to bis-(2-methoxyalkyl) and bis-(1-methoxyalk-2-yl) selenides in 68-80% total yields.




**Keywords:** Alkenes, selenium dichloride, selenium dibromide, methoxyselenenylation, regioselective addition, selanes

## Introduction

Selenium-containing reagents and organoselenium compounds play important roles in modern organic synthesis.[1-6] Further: organoselenium compounds exhibit various biological activities, including antitumor, antibacterial, antifungal, anti-inflammatory and anti-HIV actions.[7-20] A number of organoselenium compounds containing functional groups (e.g., the methoxy group) demonstrate high glutathione peroxidase-like activity (Figure 1).[14-17] It has recently been shown that the position of the methoxy group in a molecule of organoselenium compound is important for the glutathione peroxidase-like activity.[17]

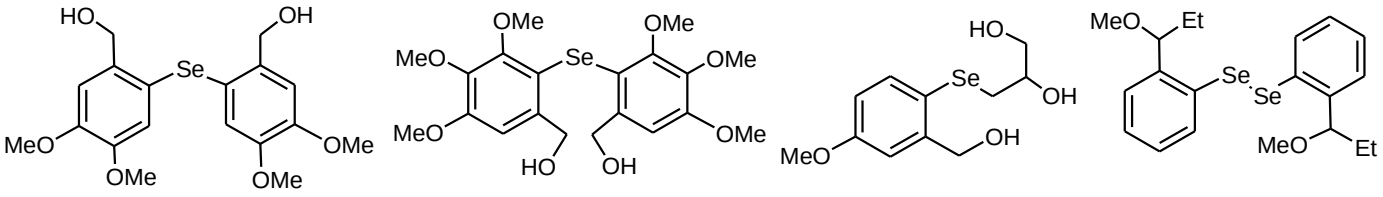
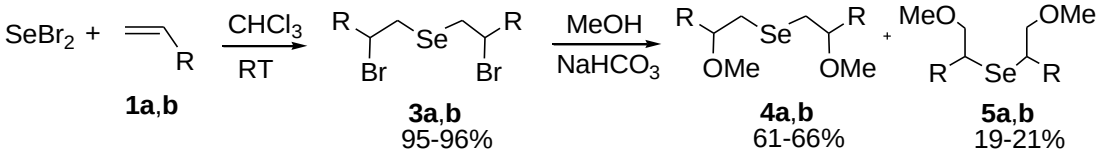



**Figure 1**. Organoselenium compounds containing the methoxy group exhibiting high glutathione peroxidase-like activity.

Regio- and stereo-selective introduction of the selenium atom into organic molecules is an important task for organic chemists. Recently it has been demonstrated that selenium dichloride and dibromide, generated *in situ* from elemental selenium and halogenating agents, can be used successfully for the selective introduction of selenium into organic molecules.[21-36]

The reactions of selenium dichloride and dibromide with alkenes usually afforded Markovnikov addition products.[21] *E.g.*, the addition of selenium dihalides to vinylic ethers gives bis-(2-halo-2-organyloxyethyl) selenides in high yields.[22,23] The formation of anti-Markovnikov addition products was detected in some reactions of selenium dihalides.[23-27] The products of anti-Markovnikov addition were found to be the only products or the major ones in the reactions with alkenes containing electron-withdrawing substituents.[25-27] The addition of selenium dichloride and dibromide to double bonds was studied in the reactions with divinyl sulfide,[28] divinyl selenide,[29] divinyl sulfone,[30,31] 1,5-cyclooctadiene,[32,33] diallyl and divinyl tellurides,[34,35] and a series of novel heterocyclic compounds was thereby synthesized.[28-33]

We already reported on the addition of selenium dibromide with 1-hexene (**1a**) and 1-octene (**1b**) to give bis-(2-bromoalkyl) selenides **3a,b** which in turn could be transformed to bis-(2-methoxyalkyl) selenides **4a**,**b** and bis-(1-methoxyalk-2-yl) selenides **5a**,**b**. However, the latter compounds were not isolated in a pure form (Scheme 1).[24]




**Scheme 1.** The previous synthesis and methanolysis of selenides **3a**,**b.** [24]

In the present paper we want to present a comprehensive study of the addition of selenium dichloride and dibromide to terminal alkenes **1a-c** including methoxyselenenylation reaction.

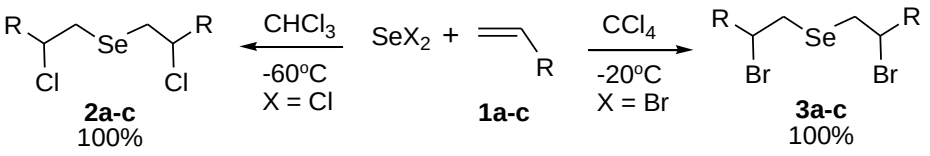
 

Methoxyselenenylation reactions have many useful applications in modern organic synthesis allowing simultaneous introduction of the selenium atom and methoxy group into double bonds.[2] Seleniranium cations are regarded as intermediates in these reactions.[2]

Recently we studied the methoxy- and ethoxy-selenenylation of selenium dihalides with styrene and its derivatives.[36] The reaction proceeded regioselectively, affording bis-(2-alkoxy-2-phenylethyl) selenides in high yields.

## Results and Discussion

Efficient synthesis of bis-(2-chloroalkyl) selenides **2a-c** and bis-(2-bromoalkyl) selenides **3a-c** in quantitative yields by chemo- and regioselective addition of selenium dichloride and dibromide to terminal alkenes **1a-c** was developed (Scheme 2).




X = Cl (**2**), Br (**3**); R = $C_4H_9$ (**a**), $C_6H_{13}$ (**b**), $C_5H_{11}$ (**c**)

**Scheme 2.** The addition reaction of selenium dihalides to 1-alkenes.

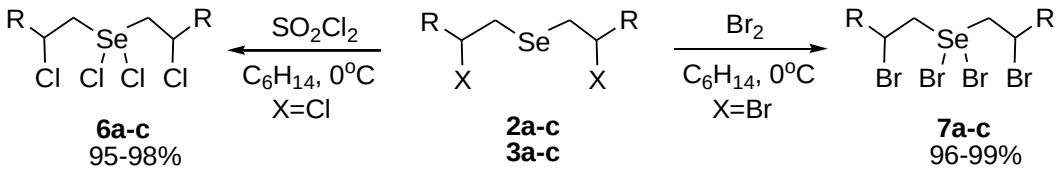
We found that addition of selenium dichloride to alkenes **1a-c** proceeds efficiently in chloroform at -60 °C, while favorable solvent for the reactions of selenium dibromide is carbon tetrachloride (at -20 °C). It is necessary to mix the reagents and to carry out the reactions at low temperature (at -60 °C in chloroform and at -20 °C in carbon tetrachloride). When the reactions were carried out at room temperature, halogenation of the double bond with the formation of 1,2-dihaloalkanes and precipitation of elemental selenium were also observed.

It is necessary to carry out the reactions by addition of selenium dichloride or dibromide to a solution of alkenes **1a-c** at low temperature (a 1 : 2 ratio molar ratio of selenium dihalide and alkene). With inverse addition, *i.e.* when a solution of alkene was added to a solution of selenium dihalide, the formation of compounds with 4-valent selenium, dihalo[bis-(2-haloalkyl)]-$\lambda^4$-selanes **6a-c** and **7a-c**, as by-products in up to 15% yield were observed. In these cases the selenium dichloride and dibromide, present in excess, behave as halogenating agents, converting the selenides **2a-c** and **3a-c** into the corresponding selanes **6a-c** and **7a-c** (Scheme 3).

Efficient synthesis of selanes **6a-c** and **7a-c** in near quantitative yields (95-99%) was developed based on the reaction of selenides **2a-c** and **3a-c** with sulfuryl chloride or bromine in hexane at 0 °C (Scheme 3). Selanes **6a-c** and **7a-c** precipitated from the reaction mixture under these conditions and can easily be isolated.

Synthesis of compound **6a** by addition of $SeCl_4$ to 1-hexene was previously reported in old works[37,38] without NMR data.

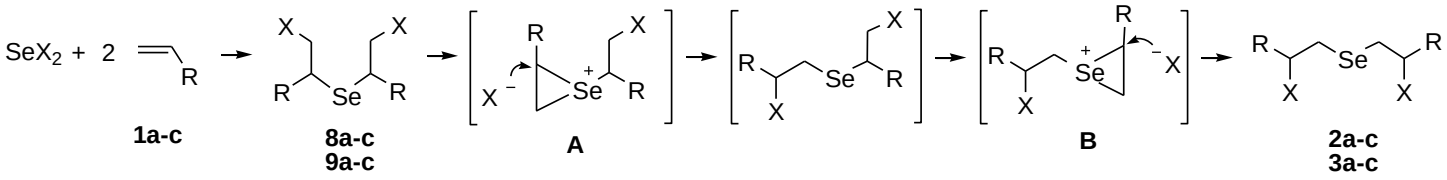
 




X = Cl (**2**,**6**), Br (**3**,**7**); R = $C_4H_9$ (**a**), $C_6H_{13}$ (**b**), $C_5H_{11}$ (**c**)

**Scheme 3.** Synthesis of selanes **6a-c** and **7a-c**.

We found that addition of selenium dihalides to alkenes **1a-c** initially led to anti-Markovnikov products, bis-(1-haloalk-2-yl) selenides **8a-c** and **9a-c** (Scheme 4), which were detected in the reaction mixture by $^1$H and $^{13}$C NMR in $CCl_4$ (the content of selenides **8a-c** or **9a-c** in the mixture with corresponding **2a-c** or **3a-c** was about 65-90%). Compounds **8a-c** and **9a-c** are kinetic products which could not be isolated in pure form since they undergo rearrangement to thermodynamic Markovnikov products **2a-c** and **3a-c** at room temperature.




X = Cl (**2**, **8**), Br (**3**, **9**); R = $C_4H_9$ (**a**), $C_6H_{13}$ (**b**), $C_5H_{11}$ (**c**)

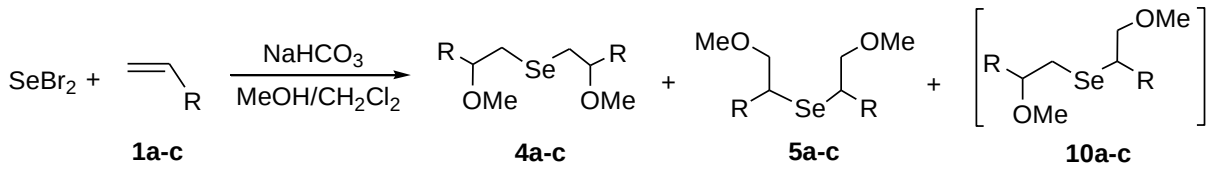
**Scheme 4.** The rearrangement of selenides **8a-c** and **9a-c**.

The content of compounds **8a-c** or **9a-c** predominated over the corresponding **2a-c** or **3a-c** at the beginning of the reactions (carbon tetrachloride, -20 °C, 1-2 h). However, if the reactions were carried out at room temperature, the Markovnikov products **2a-c** and **3a-c** predominated. For example, the ratio of compounds **2c**/**8c** was about 3:2 when the reaction was carried out for 1 h in carbon tetrachloride at room temperature (NMR data).

The rearrangement is suggested to proceed via seleniranium intermediates **A** and **B**. This reaction went slowly in low-polarity solvents (carbon tetrachloride, hexane) and faster in more polar solvents such as chloroform, methylene chloride or acetonitrile. It was found that the rearrangement proceeded faster with bromo derivatives than with the chlorine-containing analogs. On heating the rate of the rearrangement increased.

We found that methoxyselenenylation of alkenes **1a-c** with selenium dibromide in the system MeOH / $NaHCO_3$ / $CH_2Cl_2$ (or $CHCl_3$) led to a mixture of Markovnikov-type bis-(2-methoxyalkyl) selenides **4a-c** and anti-Markovnikov bis-(1-methoxyalk-2-yl) selenides **5a-c** (Scheme 5). The compounds **4a-c** were the major products in all cases (a ratio of **4a-c/5a-c** was about 3 : 1-2). The total yields of compounds **4a-c** and **5a-c** after purification by column chromatography were 68-80%. In contrast to the results of our previous work,[24] compounds **5a-c** were isolated in a pure form by column chromatography. Some unidentified by-products, presumably unsymmetrical (2-methoxyalkyl)-(1-methoxyalk-2-yl) selenides (**10a-c**), were also formed in the reaction.

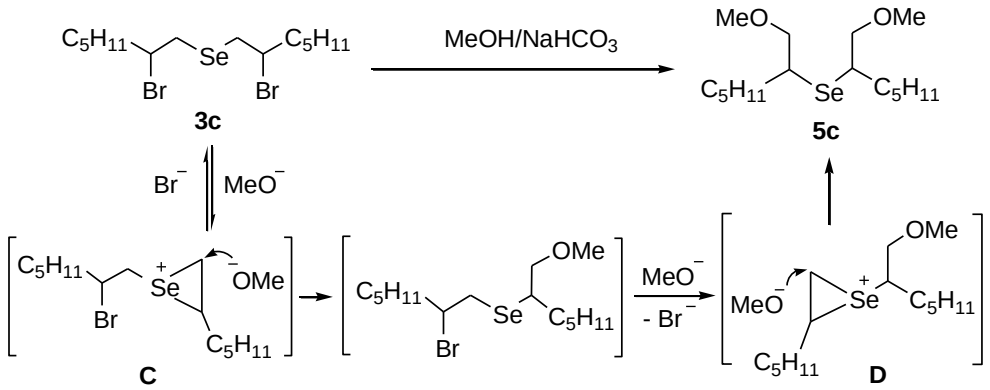



**1a-c** **4a-c** **5a-c** **10a-c**

R = $C_4H_9$ (**a**), $C_6H_{13}$ (**b**), $C_5H_{11}$ (**c**)

**Scheme 5.** Methoxyselenenylation of alkenes **1a-c**.

The methoxy derivatives can be also obtained by nucleophilic substitution of the halide atom by methanol. The methanolysis of selenide **3c** in MeOH/$NaHCO_3$/$CH_2Cl_2$ was studied. It was found that the methanolysis of **3c** led to the formation of the same products **4c** and **5c** in the same ratio (3 : 2) as in the methoxyselenenylation reaction. This fact indicates that the methoxyselenenylation reactions as well as the methanolysis proceeded via the same intermediates, seleniranium cations. The formation of anti-Markovnikov methanolysis product **5c** from Markovnikov adducts **3b** can be rationalized via assuming the generation of seleniranium intermediates **C** and **D** (Scheme 6).




**Scheme 6**. The pathway of the formation of anti-Markovnikov methanolysis product **5c** from Markovnikov adduct **3c**.

Structural assignment of the synthesized compounds was made by $^1H$ and $^{13}C$ NMR spectra and confirmed by analytical data. The values of the $^{13}C$-$^{77}Se$ coupling constants (65-70 Hz) corresponding to the direct coupling ($^1J_{C\text{-}Se}$) was observed for the $CH_2$-group, indicating that the selenium atom added to the terminal carbon of alkenes **1a-c**. The products are approximately equimolar mixtures of two diastereomers (*d,l*- and *meso*-forms, *SR*/*RS* and *RR*/*SS*), which exhibit different signals of the $SeCH_2$, SeCH, $CH_2X$, CHX (X = Cl, Br, OMe) groups in NMR spectra (in some cases the signals of two diastereomers coincided).

$^{77}Se$ NMR spectra were recorded for selenides **2b**,**c** and **3b**,**c** and selanes **6b**,**c** and **7b**,**c**. Two closely located signals corresponding to two diastereomers were observed for all these compounds. Selanes **6b**,**c** containing the Se-Cl bonds exhibit signals at lower field (526.3, 528.3 and 523.1, 525.2 ppm) compared to bromo-containing selanes **7b**,**c** (497.1, 498.8 and 495.6, 497.7 ppm) according to the electronegativity of these halogens (Cl > Br). On the contrary, chloro-containing selenides **2b**,**c** show signals at higher field (167.6, 168.8 and 163.7, 164.8 ppm) compared to bromo-containing selenides **3b**,**c** (202.0, 203.7 and 199.4, 201.1 ppm).

## Conclusions

The addition of selenium dihalides to terminal alkenes occurred in a regioselective mode affording selenides **2a-c** and **3a-c** in quantitative yield. The reaction proceeded via initially formed kinetic anti-Markovnikov products **8a-c** and **9a-c**, which underwent rearrangement to thermodynamically stable Markovnikov products **2a-c** and **3a-c**. The rearrangement was supposed to proceed via seleniranium intermediates. Efficient synthesis of selanes **6a-c** and **7a-c** in 95-99% yield was accomplished by halogenation of selenides **2a-c** and **3a-c**. The methoxyselenenylation reaction of alkenes with selenium dibromide was carried out in the system $MeOH/NaHCO_3/CH_2Cl_2$ leading to bis-(2-methoxyalkyl) **4a-c** and bis-(1-methoxyalk-2-yl) selenides **5a-c** in 68-80% total yields. The methanolysis of bis-(2-bromoheptyl) selenide leads to the formation of bis-(2-methoxyheptyl) selenide **4c** and bis-(1-methoxyhept-2-yl) selenide **5c** in the same ratio (3 : 2) as this was observed in the methoxyselenenylation reaction, thus indicating that both methoxyselenenylation and methanolysis reactions proceed through the same seleniranium intermediates. The synthesized compounds are valuable starting material for preparation of novel organoselenium compounds and intermediates for organic synthesis. Potential biological activity (e.g., glutathione peroxidase-like activity[14-17]) can be supposed for the methoxy derivatives **4a-c** and **5a-c**.

## Experimental Section

**General.** NMR spectra were recorded on a Bruker DPX-400 instrument. $^1$H NMR spectra were acquired at operating frequencies 400.13 MHz and chemical shifts were recorded relative to $SiMe_4$ (δ 0.00) or solvent resonance ($CDCl_3$ δ 7.26). $^{13}$C NMR spectra were acquired at 100.61 Hz and chemical shifts were recorded relative to solvent resonance ($CDCl_3$ δ 77.23 or $CCl_4$ δ 96.70). $^{77}$Se NMR spectra were obtained at 76.3 MHz and chemical shifts were recorded relative to $Me_2Se$ (δ 0.00).
Elemental analysis of carbon and hydrogen was performed on the THERMO Flash EA1112 analyzer. Analytical determination of chlorine, bromine and selenium was made by known methods.[39] Dried and freshly distilled solvents were used in the reactions.

**Bis-(2-chlorohexyl) selenide** (**2a**)**.** A solution of sulfuryl chloride (0.34 g, 2.5 mmol) in chloroform (1 mL) was added dropwise to a mixture of powdered selenium (0.197 g, 2.5 mmol) and chloroform (1 mL). The reaction mixture was stirred at room temperature till complete dissolution of selenium. The obtained solution of selenium dichloride (2.5 mmol) in chloroform (20 mL) was added dropwise to a cooled (-60 °C) solution of hexene-1 (0.42 g, 5 mmol) in chloroform (30 mL). The mixture was stirred at -60 °C for 2 h and 16 h (overnight) at room temperature. The solvent was removed on a rotary evaporator, and the residue was dried under reduced pressure to give the product as pale yellow oil. Yield: 0.796 g (quantitative). $^1$H NMR ($CDCl_3$, *δ*), 0.95 (t, 6H, $CH_3$), 1.30-1.41 (m, 4H, $CH_2$), 1.48-1.59 (m, 4H, $CH_2$), 1.58-1.67 (m, 2H, $CH_2$), 1.91-1.99 (m, 2H, $CH_2$), 2.90-2.99 (m, 4H, $CH_2Se$), 3.94-4.03 (m, 2H, CHCl). $^{13}$C NMR ($CDCl_3$, *δ*), 13.91 ($CH_3$), 21.85 ($CH_2$), 29.06 ($CH_2$), 33.18 ($CH_2Se$, $^1J_{CSe}$ 68 Hz), 33.34 ($CH_2Se$, $^1J_{CSe}$ 68 Hz), 37.01 ($CH_2$), 61.72 (CHCl), 61.81 (CHCl). Anal. Calcd for $C_{12}H_{24}Cl_2Se$: C, 45.30; H, 7.60; Cl, 22.28; Se, 24.82. Found: C, 45.58; H, 7.76; Cl, 21.98; Se, 25.13 %.
**Bis-(2-chlorooctyl) selenide** (**2b**). Yield: (quantitative), pale yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 0.77 (t, 6H, $CH_3$), 1.12-1.26 (m, 14H, $CH_2$), 1.34-1.43 (m, 2H, $CH_2$), 1.45-1.54 (m, 2H, $CH_2$), 1.72-1.80 (m, 2H, $CH_2$), 2.77-2.84 (m, 2H, $CH_2Se$), 2.87-2.93 (m, 2H, $CH_2Se$), 3.87-3.94 (m, 2H, CHCl). $^{13}$C NMR ($CDCl_3$, *δ*), 14.13 ($CH_3$), 22.54 ($CH_2$), 26.27 ($CH_2$), 28.59 ($CH_2$), 31.58 ($CH_2$), 33.38 ($CH_2Se$, $^1J_{CSe}$ = 66 Hz), 33.52 ($CH_2Se$, $^1J_{CSe}$ = 66 Hz), 37.37 ($CH_2$), 61.78, 61.87

(CHCl). $^{77}$Se NMR ($CDCl_3$, *δ*), 167.6, 168.8. Anal. Calcd for $C_{16}H_{32}Cl_2Se$: C, 51.34; H, 8.62; Cl, 18.94; Se, 21.10. Found: C, 51.61; H, 8.82; Cl, 19.23; Se, 20.83 %.

**Bis-(2-chloroheptyl) selenide** (**2c**). Yield: (quantitative), pale yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 0.92 (t, 6H, $CH_3$), 1.29-1.42 (m, 10H, $CH_2$), 1.50-1.60 (m, 2H, $CH_2$), 1.61-1.70 (m, 2H, $CH_2$), 1.89-1.99 (m, 2H, $CH_2$), 2.87-2.9 (m, 2H, $CH_2Se$), 2.98-3.07 (m, 2H, $CH_2Se$), 3.94-4.03 (m, 2H, CHCl). $^{13}$C NMR ($CDCl_3$, *δ*), 13.99 ($CH_3$), 22.41 ($CH_2$), 25.82 ($CH_2$), 31.13 ($CH_2$), 33.25 ($CH_2Se$, $^1J_{CSe}$ 69 Hz), 33.40 ($CH_2Se$, $^1J_{CSe}$ 69 Hz), 37.13 ($CH_2$), 61.75 (CHCl), 61.84 (CHCl). $^{77}$Se NMR ($CDCl_3$, *δ*), 163.7, 164.8. Anal. Calcd for $C_{14}H_{28}Cl_2Se$: C, 48.56; H, 8.15; Cl, 20.48; Se, 22.81. Found: C, 48.28; H, 7.97; Cl, 20.35; Se, 23.14 %.

**Bis-(2-bromohexyl) selenide** (**3a**)**.** A solution of bromine (0.4 g, 2.5 mmol) in $CCl_4$ (10 mL) was added dropwise to a mixture of powdered selenium (0.197 g, 2.5 mmol) and $CCl_4$ (10 mL). The reaction mixture was stirred at room temperature till complete dissolution of selenium. The obtained solution of selenium dibromide (2.5 mmol) in $CCl_4$ (20 mL) was added dropwise to a cooled (-20$^o$C) solution of 1-hexene (0.42 g, 5 mmol) in $CCl_4$ (30 mL). The mixture was stirred at -20 $^o$C for 2 h and 16 h (overnight) at room temperature. The solvent was removed on a rotary evaporator, and the residue was dried under reduced pressure to give the product as pale yellow oil. Yield: 1.018 g (quantitative), pale yellow oil. The $^1$H and $^{13}$C NMR spectra were the same as already described.[24]

**Bis-(2-bromooctyl) selenide** (**3b**)**.** Yield: quantitative, pale yellow oil. $^{77}$Se NMR ($CDCl_3$, *δ*), 202.0, 203.7. The $^1$H and $^{13}$C NMR spectra were the same as already described.[24]

**Bis-(2-bromoheptyl) selenide** (**3c**). Yield: quantitative, pale yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 0.92 (t, 6H, $CH_3$), 1.35 (m, 10H, $CH_2$), 1.58 (m, 2H, $CH_2$), 1.75 (m, 2H, $CH_2$), 2.01 (m, 2H, $CH_2$), 3.06 (m, 2H, $CH_2Se$), 3.21 (m, 2H, $CH_2Se$), 4.09 (m, 2H, CHBr). $^{13}$C NMR ($CDCl_3$, *δ*), 13.89 ($CH_3$), 22.29 ($CH_2$), 26.76 ($CH_2$), 30.89 ($CH_2$), 33.68 ($CH_2Se$, $^1J_{CSe}$ 70 Hz), 33.90 ($CH_2Se$, $^1J_{CSe}$ 70 Hz), 37.30 ($CH_2$), 54.30 (CHBr), 54.48 (CHBr). $^{77}$Se NMR ($CDCl_3$, *δ*), 199.4, 201.1. Anal. Calcd for $C_{14}H_{28}Br_2Se$: C, 38.64; H, 6.49; Se, 18.15; Br, 36.73. Found: C, 38.92; H, 6.68; Se, 17.87; Br, 37.02 %.

**Methoxyselenenylation.** A solution of selenium dibromide (2.5 mmol) in $CH_2Cl_2$ (10 mL) was added dropwise to a cooled (0-5 $^o$C) mixture of 1-heptene (0.49 g, 5 mmol), $NaHCO_3$ (0.42 g, 5 mmol), $CH_2Cl_2$ (15 mL) and methanol (5 mL). The mixture was stirred for 16 h (overnight) at room temperature, washed with water, dried over $Na_2SO_4$, and the solvent evaporated. The residue was subjected to column chromatography on silica gel (eluent: hexane/$CCl_4$ 7 : 1) to give products **4c** (0.447 g, 53% yield) and **5c** (0.228 g, 27% yield).

**Methanolysis.** Methanol (10 mL) and $NaHCO_3$ (0.42 g, 5 mmol) was added to a solution of selenide **3c** (1.088 g, 2.5 mmol) in $CH_2Cl_2$ (20 mL). The mixture was stirred for 16 h (overnight) at room temperature, washed with water, dried over $Na_2SO_4$, and the solvent evaporated. The residue was subjected to column chromatography on silica gel (eluent: hexane/$CCl_4$ 7 : 1) to give products **4c** (0.464 g, 55% yield) and **5c** (0.236 g, 28% yield).

**Bis-(2-methoxyhexyl) selenide** (**4a**). Yield: 51% (methoxyselenenylation), light yellow oil. The $^1$H and $^{13}$C NMR spectra were the same as already described.[24]

**Bis-(2-methoxyoctyl) selenide** (**4b**)**.** Yield: 51% (methoxyselenenylation), light yellow oil. The $^1$H and $^{13}$C NMR spectra were the same as already described.[24]

**Bis-(2-methoxyheptyl) selenide** (**4c**)**.** Yield: 53% (methoxyselenenylation), 55% (methanolysis), light yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 0.93 (t, 6H, $CH_3$), 1.24-1.40 (m, 12H, $CH_2$), 1.44-1.56 (m, 4H, $CH_2$), 2.52-2.61 (m, 2H, $CH_2Se$), 2.62-2.70 (m, 2H, $CH_2Se$), 3.25-3.31 (m, 2H, CHO), 3.34 (s, 6H, $CH_3O$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.04 ($CH_3$), 22.61 ($CH_2$), 24.88 ($CH_2$), 28.04, 28.16 ($CH_2Se$), 31.98 ($CH_2$), 33.80 ($CH_2$), 56.42 ($OCH_3$), 81.37 (CHO). Anal. Calcd for $C_{16}H_{34}O_2Se$: C, 56.96; H, 10.16; Se, 23.40. Found, %: C, 57.24; H, 10.35; Se, 23.12 %.

**Bis-(1-methoxyhex-2-yl) selenide** (**5a)**. Yield: 17% (methoxyselenenylation), light yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 0.87 (t, 6H, $CH_3$), 1.22-1.31 (m, 4H, $CH_2$), 1.48-1.54 (m, 4H, $CH_2$), 1.70-1.76 (m, 4H, $CH_2$), 2.92-2.96 (m, 2H, CHSe), 3.30 (s, 6H, $CH_3O$), 3.49-3.56 (m, 4H, $CH_2O$). $^{13}$C NMR ($CDCl_3$, *δ*), 13.81 ($CH_3$), 26.73 ($CH_2$), 29.71 ($CH_2$), 33.41

($CH_2$), 39.89, 40.54 (CHSe), 58.46 ($OCH_3$), 77.26 ($CH_2O$). Anal. Calcd for $C_{14}H_{30}O_2Se$: C, 54.36; H, 9.77; Se, 25.52. Found: C, 54.08; H, 9.61; Se, 25.86 %.

**Bis-(1-methoxyoct-2-yl) selenide** (**5b**)**.** Yield: 26% (methoxyselenenylation), light yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 1.03 (t, 6H, $CH_3$), 1.36-1.48 (m, 12H, $CH_2$), 1.49-1.54 (m, 4H, $CH_2$), 1.56-1.72 (m, 4H, $CH_2$), 2.94-3.00 (m, 2H, CHSe), 3.45 (s, 6H, $CH_3O$), 3.51-3.57 (m, 2H, $CH_2O$), 3.59-3.65 (m, 2H, $CH_2O$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.10 ($CH_3$), 26.61 ($CH_2$), 28.05 ($CH_2$), 29.14 ($CH_2$), 31.69 ($CH_2$), 32.68 ($CH_2$), 40.56, 40.81 ($CH_2Se$), 58.16 ($CH_3O$), 77.30 ($CH_2O$). Anal. Calcd for $C_{18}H_{38}O_2Se$: C, 59.16; H, 10.48; Se, 21.61. Found: C, 59.45; H, 10.28; Se, 21.91 %.

**Bis-(1-methoxyhept-2-yl) selenide** (**5c**)**.** Yield: 27% (methoxyselenenylation), 28% (methanolysis), light yellow oil. $^1$H NMR ($CDCl_3$, *δ*), 1.02 (t, 6H, $CH_3$), 1.26-1.43 (m, 12H, $CH_2$), 1.44-1.52 (m, 4H, $CH_2$), 2.92-3.00 (m, 2H, CHSe), 3.37 (s, 6H, $CH_3O$), 3.45-3.52 (m, 2H, $CH_2O$), 3.55-3.62 (m, 2H, $CH_2O$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.03 ($CH_3$), 22.53 ($CH_2$), 26.75 ($CH_2$), 29.68, ($CH_2$), 32.42 ($CH_2$), 40.50, 40.79 (CHSe), 58.14 ($OCH_3$), 77.44 ($CH_2O$). Anal. Calcd for $C_{16}H_{34}O_2Se$: C, 56.96; H, 10.16; Se, 23.40. Found: C, 56.71; H, 9.89; Se, 23.68 %.

**Dichloro[bis-(2-chlorohexyl)]-λ$^4$-selane** (**6a**)**.** A solution of sulfuryl chloride (0.35 g, 2.6 mmol) in hexane (10 mL) was added to a cooled to 0 °C solution of selenide **2a** (0.795 g, 2.5 mmol) in hexane (15 mL) and the mixture was stirred at 0 °C for 4 h and allowed to warm to room temperature. The precipitate was filtered off and dried in vacuum to give the product as white powder, m.p. 45-47 °C (hexane). Yield: 0.943 g (97 %). $^1$H NMR ($CDCl_3$, *δ*), 0.94 (t, 6H, $CH_3$), 1.48-1.56 (m, 8H, $CH_2$), 2.12-2.20 (m, 2H, $CH_2$), 2.24-2.32 (m, 2H, $CH_2$), 4.12-4.24 (m, 4H, CHSe), 4.64-4.72 м (2H, CHCl).$^{13}$C NMR ($CDCl_3$, *δ*), 14.12 ($CH_3$), 21.92 ($CH_2$), 30.56 ($CH_2$), 37.35 ($CH_2$), 56.76 (CHCl), 68.65 ($CH_2Se$) 68.72 ($CH_2Se$). Anal. Calcd for $C_{12}H_{24}Cl_4Se$: C, 37.04; H, 6.22; Cl, 36.45; Se, 20.29. Found: C, 36.87; H, 6.04; Cl, 36.78; Se, 19.93 %.

**Dichloro[bis-(2-chlorooctyl)]-λ$^4$-selane** (**6b**)**.** Yield: 98 %, white powder, m.p. 64-66 °C (hexane). $^1$H NMR ($CDCl_3$, *δ*), 0.80 (t, 6H, $CH_3$), 1.16-1.27 (m, 12H, $CH_2$), 1.33-1.45 (m, 4H, $CH_2$), 1.46-1.54 (m, 2H, $CH_2$), 1.73-1.84 (m, 2H, $CH_2$), 4.07-4.18 (m, 4H, $CH_2Se$), 4.62-4.70 (m, 2H, CHCl). $^{13}$C NMR ($CDCl_3$, *δ*), 14.24 ($CH_3$), 22.62($CH_2$), 26.37 ($CH_2$), 28.66 ($CH_2$), 31.66 ($CH_2$), 37.69 ($CH_2$), 56.84 (CHCl), 68.78 ($CH_2Se$, $^1J_{CSe}$ 60 Hz), 68.85 ($CH_2Se$, $^1J_{CSe}$ 59 Hz). $^{77}$Se NMR ($CDCl_3$, *δ*), 526.3, 528.3. Anal. Calcd for $C_{16}H_{32}Cl_4Se$: C, 43.17; H, 7.24; Cl, 31.85; Se, 17.74. Found: C, 42.93; H, 7.08; Cl, 32.14; Se, 18.02 %.

**Dichloro[bis-(2-chloroheptyl)]-λ$^4$-selane** (**6c**)**.** Yield: 95%, white powder, m.p. 65-67 °C (hexane). $^1$H NMR (400.1 MHz, $CDCl_3$): *δ* 0.90 (t, 6H, $CH_3$), 1.23-1.40 (m, 8H, $CH_2$), 1.44-1.66 (m, 4H, $CH_2$), 1.81-2.95 (m, 4H, $CH_2$), 4.11-4.31 (m, 4H, $CH_2SeCl_2$), 4.75-4.82 (m, 2H, CHCl). $^{13}$C NMR, δ, 13.97 ($CH_3$), 22.42 ($CH_2$), 26.02 ($CH_2$), 31.02 ($CH_2$), 37.59 ($CH_2$), 56.74 (CHCl), 68.77 ($CH_2Se$), 68.88 ($CH_2Se$). $^{77}$Se NMR ($CDCl_3$, *δ*), 523.1, 525.2. Anal. Calcd for $C_{14}H_{28}Cl_4Se$: C, 40.31; H, 6.77; Cl, 34.00; Se, 18.93. Found: C, 40.59; H, 6.58; Cl, 33.65; Se, 19.27 %.

**Dibromo[bis-(2-bromohexyl)]-λ$^4$-selane** (**7a**)**.** A solution of bromine (0.4 g, 2.5 mmol) in hexane (10 mL) was added to a cooled to -0 °C solution of selenide **3a** (1.02 g, 2.5 mmol) in hexane (15 mL) and the mixture was stirred at -0 °C for 4 h and allowed to warm to room temperature. The precipitate was filtered off and dried in vacuum to give the product as yellow powder, m.p. 61-63 °C (hexane). Yield: 1.39 g (98 %). $^1$H NMR ($CDCl_3$, *δ*), 0.95 (t, 6H, $CH_3$), 1.46-1.54 (m, 8H, $CH_2$), 1.96-2.03 (m, 2H, $CH_2$), 2.06-2.13 (m, 2H, $CH_2$), 4.08-4.20 (m, 4H, CHSe), 4.57-4.66 (m, 2H, CHBr). $^{13}$C NMR ($CDCl_3$, *δ*), 13.91 ($CH_3$), 18.98 ($CH_2$), 30.83 ($CH_2$), 36.89 ($CH_2$), 48.02 (CHBr), 66.72 ($CH_2Se$). Anal. Calcd for $C_{12}H_{24}Br_4Se$: C, 25.42; H, 4.27; Br, 56.38; Se, 13.93. Found: C, 25.71; H, 4.45; Br, 55.86; Se, 14.34 %.

**Dibromo[bis-(2-bromooctyl)]-λ$^4$-selane** (**7b**)**.** Yield: 95%, yellow powder, m.p. 65-67 °C (hexane). $^1$H NMR ($CDCl_3$, *δ*), 0.79 (m, 6H, $CH_3$), 1.13-1.26 (m, 12H, $CH_2$), 1.38-1.47 (m, 2H, $CH_2$), 1.55-1.64 (m, 2H, $CH_2$), 1.81-1.90 (m, 4H, $CH_2$), 4.11-4.21 (m, 4H, $CH_2Se$), 4.73-4.80 (m, 2H, CHBr). $^{13}$C NMR ($CDCl_3$, *δ*), 14.45 ($CH_3$), 22.84 ($CH_2$), 27.79 ($CH_2$), 28.64 ($CH_2$), 31.83 ($CH_2$), 38.09 ($CH_2$), 47.93 (CHBr), 66.82 ($CH_2Se$). $^{77}$Se NMR ($CDCl_3$, *δ*), 497.1, 498.8.

Anal. Calcd for $C_{16}H_{32}Br_4Se$: C, 30.85; H, 5.18; Br, 51.30; Se, 12.67. Found: C, 31.13; H, 4.97; Br, 50.97; Se, 13.04 %.

**Dibromo[bis-(2-bromoheptyl)]-λ$^4$-selane** (**7c**). Yield: 95%, yellow powder, m.p. 67-69 °C (hexane). $^1$H NMR ($CDCl_3$, *δ*), 0.88 (t, 6H, $CH_3$), 1.32-1.42 (m, 8H, $CH_2$), 1.43-1.53 (m, 2H, $CH_2$), 1.57-1.67 (m, 2H, $CH_2$), 1.86-1.98 (m, 4H, $CH_2$), 4.24-4.39 (m, 4H, $CH_2SeBr_2$), 4.85-4.96 (m, 2H, CHBr). $^{13}$C NMR ($CDCl_3$, *δ*), 14.07 ($CH_3$), 22.45 ($CH_2$), 27.30 ($CH_2$), 30.88 ($CH_2$), 38.05 ($CH_2$), 48.52 (CHBr), 66.31 ($CH_2Se$), 66.35 ($CH_2Se$). $^{77}$Se NMR ($CDCl_3$, *δ*), 495.6, 497.7. Anal. Calcd for $C_{14}H_{28}Br_4Se$: C, 28.26; H, 4.74; Br, 53.72; Se, 13.27. Found: C, 28.54; H, 4.92; Br, 54.11; Se, 12.97 %.

**Bis-(1-chloroalk-2-yl) selenide (8a-c).** A solution of selenium dichloride (1 mmol) in $CCl_4$ (5 mL) was added dropwise to a cooled (-20 °C) solution of hexene-1 (0.168 g, 2 mmol) in $CCl_4$ (5 mL). The mixture was stirred at -20°C for 1 h and studied by NMR $^1$H and $^{13}$C in order to detect selenides **8a-c**. The content of selenides **8a-c** in the mixture with corresponding **2a-c** was about 75-90%.

**Bis-(1-chlorohex-2-yl) selenide** (**8a**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.95 (t, 6H, $CH_3$), 1.34-1.39 (m, 8H, $CH_2$), 1.50-1.55 (m, 2H, $CH_2$), 1.93-1.98 (m, 2H, $CH_2$), 2.93-3.00 (m, 2H, CHSe), 3.55-3.60 (m, 2H, $CH_2Cl$), 3.80-3.83 (m, 2H, $CH_2Cl$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.00 ($CH_3$), 22.44 ($CH_2$), 29.22 ($CH_2$), 31.83 ($CH_2$), 43.29, 43.38 (CHSe), 48.34 ($CH_2Cl$).

**Bis-(1-chlorooct-2-yl) selenide** (**8b**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.92 (t, 6H, $CH_3$), 1.25-1.43 (m, 12H, $CH_2$), 1.45-1.49 (m, 2H, $CH_2$), 2.00-2.03 (m, 2H, $CH_2$), 2.96-3.02 (m, 2H, CHSe), 3.55-3.60 (m, 2H, $CH_2Cl$), 3.80-3.84 (m, 2H, $CH_2Cl$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.15 ($CH_3$), 22.57 ($CH_2$), 26.16 ($CH_2$), 28.65 ($CH_2$), 31.57 ($CH_2$), 33.42 ($CH_2$), 43.48, 43.59 (CHSe), 48.62 ($CH_2Cl$).

**Bis-(1-chlorohept-2-yl) selenide** (**8c**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.92 (t, 6H, $CH_3$), 1.25-1.43 (m, 12H, $CH_2$), 1.45-1.49 (m, 2H, $CH_2$), 2.00-2.03 (m, 2H, $CH_2$), 2.96-3.02 (m, 2H, CHSe), 3.55-3.60 (m, 2H, $CH_2Cl$), 3.80-3.84 (m, 2H, $CH_2Cl$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.24 ($CH_3$), 22.67 ($CH_2$), 26.07 ($CH_2$), 28.75 ($CH_2$), 33.92 ($CH_2$), 43.43, 43.52 (CHSe), 48.52 ($CH_2Cl$).

**Bis-(1-bromoalk-2-yl) selenide** (**9a-c**)**.** A solution of selenium dibromide (1 mmol) in $CCl_4$ (5 mL) was added dropwise to a cooled (-20 °C) solution of alkene (2 mmol) in $CCl_4$ (5 mL). The mixture was stirred at -20 °C for 1 h and studied by NMR $^1$H and $^{13}$C in order to detect selenides **9a-c**. The content of selenides **9a-c** in the mixture with corresponding **3a-c** was about 70-80%.

**Bis-(1-bromohex-2-yl) selenide** (**9a**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.92 (t, 6H, $CH_3$), 1.24-1.42 (m, 8H, $CH_2$), 1.50-1.57 (m, 2H, $CH_2$), 1.57-1.67 (m, 2H, $CH_2$), 3.11-3.20 (m, 2H, CHSe), 3.43-3.48 (m, 2H, $CH_2Br$), 3.73-3.78 (m, 2H, $CH_2Br$). $^{13}$C NMR ($CDCl_3$, *δ*), 13.65 ($CH_3$), 22.00 ($CH_2$), 28.81, 28.88 ($CH_2$), 31.75, 32.20 ($CH_2$), 36.84, 36.95 ($CH_2Br$), 42.61, 43.24 (CHSe).

**Bis-(1-bromooct-2-yl) selenide** (**9b**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.86 (t, 6H, $CH_3$), 1.22-1.34 (m, 12H, $CH_2$), 1.36-1.42 (m, 2H, $CH_2$), 1.48-1.56 (m, 2H, $CH_2$), 1.69-1.78 (m, 2H, $CH_2$), 1.95-2.03 (m, 2H, $CH_2$), 3.12-3.16 (m, 2H, CHSe), 3.47-3.55 (m, 2H, $CH_2Br$), 3.75-3.79 (m, 2H, $CH_2Br$). $^{13}$C NMR ($CDCl_3$, *δ*), 14.10 ($CH_3$), 22.59 ($CH_2$), 30.12 ($CH_2$), 28.47 ($CH_2$), 31.61 ($CH_2$), 34.84 ($CH_2$), 38.64, 38.79 ($CH_2Br$), 44.55, 44.83 (CHSe).

**Bis-(1-bromohept-2-yl) selenide** (**9c**)**.** $^1$H NMR ($CCl_4$, *δ*), 0.95 (t, 6H, $CH_3$), 1.25-1.49 (m, 12H, $CH_2$), 1.52-1.65 (m, 2H, $CH_2$), 1.70-1.79 (m, 2H, $CH_2$), 3.06-3.09 (m, 2H, CHSe), 3.47-3.51 (m, 2H, $CH_2Br$), 3.75-3.81 (m, 2H, $CH_2Br$). $^{13}$C NMR ($CDCl_3$, *δ*), 13.94 ($CH_3$), 22.33 ($CH_2$), 26.52 ($CH_2$), 26.79 ($CH_2$), 31.94 ($CH_2$), 36.69, 36.78 ($CH_2Br$), 42.88, 43.26 (CHSe).

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