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Note

The reaction of selenium dichloride with divinyl sulfide

Svetlana V. Amosova *, Maxim V. Penzik, Alexander I. Albanov, Vladimir A. Potapov

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Division of the Russian Academy of Sciences, 664033 Irkutsk, 1 Favorsky Str., Russian Federation

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ABSTRACT

A synthesis of novel selenium heterocycles based on the reaction of selenium dichloride with divinyl sulfide has been described. At −50 °C the reaction affords 2,6-dichloro-1,4-thiaselenane in quantitative yield. At room temperature the reaction gives 2,6-dichloro-1,4-thiaselenane and 5-chloro-2-chloromethyl-1,3-thiaselenolane. Upon standing in chloroform solution, 2,6-dichloro-1,4-thiaselenane undergoes spontaneous rearrangement to 5-chloro-2-chloromethyl-1,3-thiaselenolane. Under the action of pyridine, 2,6-dichloro-1,4-thiaselenane is converted to 2-chloromethyl-1,3-thiaselenole in 95% yield.

1. Introduction

Data on reactions of selenium dichloride are very scarce in the literature. It is known that, in solution, selenium dichloride is in equilibrium with Se_2Cl_2 and $SeCl_4$ [1–4]. Solutions of $SeCl_2$ have been studied by photoelectron, UV–Vis, Raman spectroscopy, and ^{77}Se NMR techniques, and selenium dichloride was shown to be the predominant compound in the equilibrium [1–4].

Selenium dichloride has been obtained by disproportionation of selenium and $SeCl_4$ and used for the preparation of diselenadiazolium chloride, a compound with a N–Se bond [5,6]. A complex of $SeCl_2$ with tetrahydrothiophene has been synthesized and characterized by X-ray analysis [7]. The reaction of selenium dichloride with *t*-butylamine affording compounds with selenium and nitrogen backbones has been described [8,9].

Reactions of both selenium tetrachloride and selenium dichloride with N,N-bis(trimethylsilyl)-2,6-diisopropylaniline led to 4-N,N-bis(trimethylsilyl)amino-3,5-diisopropylphenylselenium trichloride. In a second case, selenium dichloride underwent disproportionation to Se_2Cl_2 and selenium tetrachloride, which was involved in an electrophilic aromatic substitution reaction [10].

The addition of selenium dichloride to dimethyl diethynyl silane giving unsaturated 5-membered heterocycles was the first reported synthesis of an organoselenium compound using $SeCl_2$ [11,12]. This reaction was performed with various diorganyl diethynyl silanes and germanes providing previously unknown 1-selena-4-silafulvenes and 4-germafulvenes [13–18].

After the first publications [11,12], some further reports on reactions of selenium dichloride with organic compounds have appeared in the literature [20,21]. Reactions of selenium dichloride with organolithium and organomagnesium reagents were employed to obtain diaryl selenides [20], while the addition of selenium dichloride to propargylic alcohols provided the corresponding divinyl selenides [21].

Divinyl sulfide is a versatile starting material for the preparation of a variety of organosulfur compounds including heterocycles [22–24]. Continuing our investigations of the addition of new reagents, selenium dichloride and dibromide, to unsaturated compounds with a goal to obtaining new selenium heterocycles [11–19], we have studied the reaction of selenium dichloride with divinyl sulfide.

2. Results and discussion

We found that the reaction of selenium dichloride with divinyl sulfide at −50 °C in chloroform afforded a 6-membered heterocyclic compound, 2,6-dichloro-1,4-thiaselenane (**1**), in virtually quantitative yield (Scheme 1). Thiaselenane **1** consisted of two diastereomers in a 6:1 ratio.

When the reaction of selenium dichloride with divinyl sulfide was performed at room temperature in CCl_4, the only product was thiaselenane **1** (the ratio of diastereomers was 3:1), but a part of divinyl sulfide remained unconverted.

Thiaselenane **1** was stable upon storage for several weeks at −20 °C. At room temperature in chloroform solution, thiaselenane **1** underwent rearrangement to the 5-membered heterocyclic isomer, 5-chloro-2-chloromethyl-1,3-thiaselenolane (**2**) (Scheme 2).

* Corresponding author.
E-mail address: amosova@irioch.irk.ru (S.V. Amosova).

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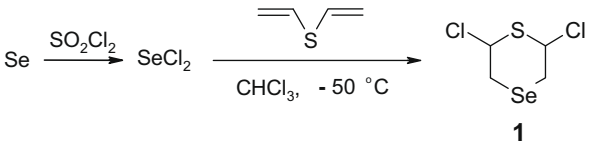
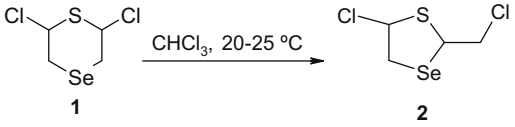
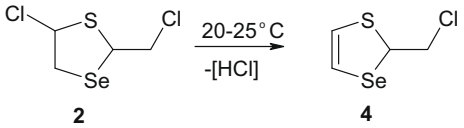


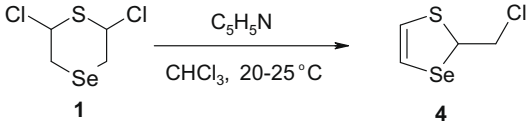
Scheme 1.



Scheme 2.



Scheme 5.



Scheme 6.

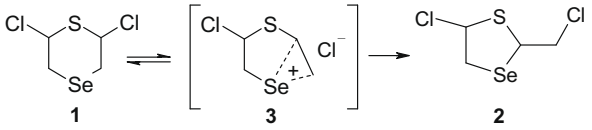
When a chloroform solution of thiaselenane **1** was allowed to stand at room temperature, the appearance of thiaselenolane **2** occurred. This process was monitored by ^{1}H NMR spectroscopy. A diminution of intensity of the signals of thiaselenane **1** with a concomitant increase in the intensity of signals of thiaselenolane **2** was observed in the ^{1}H NMR spectra. Thiaselenolane **2** consisted of two diastereomers in a 2:1 ratio.

The reaction of selenium dichloride with divinyl sulfide at room temperature gave thiaselenane **1** (3:1 ratio of diastereomers) in 72% yield and thiaselenolane **2** (1:1 ratio of diastereomers) in 24% yield.

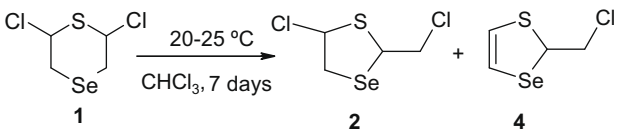
To rationalize these facts we suggest that thiaselenane **1** is the kinetic product and thiaselenolane **2** is the thermodynamic product. The rearrangement of thiaselenane **1** to thiaselenolane **2** was assumed to proceed via intermediate **3** (Scheme 3).

It is known that a halogen atom is strongly activated in 2-haloethyl sulfides and selenides due to the anchimeric assistance effect with participation of sulfur or selenium atoms [25–28]. Nucleophilic substitution of halogen atoms in such compounds proceeds easily with a variety of nucleophiles. The formation of similar 3-membered intermediates has been suggested to explain the considerable increase of the rate of the nucleophilic substitution reactions. In the case of thiaselenane **1**, intermediate **3** can be stabilized not only by the selenium atom but by the sulfur atom as well. The ability of a sulfur atom to stabilize an adjacent positive charge is well known.

The complete conversion of thiaselenane **1** to thiaselenolane **2** occurred when a chloroform solution of the former was allowed to stand at room temperature for 7 days. However, along with thiaselenolane **2** (61% yield), the formation of 2-chloromethyl-1,3-thiaselenole (**4**) (36% yield) was observed in this case (Scheme 4).



Scheme 3.



Scheme 4.

We have found that, upon standing at room temperature in chloroform solution, thiaselenolane **2** underwent slow dehydrochlorination to thiaselenole **4** (Scheme 5). The same reaction occurred faster when the solution was warmed on a water bath. Vacuum distillation of the mixture of compounds **1** and **2**, which was obtained in chloroform at room temperature, led to thiaselenole **4** in 30% yield.

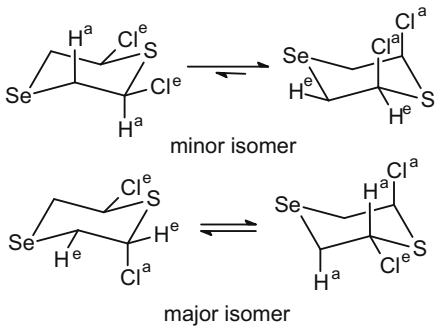
When we added pyridine to a solution of thiaselenane **1** in chloroform in order to perform dehydrochlorination, the only product was thiaselenole **4**. This approach was used to obtain thiaselenole **4** from compound **1** in 95% yield (Scheme 6).

It was found that the addition of pyridine exhibited a catalytic effect on the rearrangement of thiaselenane **1** to thiaselenolane **2**. Thus, after addition of a catalytic amount of pyridine (2% with respect to thiaselenane **1**) to a chloroform solution of thiaselenane **1**, the mixture contained thiaselenolane **2** (42%), thiaselenole **4** (9%) and unconverted thiaselenane **1** (49%) (from the data of the ^{1}H NMR spectrum, which was recorded in 1 h after the addition of pyridine). Therefore, the addition of pyridine caused the rearrangement of thiaselenane **1** to thiaselenolane **2** followed by dehydrochlorination.

The structural assignments of compounds **1**, **2** and **4** were made with ^{1}H, ^{13}C, ^{77}Se NMR and GC–MS.

The protons of the CH_2Se group were not equivalent in the ^{1}H NMR spectra of thiaselenane **1** and each of the protons appeared as a doublet of doublets with geminal coupling constants of $^2J_{HH}$, 12.7 (major diastereomer) and 12.4 Hz (minor diastereomer). The ^{13}C spectra were characterized by signals for sp^3-hybridized carbon atoms at δ 27.98 (CH_2Se) and 59.09 (CHCl) (major diastereomer), 27.04 (CH_2Se) and 58.72 (CHCl) (minor diastereomer). The values of the coupling constants of the selenium atom with the carbon atom of the CH_2 group (67.1 and 65.2 Hz for major and minor diastereomers) indicated that these were the direct coupling constants, $^1J_{Se-C}$, and the selenium atom was adjacent to the CH_2 group. Therefore, this product could not be the 4-membered heterocycle, 2,4-bis(chloromethyl)-1,3-thiaselenetane, which might show similar signals in the NMR spectra but contains the SeCH fragment and therefore would exhibit $^1J_{Se-C}$ for the SeCH fragment rather then for the $SeCH_2$ group.

Thiaselenane **1** consisted of two diastereomers, whose ratio varied from 6:1 (the reaction at −50 °C) to 3:1 (the reaction at room temperature). The ^{1}H NMR spectra of thiaselenane **1** revealed two sets of the signals of the ABX resonance spin system corresponding to two diastereomers. For the minor isomer, the ^{1}H NMR data showed that it was the *cis*-isomer with both chlorine atoms in the axial positions (Scheme 7). The coupling constant $^3J_{HH}$ between the equatorial protons in the minor isomer was 10.5 Hz. Based on the values of the coupling constant between the equatorial protons in the major isomer ($^3J_{HH}$ = 7.7 Hz) we



Scheme 7.

supposed that it was the *trans*-isomer with one chlorine atom in the axial position and another chlorine atom in the equatorial position (Scheme 7). The chair conformations of the major *trans*-isomer were easily inverted and the inversion led to a decrease in the value of the coupling constant ($^3J_{HH}$ = 7.7 Hz). It is known that the values of $^3J_{HH}$ between equatorial protons 6–8 Hz indicate reversible inversion in dihalo-1,4-dichalcogenanes [29]. On the contrary, in the case of the minor *cis*-isomer, the ring inversion did not occur on the NMR timescale and there was a substantial amount of the axial, axial conformer despite 1,3-diaxial repulsion and dipole–dipole repulsion. On the other hand, however, there were favorable anomeric effects [29,30].

Unlike thiaselenane **1**, the structure of thiaselenolane **2** was unsymmetrical and according to the ^{1}H NMR spectra there were no identical protons. Measurements of coupling constants between the selenium atom and the carbon atom of the CH-group (70.5 Hz for major diastereomer and 75.6 Hz for minor diastereomer) in thiaselenolane **2** showed that these were the direct coupling constants, $^1J_{Se-C}$, and that the selenium atom was bonded with the carbon atom of the CH-group. These data were important for the structural assignment of thiaselenolane **2**. Although, the NMR data did not allow unambiguous assignment of the diastereomers, we suppose that the major isomer was thermodynamically more stable and possessed *trans*-stereochemistry and the minor isomer had *cis*-stereochemistry.

In conclusion, we have presented studies of the reaction of selenium dichloride with divinyl sulfide leading to thiaselenane **1**. The synthesis of the novel compounds **1**, **2** and **4** has been described. A previously unknown rearrangement, which proceeded spontaneously at room temperature, has been found. The driving force of the rearrangement is suggested to be an anchimeric assistance effect with participation of the selenium atom. This effect is important for stabilization of the cation **3**, which is assumed to be the intermediate in the rearrangement.

3. Experimental

3.1. General

^{1}H (400.1 MHz), ^{13}C (100.6 MHz) and ^{77}Se (76.3 MHz) NMR spectra were recorded on a Bruker DPX-400 spectrometer in 5–10% solution in CCl_4 or $CDCl_3$, referenced to HMDS (^{1}H and ^{13}C NMR, internal) and Me_2Se (^{77}Se NMR, external). GC–MS spectra were recorded on a Shimadzu QP5050A spectrometer at an electron energy of 70 eV.

3.2. 2,6-Dichloro-1,4-thiaselenane (*1*)

A solution of sulfuryl chloride (1.8 g, 13.3 mmol) in chloroform (15 ml) was added to a mixture of selenium (1.05 g, 13.3 mmol) and chloroform (30 ml) and the resulted mixture was stirred overnight at room temperature. The solution of selenium dichloride thus prepared, and a solution of divinyl sulfide (1.14 g, 13.3 mmol) in chloroform (10 ml) were added separately and simultaneously with stirring over 2.5 h to a flask with chloroform (50 ml) cooled to −50 °C. The mixture was allowed to warm to 10 °C with stirring. The solvent was distilled off *in vacuo* and the residue was analyzed by NMR. The NMR analysis showed that the residue (3.31 g) contained thiaselenane **1** (the purity was about 95%, the yield was near quantitative). The ratio of diastereomers was 6:1. Major diastereomer. ^{1}H NMR (CCl_4, δ): 5.55 (d d, 2H, CHCl, $^3J_{HH}$ = 2.3 Hz, $^3J_{HH}$ = 7.7 Hz), 3.31 (d d, 2H, CH_2SeCH_2, $^3J_{HH}$ = 2.3 Hz, $^2J_{HH}$ = 12.7 Hz), 3.12 (d d, 2H, CH_2SeCH_2, $^3J_{HH}$ = 7.7 Hz, $^2J_{HH}$ = 12.7 Hz). ^{13}C NMR (CCl_4, δ): 27.98 (CH_2Se, $^1J_{Se-C}$ = 67.1 Hz), 59.09 (CHCl). ^{77}Se NMR (CCl_4, δ): 164 ($^2J_{SeH}$ = 16.3 Hz). Minor diastereomer. ^{1}H NMR (CCl_4, δ): 5.26 (d d, 2H, CHCl, $^3J_{HH}$ = 3.1 Hz, $^3J_{HH}$ = 10.5 Hz), 3.20 (d d, 2H, CH_2SeCH_2, $^3J_{HH}$ = 3.1 Hz, $^2J_{HH}$ = 12.4 Hz), 3.12 (d d, 2H, CH_2SeCH_2, $^3J_{HH}$ = 10.5 Hz, $^2J_{HH}$ = 12.4 Hz). ^{13}C NMR (CCl_4, δ): 27.04 (CH_2Se, $^1J_{Se-C}$ = 65.2 Hz), 58.72 (CHCl). GC–MS, *m*/*z* (rel. int.): 236 ($M^{+\cdot}$, 19) 200 (11), 165 (7), 151 (37), 139 (15), 112 (15), 93 (11), 86 (100), 85 (73), 59 (80), 47 (60). Anal. Calc. for $C_4H_6Cl_2SSe$: C, 20.36; H, 2.56; Cl, 30.04; S, 13.59; Se, 33.45. Found: C, 20.78; H, 2.22; Cl, 30.97; S, 14.21; Se, 32.45%.

3.3. The rearrangement of 2,6-dichloro-1,4-thiaselenane (*1*) to 5-chloro-2-chloromethyl-1,3-thiaselenolane (*2*)

The residue containing 95% thiaselenane **1**, which was obtained in experiment 3.2, was dissolved in chloroform and allowed to stand for several days at room temperature. The rearrangement was monitored by ^{1}H NMR spectroscopy. The complete conversion of thiaselenane **1** to thiaselenolane **2** was observed after 7 days. The solvent was distilled off *in vacuo* and the residue was analyzed by NMR. The NMR analysis showed that the residue (3.18 g) contained thiaselenolane **2** (a content of thiaselenolane **2** in the mixture was 60% that corresponded to 61% yield) and thiaselenole **4** (30%, the yield was 36%). The ratio of diastereomers of thiaselenolane **2** was 2 : 1. Major diastereomer of thiaselenolane **2**, ^{1}H NMR (CCl_4, δ): 3.80 (d, 2H, CH_2Se, $^3J_{HH}$ = 3.1 Hz), 3.94 (d d, 1H, CH_2Cl, $^2J_{HH}$ = 10.9 Hz, $^3J_{HH}$ = 8.5 Hz), 4.06 (d d, 1H, CH_2Cl, $^2J_{HH}$ = 10.9 Hz, $^3J_{HH}$ = 7.3 Hz), 4.93 (d d, 1H, SeCHS, $^3J_{HH}$ = 7.3 Hz, $^3J_{HH}$ = 8.5 Hz), 6.11 (t, 1H, SCHCl, $^3J_{HH}$ = 3.1 Hz). ^{13}C NMR (CCl_4, δ): 44.58 (CH_2Se), 50.38 (CH_2Cl), 47.58 (SeCHS, $^1J_{Se-C}$ = 70.5 Hz), 72.60 (SCHCl). ^{77}Se NMR ($CDCl_3$, δ): 389. GC–MS, *m*/*z* (rel. int.): 236 ($M^{+\cdot}$, 28), 200 (24), 187 (22), 174 (9), 165 (8), 151 (71), 139 (14), 121 (34), 112 (22), 93 (18), 86 (67), 85 (77), 58 (100), 45 (49). Minor diastereomer of thiaselenolane **2**, ^{1}H NMR (CCl_4, δ): 3.62 d d (1H, CH_2Cl, ^2J = 11.3 Hz, ^3J = 7.9 Hz), 3.72 (d d, 1H, CH_2Se, ^2J = 11.4 Hz, ^3J = 3.5 Hz), 3.82 d d (1H, CH_2Cl, ^2J = 11.3 Hz, ^3J = 6.9 Hz), 3.88 (m, 1H, CH_2Se), 5.02 (d d, 1H, SeCHS, ^3J = 6.9 Hz, ^3J = 7.9 Hz), 5.98 (t, 1H, SCHCl, ^3J = 3.1 Hz). ^{13}C NMR (CCl_4, δ): 42.60 (CH_2Se); 48.78 (CH_2Cl); 46.23 (SeCHS, $^1J_{Se-C}$ = 75.6 Hz); 71.57 (SCHCl). ^{77}Se NMR ($CDCl_3$, δ): 387. GC–MS, *m*/*z* (rel. int.): 236 ($M^{+\cdot}$, 27), 200 (26), 187 (28), 174 (12), 165 (7), 151 (82), 139 (12), 121 (46), 112 (45), 107 (14), 94 (21), 86 (39), 85 (73), 58 (100), 45 (51).

3.4. The reaction of selenium dichloride with divinyl sulfide at room temperature

A solution of sulfuryl chloride (1.8 g, 13.3 mmol) in chloroform (15 ml) was added to a mixture of selenium (1.05 g, 13.3 mmol) and chloroform (30 ml) and the resulting mixture was stirred overnight at room temperature. The prepared solution of selenium dichloride and a solution of divinyl sulfide (1.14 g, 13.3 mmol) in chloroform (10 ml) were added separately and simultaneously with stirring over 1.5 h to a flask with chloroform (50 ml) at room

temperature. The mixture was stirred at room temperature for 30 min. The solvent was distilled off *in vacuo* and the residue was analyzed by NMR. The NMR analysis showed that the residue (3.23 g) contained thiaselenane **1** (a content of thiaselenane **1** in the mixture was 70% that corresponded to 72% yield, the ratio of diastereomers was 3:1) and thiaselenolane **2** (23%, the yield was 24%, the ratio of diastereomers was 1:1).

3.5. 2-Chloromethyl-1,3-thiaselenole (**4**)

Distillation of the residue, which was obtained in experiment 3.4, led to pure thiaselenole **4** (0.77 g, 30% yield). Bp 64–66 °C (10^{-3} mm Hg). ^{1}H NMR (CCl_4, δ): 6.59 (d, SeCH, $^3J_{HH}$ = 6.3 Hz), 6.38 (d, SCH, $^3J_{HH}$ = 6.3 Hz), 5.00 (t, SeCHS, $^3J_{HH}$ = 7.6 Hz), 3.70 (m, CH_2Cl). ^{13}C NMR ($CDCl_3$, δ): 47.93 (SeCHS, $^1J_{Se-C}$ = 71.9 Hz), 48.42 (CH_2Cl, $^2J_{SeC}$ = 9.5 Hz), 113.63 (=CHS), 119.74 (=CHSe). ^{77}Se NMR ($CDCl_3$, δ): 504 ($^2J_{SeH}$ (SeCH=CH) = 49.8 Hz, $^2J_{SeH}$ (SeCHS) = 23.7 Hz, $^3J_{SeH}$ (SeCH=CH) = 10.4 Hz, $^3J_{SeH}$ ($SeCHCH_2Cl$) = 3.7 Hz). GC–MS, *m*/*z* (rel. int.): 200 (M^+, 45), 166 (38), 119 (100), 94 (29), 83 (48), 47 (60). Anal. Calc. for C_4H_5ClSSe: C, 24.08; H, 2.53; S, 16.07. Found: C, 23.80; H, 2.54; S, 15.99%.

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