

SHORT
COMMUNICATIONS

Synthesis of 2,6-Dichloro-1,4-thiaselenane from Divinyl Sulfide and Selenium Dichloride

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Selenium dichloride in a solution exists in an equilibrium with SeCl_4 and Se_2Cl_2 [1]. Prior to our research [2, 3] no data were published on the possibility of the synthesis of organoselenium compounds from the selenium dichloride. We performed the reactions of SeCl_2 and SeBr_2 with dimethyldiethynylsilane which led to the formation of 3,6-dihalo-4,4-dimethyl-1,4-selenasilafulvenes [2, 3]. This reaction extended further to dialkyldiethynylsilanes and –germanes showed the way to new classes of heterocycles containing in the ring atoms of selenium and the elements of 16 group [4–8].

We previously reported that on adding divinyl sulfide to a solution of SeCl_2 in chloroform at room temperature a five-membered heterocycle was formed, 2-chloromethyl-1,3-thiaselenol [9].

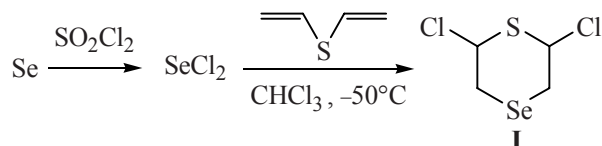
At -50°C the simultaneous uniform addition into a reactor of the solutions of the mentioned reagents in chloroform (at equimolar ratio of selenium dichloride and divinyl sulfide) resulted in the formation in 90% yield of the previously unknown six-membered heterocycle, 2,6-dichloro-1,4-thiaselenane (**I**).

2,6-Dichloro-1,4-thiaselenane (I). Major isomer. ^1H NMR spectrum, δ , ppm: 5.55 d.d (2H, CHCl , 3J 2.3, 3J 7.7 Hz), 3.32 d.d (2H, CH_2Se , 3J 2.3, 2J 12.7 Hz), 3.12 d.d (2H, CH_2Se , 3J 7.7, 2J 12.7 Hz). ^{13}C NMR spectrum, δ , ppm: 27.98 (CH_2Se , $^1J_{\text{Se-C}}$ 67.1 Hz), 59.09 (CHCl). ^{77}Se NMR spectrum, δ , ppm: 164. Minor isomer. ^1H NMR spectrum, δ , ppm: 5.26 d.d (2H, CHCl , 3J 3.1, 3J 10.5 Hz), 3.20 d.d (2H, CH_2Se , 3J 3.1, 2J 12.4 Hz), 3.12 d.d (2H, CH_2Se , 3J 10.5, 2J 12.4 Hz). ^{13}C NMR spectrum, δ , ppm: 27.04 (CH_2Se , $^1J_{\text{Se-C}}$ 65.2 Hz), 58.72 (CHCl). Mass spectrum, m/z (I_{rel} , %): 236 [M]⁺ (19), 200 (11), 165 (7), 151 (37), 139 (15), 112 (15), 93 (11), 86 (100), 85 (73), 59 (80), 47 (60). Found, %: C 20.78; H 2.42; Cl 30.47. $\text{C}_4\text{H}_6\text{Cl}_2\text{SSe}$. Calculated, %: C 20.36; H 2.56; Cl 30.04. M 236.02.

NMR spectra were registered on a spectrometer Bruker DPX-400 at operating frequencies 400.13 (^1H , HMDS), 100.61 (^{13}C , HMDS), and 76.30 MHz (^{77}Se , Ph_2Se_2).

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Compound **I** is a dark fluid decomposed on distillation. It is a mixture of diastereomers in a ratio 6 : 1. The composition and structure of the substance were proved by ^1H , ^{13}C , ^{77}Se NMR, mass spectra, and elemental analysis.

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