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LETTERS TO THE EDITOR

Expedient Procedure for Preparation of 2-Chloromethyl-1,3-diselenol

V. A. Potapov, K. A. Volkova, M. V. Penzik, and S. V. Amosova

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

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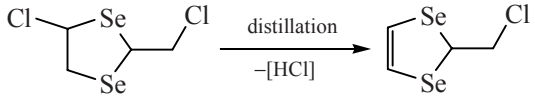
We are engaged in systematic study of reactions of selenium dichloride and dibromide with divinylchalcogenides [1–5]. Earlier, we have reported on synthesis of 4-chloro-2-chloromethyl-1,3-diselenolane (**I**) by the reaction of addition of selenium dichloride to divinylselenide [4]. Diselenolane **I** was purified by chromatography on silica and represents a mixture of diastereomers in the ratio of 4 : 1.

We have found that distillation of diselenolane **I** in vacuum (1 mm Hg) results in its dehydrochlorination and formation of new unsaturated heterocycle, 2-chloromethyl-1,3-diselenol **(II)**. Dehydrochlorination of diselenolane **I** proceeds with high selectivity with elimination of the chlorine atom only from the 4 position. No dehydrochlorination with the chlorine elimination from the chloromethyl group occurs under these conditions.



I **II**

It should be noted that upon distillation the individual diselenol **II** is distilled out, it does not contain diselenolane **I** or other impurities and does not require additional purification. The yield of diselenol **II** is 42% (non-optimized). After distillation some tarry stillage residue is remained.

Diselenol **II** was not described in the literature until now. It is of interest as potential reagent for synthesis of 1,4-diselenafulvene by dehydrochlorination. It is known that 1,4-diselenafulvene [6] is the reagent for synthesis of tetraselenafulvalene, the precursor of "organic metals" [7], complexes with charge transfer and ion-radical salts possessing high electric conductivity. Besides, the presence of the double bond and the chlorine atom capable of substitution provide additional possibilities for further functionalization.

2-Chloromethyl-1,3-diselenol (II), light-yellow liquid, bp 91–93°C (1 mm Hg). ^{1}H NMR, δ, ppm: 3.80 d (2H, CH_2Cl, ^3J 7.7 Hz), 5.10 t (1H, SeCHSe, ^3J 7.7 Hz), 7.04 s (2H, CH=CH). ^{13}C NMR, δ, ppm: 38.84 (SeCHSe), 48.88 (CH_2Cl), 117.52 (CH=). Mass spectrum, *m/z* (I_{rel}, %): 248 $[M]^+$ (42), 199 (100), 160 (18), 133 (10), 106 (22), 80 (16), 53 (12), 27 (20). Found, %: C 19.89; H 1.87; Cl 13.98. $C_4H_5ClSe_2$. Calcd, %: C 19.49; H 2.04; Cl 14.39.

NMR spectra were registered on a Bruker DPX 400 in $CDCl_3$ at working frequencies of 400.13 (^{1}H, HMDS) and 100.61 MHz (^{13}C, HMDS). Mass spectrum was taken on a GCMS–QP5050A Shimadzu chromatomass spectrometer with ionization by electron impact with the energy of ionizing electrons of 70 eV.

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