

LETTERS
TO THE EDITOR

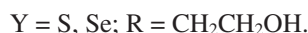
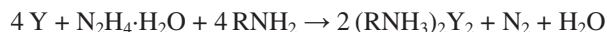
2-Hydroxy-3-mercaptopropaneselenol-1

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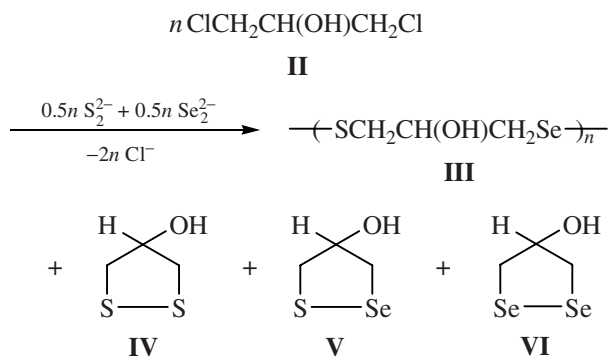
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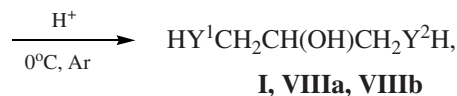
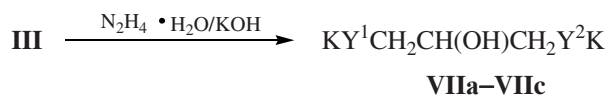
Derivative of glycerol containing in one molecule OH, SH and SeH groups, 2-hydroxy-3-mercaptopropaneselenol-1 (**I**), was not described in the literature. For its synthesis we have used elemental sulfur and selenium, 1,3-dichloropropanol-2 (**II**) and the approaches described in [1]. Preactivation of sulfur and selenium was performed in the system hydrazine hydrate–monoethanolamine where S_2^{2-} and Se_2^{2-} anions are generated from these elements.



Combined solutions of sulfur and selenium in the system used were introduced into the reaction with dichloride **II** to obtain oligomer product **III** (81% to dichloride **II** brought into the reaction). Chromatomass spectrometry of the dichloromethane extract of the reaction mixture revealed the presence of 4-hydroxy-1,2-dithiolane **IV** (M^+ 122 m/z) (yield ~2%), 4-hydroxy-1-thio-2-selenolane **V** (M^+ 170 m/z for ^{80}Se) (~1%), and 4-hydroxy-1,2-diselenolane (**VI**) (M^+ 218 m/z) (6 %).



Oligomer **III** was subjected to reductive splitting in the system hydrazine hydrate–KOH that led to the formation of three types of chalcogenolates **VIIa–VIIc**. Mild acidification of the solution of dichalcogenolates **VII** results in the formation of a mixture of dichalcogenols **I**, **VIIIa**, and **VIIIb**. Fractions enriched with compounds **VIIIa**, **I**, and **VIIIb** were isolated by vacuum distillation.



VIIa, VIIIa, $Y^1 = Y^2 = S$; **VIIIb, I**, $Y^1 = S$, $Y^2 = Se$; **VIIc, VIIIb**, $Y^1 = Y^2 = Se$.

Separately prepared at 60–65°C solutions of 3.2 g of sulfur and 7.8 g of selenium, each in the mixture of 15 ml of hydrazine hydrate and 1.8 ml of monoethanolamine, were combined. To the obtained mixture at 50–55°C 11.7 ml of dichloride **II** was added dropwise. The reaction mixture was heated for 4.5 h at 65–75°C. The polymer formed was separated by decanting, washed with water, ethanol and ether; 17 g of brown polymer was obtained. The hydrazine layer was treated with dichloromethane, the solvent was removed, and the residue was analyzed by chromatomass spectrometry. The prepared polymer (10 g) was added to the solution of 16.5 g of KOH in 74 ml of hydrazine hydrate and on complete dissolution of the polymer the reaction mixture was poured in an argon

flow into a beaker with 100 g of ice and 175 ml of conc. HCl. The content of the beaker in an argon flow was extracted with dichloromethane, the extract was dried over magnesium sulfate, the solvent was removed, and the residue (6.8 g) containing, by the data of ^1H NMR analysis, 2.7 g of compound **I**, was distilled in vacuum. Fraction with bp 115–120°C (2.5 mm Hg) contained ca. 75% of compound **I**, its yield being 27% to the polymer taken. ^1H NMR (CDCl_3), δ , ppm: 0.49 t (1H, SeH), 1.54 t (1H, SH), 2.74 m (4H, CH_2S , CH_2Se), 3.2 br.s (1H, OH), 3.72 m (1H, CHO). ^{13}C NMR (CDCl_3), δ_{C} , ppm, (J , Hz): 23.3 (C–Se, $J_{\text{C-Se}}$ 53.3), 30.4 (C–S), 72.6 (C–O). IR spectrum, cm^{-1} : 2296 $\nu_{\text{Se-H}}$, 2549 $\nu_{\text{S-H}}$. Mass spectrum, m/z ($I_{\text{rel.}}$, ^{80}Se): 170 (1) $[M - \text{H}_2]^+$, 154 (12) $[M - \text{H}_2\text{O}]^+$, 125 (7) $[M - \text{CH}_3\text{S}]^+$, 112 (10) $[\text{SSe}]^+$, 107 (8) $[M - \text{H}_2\text{O} - \text{CH}_2\text{SH}]^+$,

94, 93 (9) $[\text{CH}_2\text{Se}]^+$, $[\text{CHSe}]^+$, 77 (1) $[M - \text{CH}_3\text{Se}]^+$, 73 (3) $[M - \text{H}_2\text{O} - \text{HSe}]^+$, 59 (12) $[M - \text{H}_2\text{O} - \text{CH}_3\text{Se}]^+$, 47 (10) $[\text{CH}_3\text{S}]^+$, 45 (8) $[\text{CHS}]^+$, 44 (3), 43 (7), 41 (9).

IR spectra were recorded on a Bruker IFS-25 instrument, ^1H and ^{13}C NMR spectra, on a Bruker DPX-400 spectrometer (at 400.12 and 100.63 MHz, respectively) in CDCl_3 solutions, internal standard HMDS. Mass spectra were obtained on a Shimadzu GCMS-QP5050A spectrometer with quadrupole mass-analyzer, energy of ionizing electrons 70 eV.

REFERENCES

1. Grabel'nykh, V.A., Sukhomazova, E.N., Levanova, E.P., Vshivtsev, V.Yu., Zhanchipova, E.R., Klyba, L.V., Albanov, A.I., Russavskaya, N.V., and Korchevin, N.A., *Russ. J. Gen. Chem.* 2007, vol. 77, no. 10, p. 1817.