ISSN 1070-3632, Russian Journal of General Chemistry, 2008, Vol. 78, No. 4, pp. 608–613. © Pleiades Publishing, Ltd., 2008.
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Chalcogen-containing Analogs of Ethylene Glycol and Its Derivatives

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Abstract—Ethylene chlorohydrin when reacted with elemental chalcogens or dimethyl dichalcogenides in the hydrazine hydrate–alkali system forms chalcogen-containing analogs of ethylene glycol and its derivatives (dichalcogenated β-diglycols, chalcogenated ethanols, and chalcogenated methyl cellosolves).

DOI: 10.1134/S1070363208040166

Ethylene glycol and its ethers (cellosolves and glymes) are important ligands for complex formation, and they are widely used as effective solvents possessing specific solvation ability [1]. Replacement of oxygen in these compounds by chalcogens (S, Se, or Te) enhances their donor power, especially with respect to heavy metal cations [2]. Ethylene glycol and its monothio analog (2-sulfanylethanol) are used in multistage organic syntheses for protection of the carbonyl group, and, therewith, 2-sulfanylethanol is more effective both for introducing and removing the protective group [3]. The use of 2-sulfanylethanol in organic synthesis opens the way to functionalized sulfides and sulfoxides [4]. It was found just recently that 2-sulfanylethanol is a stabilizing ligand for preparing semiconductor zinc sulfide nanoparticles [5]. However, sulfur-containing and, moreover, selenium- or tellurium-containing analogs of ethylene glycol and its simplest derivatives still remain hardly accessible compounds.

We have developed convenient methods for synthesis of dichalcogenated β-diglycols $(HOCH_2CH_2)Y_2$ (Y = S, Se, Te) **Ia–Ic** and hetero analogs of ethylene glycol $HOCH_2CH_2YH$ (Y = S, Se) **IIa, IIb** and methyl cellosolve $HOCH_2CH_2YCH_3$ (Y=S, Se, Te) **IIIa–IIIc**, based on reactions of ethylene chlorohydrin with elemental chalcogens and dimethyl dichalcogenides in the hydrazine hydrate–alkali system (Table 1).

The hydrazine hydrate–alkali redox system activates elemental chalcogens via formation of polychalcogenide anions Y_x^{2-} [6]. For preferred formation of anions with a required x value, the Y : KOH ratio is varied. To form dichalcogenide anions Y_2^{2-}, a 1:1 Y:KOH ratio is needed. In the case of sulfur or selenium, the reaction is performed in the presence of water as solvent, and in the case of tellurium, excess hydrazine hydrate is used [6].

$$4Y + 4\ KOH + N_2H_4 \cdot H_2O \rightarrow 2\ K_2Y_2 + N_2 + 5\ H_2O. \quad (1)$$

To prevent conversion of chalcogens to compounds with a positive oxidation degree, an excess of hydrazine hydrate with respect to the stoichiometry of reaction (1) was used in all cases.

In the reaction of ethylene chlorohydrin with elemental sulfur activated according to reaction (1), the organosulfur compounds that are formed do not separate as an organic phase, evidently due to their high solubility in the water–hydrazine medium. Extraction of the reaction mixture with methylene chloride allows two main products to be isolated in a total yield of about 60%. Dithio-β-diglycol **Ia** is formed in 29% yield.

$$2\ ClCH_2CH_2OH \xrightarrow[-2\ KCl]{K_2S_2} (HOCH_2CH_2)_2S_2. \quad (2)$$

Ia

Along with this disulfide product, 2-sulfanylethanol (**IIa**) was obtained in a fairly high yield (30%). Thiol **IIa** is evidently formed by reductive cleavage of

Table 1. Physicochemical characteristics of compounds **I–III**

Comp. no.	bp., °C (*p*, mm Hg)	Found, %			Formula	Calculated, %		
		C	H	Y		C	H	Y
Ia	152–156 (2)	31.51	6.56	S 40.87	$C_4H_{10}O_2S_2$	31.17	6.49	41.56
Ib	185–190 (2)	19.01	3.98	Se 63.80	$C_4H_{10}O_2Se_2$	19.36	4.03	63.70
IIa[a]	70–72 (29)	31.03	7.78	S 41.00	C_2H_6OS	30.77	7.69	41.02
IIb	81–82 (31)	18.92	4.83	Se 63.07	C_2H_6OSe	19.20	4.80	63.20
IIIa	93–94 (50)	38.98	8.56	S 34.82	C_3H_8OS	39.13	8.69	34.78
IIIb	89–90 (23)	25.95	5.80	Se 56.84	C_3H_8OSe	25.91	5.75	56.82
IIIc	84–85 (5)	19.32	4.45	Te 67.73	C_3H_8OTe	19.19	4.26	68.02

[a] Characteristics of compound **IIa** agree with published data [13].

disulfide **Ia** with excess hydrazine present in the water–hydrazine phase.

$$2\,\mathbf{Ia} + N_2H_4{\cdot}H_2O \rightarrow 4\,HOCH_2CH_2SH + N_2 + H_2O. \quad (3)$$
$$\mathbf{IIa}$$

Disulfides are known to be readily reduced with hydrazine hydrate even in the absence of alkali [7]. The most facile reaction occurs, when disulfide and hydrazine hydrate are present in the same phase, which is the case in reaction (3).

By GC–MS we also detected in the dichloromethane extract of the reaction mixture monothio-β-diglycol **IVa** (yield ~1% yield). Most probably, this product is formed due to partial transformation of sulfur in reaction (1) to the monosulfide anion.

$$2ClCH_2CH_2OH \xrightarrow[-2\,KCl]{K_2S} (HOCH_2CH_2)_2S. \quad (4)$$
$$\mathbf{IVa}$$

The reaction of selenium with ethylene chlorohydrin in the hydrazine hydrate–alkali system proceeds more unambiguously as compared to sulfur. Diseleno-β-diglycol **Ib** separates as an organic layer in 75% yield.

$$2ClCH_2CH_2OH \xrightarrow[-2\,KCl]{K_2Se_2} (HOCH_2CH_2)_2Se_2. \quad (5)$$
$$\mathbf{Ib}$$

The lower solubility of product **Ib** in the water–hydrazine medium hinders, under the conditions of reaction (5), reduction of Se–Se bonds with excess hydrazine hydrate and formation of 2-selanylethanol (**IIb**). Note also that diselenides are more difficult to reduce with hydrazine than disulfides [7]. Only traces of monoseleno-β-diglycol **IVb** were found in this reaction by means of ^1H and ^{77}Se NMR spectroscopy (δ CH_2SeC 2.75 ppm, t; δ_{Se} 105.73 ppm), what agrees with the observation in [8] that selenium is less ambiguously, compared to sulfur, activated in the aqueous hydrazine–alkali system [8].

Tellurium dissolved in the hydrazine hydrate–alkali system when reacted with ethylene chlorohydrin forms ditelluro-β-diglycol **Ic** in 32% yield. Regeneraton of the elemental tellurium is observed in the course of the reaction (conversion 48%). Monotelluro-β-diglycol **IVc** is formed in 11% yield, most probably, from ditelluride **Ic** which is unstable, like many ditellurides [9].

$$2\,ClCH_2CH_2OH \xrightarrow[-2\,KCl]{K_2Te_2} (HOCH_2CH_2)_2Te_2$$
$$\mathbf{Ic}$$
$$\xrightarrow[-Te]{} (HOCH_2CH_2)_2Te. \quad (6)$$
$$\mathbf{IVc}$$

Compounds **Ic** and **IVc** were characterized by ^1H NMR spectroscopy (Table 2). Behavior of the molecular ions of **Ib, IIb, IIIb**, and **IIIc** , formed under electron impact, was studied. The fragmentation pattern of di-seleno-β-diglycol **Ib** and 2-selanylethanol (**IIb**) is sim-ilar to that of their sulfur-containing analogs [10, 11], though the molecular ion is very unstable (in both cases, W_M 2) (Scheme 1).

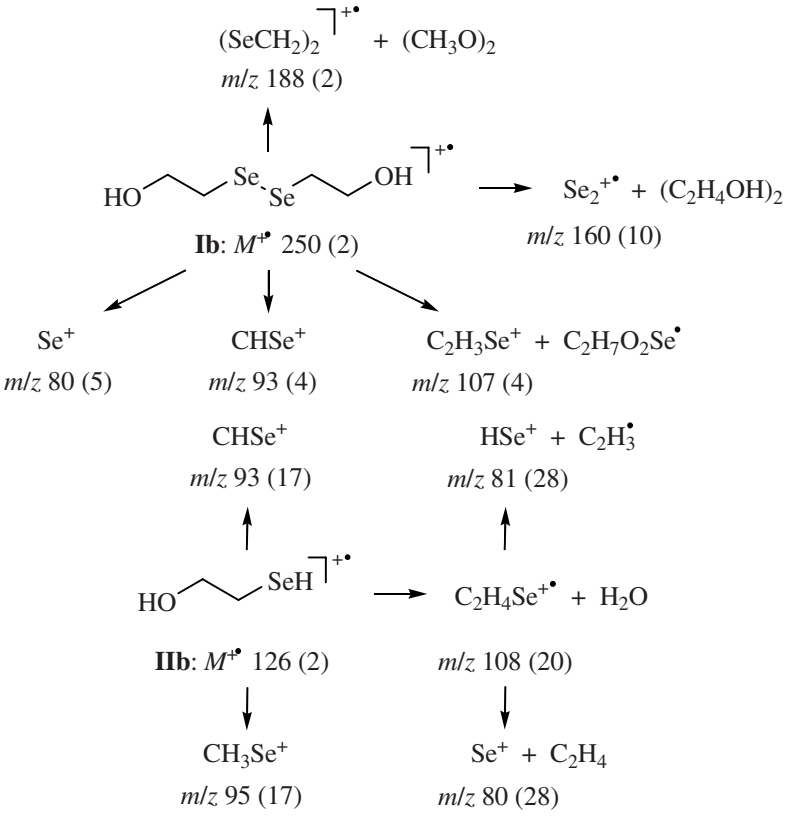
By contrast, 2-methylselanyl- and 2-methyltellanylethanols (**IIIb**, **IIIc**) form stable molecular ions

Table 2. Spectral characteristics of compounds **I–III**

Comp. no.	IR spectrum, cm^{-1}	NMR spectrum, δ, ppm		Mass spectrum, *m/z* (*I*, %, of the total ion current) ^{32}S, ^{80}Se, ^{130}Te
		^{1}H	^{13}C	
Ia	3312 (ν_{OH}); 2922, 2873 (ν_{CH}); 1654 (δ_{OH}); 1461, 1400, 1286 [$\delta(CH_2)$]; 1218, 1155, 1060, 1045, 1007, 819, 640, 485	2.84 t (CH_2S), 3.82 t (CH_2O), 4.16 br.s (OH)	40.87 (CH_2S), 60.03 (CH_2O)	154 (3) $[M]^{+\cdot}$ [10]
Ib	3300 (ν_{OH}); 2900, 2845 (ν_{CH}); 1440, 1390, 1245 [$\delta(CH_2)$]; 1110, 1040, 1015, 980, 950, 900, 770, 710, 620, 520, 425	3.09 t (CH_2Se), 3.91 t (CH_2O), 2.82 s (OH)	32.58 (CH_2Se), $^{1}J_{C-Se}$ 75.7 Hz; 61.75 (CH_2O)[a]	250 (1) $[M]^{+\cdot}$, 204 (1) $[C_2H_5OSe_2]^{+\cdot}$, 188 (2) $[C_2H_4Se_2]^{+\cdot}$, 175 (2) $[CH_3Se_2]^{+}$, 160 (10) $[Se_2]^{+\cdot}$, 107 (4) $[C_2H_3Se]^{+}$, 93 (10) $[CHSe]^{+}$, 80 (5) $[Se]^{+}$, 45 (40) $[C_2H_5O]^{+}$, 44 (5) $[C_2H_4O]^{+\cdot}$, 43 (15) $[C_2H_3O]^{+}$, 42 (3) $[C_2H_2O]^{+\cdot}$
Ic	3332, 3189 (ν_{OH}); 2924, 2861 (ν_{CH}); 2630, 2033, 1611 (δ_{OH}); 1459, 1402, 1361 [$\delta(CH_2)$]; 1264, 1104, 1066, 1038, 1003, 888, 714, 615	2.84 t, 3.31 t (CH_2Te), 3.83 m (CH_2O), 3.18 br.s (OH)	7.46, 8.70 (CH_2Te); 63.30, 64.28 (CH_2O)	–
IIa	3353 (ν_{OH}); 2934, 2874 (δ_{CH}); 2556 (ν_{S-H}); 1635 (δ_{OH}); 1463, 1417, 1384 [$\delta(CH_2)$]; 1293, 1236, 1169, 1062, 1048, 1014, 941, 908, 758, 661, 471	1.51 t (SH), $^{3}J_{H-S-C-H}$ 8.2 Hz; 2.68 m (SCH_2), 3.70 t (OCH_2), $^{3}J_{H-C-C-H}$ 5.9 Hz	27.26 (SCH_2), 63.77 (OCH_2)	78 (12) $[M]^{+\cdot}$ [11]
IIb	3327 (ν_{OH}); 2979, 2938, 2871 (ν_{C-H}); 2299 (ν_{Se-H}); 1637 (δ_{OH}); 1461, 1413, 1381 [$\delta(CH_2)$]; 1277, 1201, 1153, 1067, 1046, 1011, 933, 862, 799, 651, 456	–0.68 t (SeH), $^{3}J_{H-Se-C-H}$ 7.5 Hz; 2.74 m (CH_2Se), 3.73 t (CH_2O), $^{3}J_{H-C-C-H}$ 6.2 Hz; 2.00 (OH)	21.13 ($SeCH_2$), 63.98 (OCH_2)	126 (2) $[M]^{+\cdot}$, 108 (20) $[C_2H_4Se]^{+\cdot}$, 95, 93 (17) $[CH_3Se]^{+}$, $[CHSe]^{+}$, 81, 80 (28) $[HSe]^{+}$, $[Se]^{+}$
IIIa	3385 (ν_{OH}); 2962, 2920, 2878 (ν_{CH}); 2041, 1720, 1654, 1638 (δ_{OH}); 1460, 1427, 1322, 1288, 1228 [$\delta(CH_2)$]; 1169, 1044, 958, 939, 816, 770, 699, 654, 470	2.10 s (CH_3S), 2.68 t (CH_2S), 2.88 s (OH), 3.72 t (OCH_2)	14.89 (CH_3S), 36.90 (CH_2S), 59.73 (OCH_2)	[14]
IIIb	3366 (ν_{OH}); 2994, 2967, 2925, 2872, 2827 (ν_{C-H}); 2176, 2041, 1865, 1610 (δ_{OH}); 1463, 1423, 1407, 1385, 1341, 1269 [$\delta(CH_2)$]; 1194, 1156, 1065, 1043, 1002, 972, 902, 811, 793, 739, 672, 591, 455	1.99 s (CH_3Se), $^{2}J_{H-Se}$ 10.39 Hz; 2.71 t (CH_2Se), 3.21 s (OH), 3.75 t (OCH_2)	3.65 (CH_3Se), $^{1}J_{C-Se}$ 61.62 Hz; 28.37 (CH_2Se), $^{1}J_{C-Se}$ 61.81 Hz; 60.52 (OCH_2)[b]	140 (11) $[M]^{+\cdot}$, 109 (12) $[M - CH_3O]^{+}$, 95, 93 (32) $[CH_3Se, CHSe]^{+}$, 80 (10) $[Se]^{+}$, 45 (18) $[C_2H_5O]^{+}$
IIIc	3345 (ν_{OH}); 2970, 2922, 2868 (ν_{C-H}); 2427, 2031, 1654 (δ_{OH}); 1416, 1264, 1221[$\delta(CH_2)$]; 1150, 1061, 1037, 995, 944, 922, 833, 708, 624, 523	1.82 s (CH_3Te), $^{2}J_{H-C-^{125}Te}$ 20.23 Hz; 2.73 t (CH_2Te), 2.77 s (OH), 3.72 t (OCH_2)	–22.12 (CH_3Te), $^{1}J_{C-^{125}Te}$ 157.8 Hz; 8.16 (CH_2Te), $^{1}J_{C-^{125}Te}$ 156.6 Hz; 63.3 (OCH_2)[c]	190 (9) $[M]^{+\cdot}$, 162 (8), 145–143 (21) $[CH_3Te, CHTe]^{+}$, 130 (14) $[Te]^{+}$, 45 (32) $[C_2H_5O]^{+}$

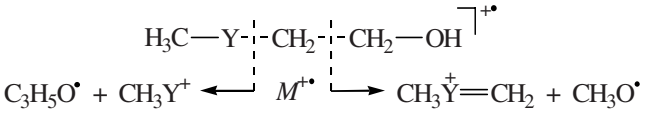
[a] ^{77}Se NMR spectrum, δ_{Se} 268.88 ppm. [b] ^{77}Se NMR spectrum, δ_{Se} 21.29 ppm. [c] ^{125}Te NMR spectrum, δ_{Te} 28.63 ppm.

Scheme 1.

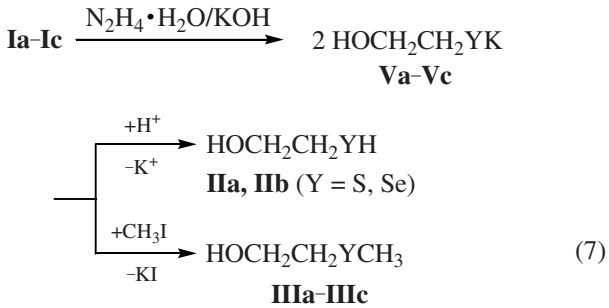


[$M^{+\cdot}$] (Table 2). Their main fragmentation patterns involve cleavage of C–C and C–Y bonds (Scheme 2).

Scheme 2.



All three dichalcogenated β-diglycols **Ia–Ic** under the action of the hydrazine hydrate–alkali system without an additional solvent undergo reductive cleavage by the Y–Y bond to form chalcogenolates **Va–Vc**.



Acidification of solution of chalcogenolates **Va, Vb** under mild conditions (0°, argon) provides 2-sulfanylethanol (**IIa**) and 2-selanylethanol (**IIb**) in yields of 65 and 47%, respectively.

Thiol **IIa** is fairly widely used in organic synthesis, while selenol **IIb** (like most other selenols [12]) is a scarcely accessible compound, even though its synthetic potential is as inexhaustible as that of thiol **IIb**.

2-Tellanylethanol (**IIc**) could not be prepared by reaction (7), evidently due to its extreme instability [9, 12].

Treatment of solutions of chalcogenolates **Va–Vc** with methyl iodide gave chalcogenated analogs of methyl cellosolve **IIIa–IIIc**. Their yield decreases in going from selenium **IIIb** (63%) to sulfur **IIIa** (41%) and tellurium **IIIc** (20%) derivatives.

Considering the possible perspectives of application of compounds **IIIb**, **IIIc** (by analogy with sulfide **IIIa**) in organic synthesis and in the chemistry of coordination compounds, a new procedure of their synthesis was developed. The procedure is based on reductive activation of dimethyl dichalcogenides with

the hydrazine hydrate–alkali system, followed by reaction (without isolation of corresponding methanechalcogenolates) with ethylene chlorohydrin.

$$2\,(CH_3)_2Y_2 + N_2H_4\cdot H_2O + 4\,KOH \rightarrow 4\,CH_3YK + N_2 + 5\,H_2O, \quad (8)$$

$$CH_3YK + ClCH_2CH_2OH \rightarrow \textbf{IIIb, IIIc}, \quad (9)$$

Y = Se, Te.

Compound **IIIb**, **IIIc** were prepared by reactions (8) and (9) in yields of 61 and 57 %, respectively.

Hence, we offered simple and effective methods for preparing chalcogen-containing derivatives of ethylene glycol, promising reagents for organic synthesis and ligands for complex formation, based on the use of the hydrazine hydrate–alkali system.

EXPERIMENTAL

The IR spectra were obtained on Specord IR-75 and Bruker IFS-25 spectrometers in thin layer. The ^{1}H, ^{13}C, ^{77}Se, and ^{125}Te NMR spectra were taken on a Bruker DPX-400 spectrometer (400.1, 100.6, 76.3, and 126.2 MHz respectively) in $CDCl_3$, CD_3OD, and mixtures of these solvents; internal reference HMDS.

The mass spectra were measured on a Shimadzu GCMS-QP5050A instrument (SPB™-5 column, 60000 × 0.25 mm, film thickness 0.25 μm). Injector temperature 250°; carrier gas helium, flow rate 0.7 ml min^{-1}; temperature program 60°–260°, heating rate 15 deg min^{-1}; detector temperature 250°; ionization energy 70 eV, ion source temperature 200°; detected mass range 34–650 D.

Analysis of liquid reaction products was carried out on a Tsvet-500 chromatograph (steel column, 2000 × 5 mm, packing XE-60 on Inerton N-AW-HMDS; linear temperature programming from 30° to 230° at a rate 12 deg min^{-1}; carrier gas helium) and on an LKhM 80-MD-2 chromatograph (column 2000 × 3 mm, packing 5% XE-60 on Inerton AW-HMDS; linear temperature programming at a rate 12 deg min^{-1}; carrier gas helium).

Bis(2-hydroxyethyl) disulfide (Ia). Potassium hydroxide, 15.7 g, hydrazine hydrate, 7 g, and 15 ml of water were placed in a flask equipped with a stirrer, a reflux condenser, a thermometer, and a reagent supply system. Powdered sulfur, 9 g, was added to the solution, and the resulting mixture was kept for 2 h at 80°–85° and cooled to 40°. Ethylene chlorohydrin, 22.5 g, was then added to the mixture, and it was stirred for 1.5 h at 55–60°C, cooled to 25°, and treated with methylene chloride (2 × 30 ml). The extract was dried over magnesium sulfate, and the solvent was removed. The residue was distilled in a vacuum to obtain 6.1 g of compound **Ia** as a colorless liquid crystallising in the condenser, mp 35°, and 6.3 g of 2-sulfanylethanol (**IIa**). The characteristics of the compounds obtained are listed in Tables 1 and 2.

Bis(2-hydroxyethyl) diselenide (Ib) was obtained analogously from 31.4 g of KOH, 14 g of hydrazine hydrate, 30 ml of water, 44.2 g of powdered selenium, and 45.1 g of ethylene chlorohydrin. The organic layer formed was separated, washed with water, dried over $MgSO_4$, and distilled in a vacuum to obtain 52 g of compound **Ib** as a viscous light yellow oil. Its characteristics are listed in Tables 1 and 2.

Bis(2-hydroxyethyl) ditelluride (Ic) was obtained analogously from 15.7 g of KOH, 140 g of hydrazine hydrate, 35.7 g of powdered tellurium (in the absence of water), and 22.5 g of ethylene chlorohydrin. Precipitation of elemental tellurium (18.5 g) was observed in the course of the reaction. It was removed, and the reaction mixture was treated with methylene chloride. The extract was dried over $MgSO_4$, and the solvent was distilled off to obtain 10.62 g of a dark red liquid residue. According to the ^{1}H and ^{13}C NMR spectra, it is a mixture of compounds **Ic** and **IVc**. Their characteristics are listed in Table 2.

2-Selanylethanol (IIb). Compound **Ib**, 5.91 g, was dissoled in a mixture of 21 g of hydrazine hydrate and 7 h of KOH at 75°–80° for 2.5 h. The reaction mixture was cooled to 25° and poured with stirring under argon into a mixture of ice and 90 ml of conc. HCl. The mixture obtained was extracted with methylene chloride and dried over $MgSO_4$, the solvent was distilled off, and the residie, 2.3 g, was distilled in a vacuum. The fraction with bp 81° (31 mm Hg) contained a practically pure compound **IIb**, 1.3 g, colorless liquid. Its characteristics are listed in Tables 1 and 2.

2-(Methylsulfanyl)ethanol (IIIa). Compound **Ia**, 2.06 g, was dissolved in a mixture of 20.1 g of hydrazine hydrate and 4.5 g of KOH at 80°–85° for 2.5 h. The reaction mixture was cooled to room temperature, and 9.51 g of methyl iodide was slowly added. The mixture was heated at 33–37°C for 3.5 h, extracted

with methylene chloride, and the extract was dried over $MgSO_4$. The solvent was removed, and the residue, 1.5 g, was distilled in a vacuum. The fraction with bp 93–94°C (50 mm Hg) contained a practically pure com-pound **IIIa**, 1 g, colorless liquid. Its characteristics are listed in Tables 1 and 2.

2-(Methylselanyl)ethanol (IIIb). *a*. Compound **Ib**, 3.02 g, was dissolved in a mixture of 18.3 g of hydrazine hydrate and 4.1 g of KOH at 80–85°C for 2.5 h. The reaction mixture was cooled to room temperature, and 5.2 g of methyl iodide was slowly added. The mixture was heated for 35–40°C for 1.5 h, extracted with methylene chloride, and the extract was dried over $MgSO_4$. The solvent was removed, and the residue was distilled in a vacuum to give a fraction with bp 89–90° C (23 mm Hg), containing a practically pure compound **IIIb**, 2.12 g, colorless liquid. Its characteristics are listed in Tables 1 and 2.

b. To a solution of 3.13 g of potassium hydroxide in 14 g of hydrazine hydrate, 2.1 g of dimethyl diselenide was added, and the mixture was stirred for 2 h at 75–80°C. After that, 1.8 g of ethylene chlorohydrin was slowly added dropwise at 40°, and the reaction mixture was stirred for 2.5 h at 55–60°C, extracted with methylene chloride, and the extract was dried over $MgSO_4$. The solvent was removed, and the residue was distilled in a vacuum as described above to obtain 1.9 g of pure compound **IIIb**.

2-(Methyltellanyl)ethanol (IIIc). *a*. Compound **Ic** was dissoloved in a mixture of 13.1 g of hydrazine hydrate and 2.94 g of KOH at 80–85°C for 2.5 h. The reaction mixture was cooled to room temperature, and 3.72 g of methyl iodide was slowly added to it. The mixture was kept at 35–40°C for 1 h and extracted with methylene chloride. The extract was dried over $MgSO_4$. The solvent was removed, and the residue was distilled in a vacuum to obtain 0.65 g of a fraction with bp 84–85°C (5 mm Hg), containing a practically pure compound **IIIc**, a reddish clear transparent liquid. Its characteristics are listed in Tables 1 and 2.

b. Dimethyl ditelluride, 2.21 g, was added to a solution of 2.6 g of potassium hydroxide in 11.6 g of hydrazine hydrate, and the resulting mixture was stirred for 2 h at 80°C. After that 1.25 g of ethylene chlorohydrin was slowly added dropwise at 40°C. The mixture was stirred for 2.5 h at 55–60°C, cooled, and extracted with CH_2Cl_2. The extract was dried over $MgSO_4$, the solvent was removed, and the residue was distilled in a vacuum was described above to obtain 1.65 g of pure compound **IIIb**.

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