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Dedicated to Full Member of the Russian Academy of Sciences B.A. Trofimov on his 70th anniversary

Selective Synthesis of Isomeric Dithioglycerols

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Abstract—Reactions of 2,3-dibromopropan-1-ol and 1,3-dichloropropan-2-ol with elemental sulfur activated in the system hydrazine hydrate–2-aminoethanol at 30–35°C gave oligomeric polysulfides which were subjected to reductive cleavage with formation of individual dithioglycerol isomers. Activation of sulfur with the use of alkali gives rise to mixtures of isomeric products.

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The role of glycerol in organic synthesis and biochemical processes is difficult to overestimate [1]. The use of superbasic systems proposed by B.A. Trofimov made it possible to obtain glycerol trivinyl ether from glycerol and acetylene [2], while vinylation of glycerol with acetylene in cesium superbasic system was accompanied by elimination with formation of 1,2- and 2,3-divinyloxypropenes [3]. Sulfur-containing analogs of glycerol are effective antidotes for heavy metals [4], analytical reagents [5], and ligands stabilizing nanoparticles of chalcogenide materials [6]. 2,3-Disulfanylpropan-1-ol (**Ia**) and some its derivatives exhibit tuberculostatic activity [7], and 1,3-disulfanylpropan-2-ol (**Ib**) is used as starting material in the synthesis of effective growth regulator Brugierol [8]. Nevertheless, wide application of dithioglycerols **Ia** and **Ib** in organic synthesis is restricted due to difficulties in their preparation, especially as individual isomers.

Dithioglycerols are commonly prepared by reaction of the corresponding dihalohydrins with an alcoholic solution of sodium hydrogen sulfide [7]. However, the scope of application of this procedure is limited because of difficult preparation of sodium hydrogen sulfide [9] and high probability for dehydrohalogenation by the action of its alcoholic solution [10].

Trofimov and co-workers previously synthesized trithioglycerol [11] and proposed a procedure for the preparation of dithioglycerols **Ia** and **Ib** by reactions of 2,3-dibromopropan-1-ol (**II**) and 1,3-dichloropropan-2-ol (**III**) with a solution of sulfur in the system aqueous hydrazine–alkali at 60°C, followed by reduction of polysulfide oligomers thus formed [12]. The yield of dithiols **Ia** and **Ib** was 37–42%, and it was improved to 62–64% using lead acetate at the isolation stage. However, although initial compounds **II** and **III** were pure isomers, in both cases dithiols **Ia** and **Ib** were isolated as mixtures containing 10% and more of isomerization product, which were difficult to separate. It was presumed [12] that the isomerization occurs at the stage of formation of polysulfide oligomers, where strongly basic hydrazine hydrate–alkali system promotes dehydrohalogenation to give oxirane fragments; subsequent opening of the oxirane ring by the action of disulfide ion (S_2^{2-}) gives rise to isomeric products.

We found that the synthesis of polysulfide oligomers from dihalopropanols **II** and **III** and a solution of sulfur in the system hydrazine hydrate–2-aminoethanol, which was successfully used previously for the preparation of di- and polythiols [13], gives (after reduction) dithioglycerols **Ia** and **Ib** as almost pure isomers. The process may be described as follows. Dissolution of sulfur in a mixture of hydrazine hydrate

Scheme 1.

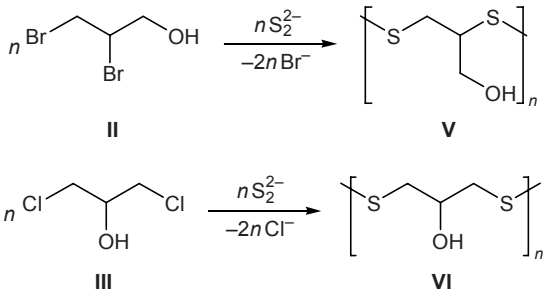
$$1/2\,S_8 + N_2H_4\cdot H_2O + 4\,H_2NCH_2CH_2OH \longrightarrow (HOCH_2CH_2\overset{+}{N}H_3)_2S_2^{2-} + (H_2N\overset{+}{N}H_3)_2S_2^{2-} + N_2 + H_2O$$

IVa **IVb**

with 2-aminoethanol results in preferential formation of 2-hydroxyethylammonium and hydrazinium disulfides **IVa** and **IVb** [14] (Scheme 1).

Addition of dihalopropanol **II** or **III** to the resulting solution of disulfides **IV** at 30–35°C leads to fast formation of oligomeric products **V** and **VI**, respectively (Scheme 2).

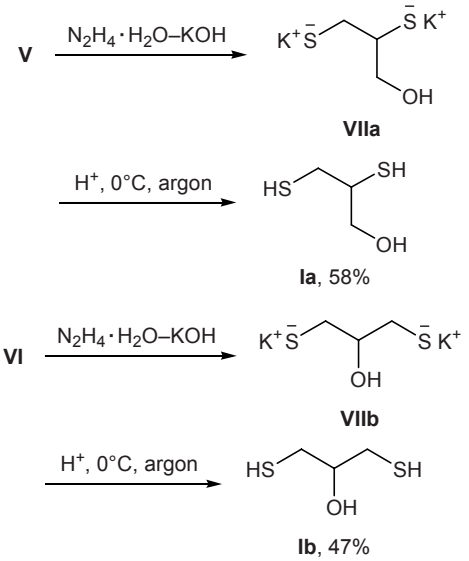
Scheme 2.



Oligomers **V** and **VI** are formed in almost quantitative yield; judging by the concentration of residual bromine and chlorine, their molecular weight is about 2000 a.m.u., which corresponds to an average *n* value of 15 to 17. It should be noted that the molecular weight of analogous oligomers obtained in the system hydrazine hydrate–alkali was approximately twice as large [12].

Reductive cleavage of oligomers **V** and **VI** at S–S bonds by the action of hydrazine hydrate–KOH [15] afforded the corresponding dipotassium dithiolates **VIIa** and **VIIb** which were converted (without isolation) into dithiols **Ia** and **Ib** (Scheme 3). According to

Scheme 3.

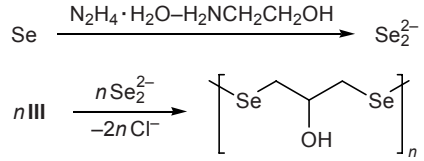


the GLC, GC–MS, and ^{1}H and ^{13}C NMR data, compounds **Ia** and **Ib** were pure isomers.

Thus, with account taken of the data given in [12], dehydrohalogenation of dihalopropanols **II** and **III** to the corresponding oxiranes is unlikely to occur in the system hydrazine hydrate–2-aminoethanol at a temperature below 35°C (cf. [16]). A probable reason is lower basicity of the system hydrazine hydrate–2-aminoethanol as compared to hydrazine hydrate–alkali. The reaction of elemental sulfur with 2-(chloromethyl)oxirane (**VIII**) in hydrazine hydrate–2-aminoethanol at 20–25°C, i.e., even under milder conditions, also gives oligomeric product whose reductive cleavage leads to formation of 70% of dithiol **Ib** containing 3–5% of isomer **Ia** (according to the GLC and ^{1}H NMR data). Raising the temperature favors formation of dithiol **Ia**. Therefore, had the dehydrohalogenation process occurred at the stage of formation of oligomers from dihalides **II** and **III**, the formation of isomeric dithiols could not be avoided.

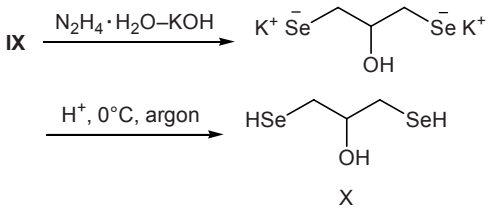
An additional proof for the proposed reaction path (i.e., that involving no dehydrohalogenation) in the system hydrazine hydrate–2-aminoethanol is provided by the synthesis in the same system of 1,3-diselenoglycerol **IX** from dichloride **III** and elemental selenium [17] (Scheme 4).

Scheme 4.



We previously showed [18] that neither compounds **II** and **III** nor 2-(chloromethyl)oxirane (**VIII**) gave rise to oligomeric products in the reactions with selenium in hydrazine hydrate–alkali. Here, the major product was allyl alcohol, and elemental selenium was regenerated. It was presumed that 1,3-dichloropropan-2-ol (**III**) is preliminarily converted into 2-(chloromethyl)oxirane (**VIII**) and that reaction of the latter with diselenide ion (Se_2^{2-}) is responsible for the formation of allyl alcohol. On the other hand, dichloride **III** reacted with selenium in the system hydrazine hydrate–2-aminoethanol to give oligomer **IX** (Scheme 4). The yield of **IX** is 95%; its average molecular weight (determined on the basis of the concentration of residual chlorine) was 3500 (n = 16). Reductive cleavage of compound **IX** gave 78% of diselenol **X** (Scheme 5).

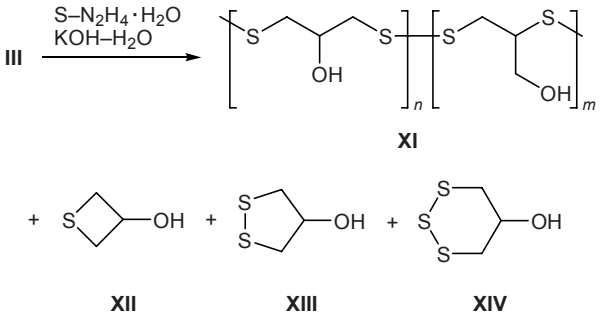
Scheme 5.



According to the GLC, GC–MS, and ^{1}H NMR data, no isomeric 1,2-diselenoglycerol was present in the product.

Using as an example the reaction of dichloride **III** with sulfur we examined the effect of the basicity of the hydrazine hydrate–KOH system (the basicity was varied by dilution with water) on the selectivity of formation of dithiol **Ib** and its isomer **Ia**. Oligomeric polysulfides were obtained in [12] using 300 ml of water per mole of sulfur. We carried out the reaction of 1,3-dichloropropan-2-ol (**III**) with sulfur in the system containing 1800 ml of water per mole of sulfur (sixfold dilution). As a result, the yield of oligomer **XI** decreased to 29%, and its composition and properties resembled those of oligomer **VI**. The reaction was accompanied by formation of cyclic products **XII**–**XIV** in an overall yield of 28% (Scheme 6).

Scheme 6.



In the reaction of dichloride **III** with sulfur in hydrazine hydrate–2-aminoethanol only traces of compounds **XII**–**XIV** were detected; however, by treatment of oligomer **VI** with chloroform-*d* we succeeded in isolating fairly pure compound **XIII** (yield ~1%) and recording its NMR spectra (see Experimental). When the polycondensation was carried out under the conditions given in [12], the overall yield of heterocyclic compounds **XII**–**XIV** did not exceed 5%. Increase in the yield of cyclic products with reduction of the monomer concentration (in our case S_x^{2-}) is consistent with the general relations holding in polycondensation processes [19].

Oligomer **XI** has a molecular weight of ~4000 ($n \approx 33$) (cf. [12]), and its reduction leads to a mixture of dithiols **Ia** and **Ib** at a ratio of 9 : 1 (GLC and ^{1}H data). Thus dilution of the system with water does not reduce the contribution of dehydrochlorination of compound **III**, and oligomeric product **XI** contains structural fragments intrinsic to oligomers **V** and **VI**.

Heterocyclic compounds **XII**–**XIV** were identified by GLC and GC–MS analysis of the reaction mixture. At a KOH-to-sulfur ratio of 1 : 1 (preferential generation of K_2S_2 [15]) compounds **XII**–**XIV** were formed at a ratio of 1 : 10 : 1.5. We failed to isolate heterocyclic products **XII**–**XIV** by vacuum distillation, though Dittmer and Christy [20] previously reported that 3-hydroxythietane (**XII**) can be distilled under reduced pressure. Obviously, the presence in the product mixture of thermally unstable trisulfide derivative, 5-hydroxy-1,2,3-trithiane (**XIV**), promotes fast polymerization of all compounds. The reductive cleavage of oligomers **XI** may be represented by Scheme 7.

Scheme 7.

Thus the use of the hydrazine hydrate–2-aminoethanol system ensures preparation of individual isomeric dithioglycerols from the corresponding dihalopropanols and elemental sulfur.

EXPERIMENTAL

The progress of reactions was monitored, and liquid products were analyzed, by GLC on LKhM 80-MD-2 (2000×3-mm column packed with 5% of DC-550 on Chromaton N-AW-HMDS; linear oven temperature programming at a rate of 12 deg/min; carrier gas helium) and Tsvet-500 chromatographs (2000×5-mm steel column packed with 5% of XE-60 on Chromaton N-AW-HMDS; linear oven temperature programming from 30 to 230°C at a rate of 12 deg/min; carrier gas helium). The IR spectra were recorded on Specord 75IR and Bruker IFS-25 spectrometers from thin films. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker DPX-400 instrument (400.1 and 100.6 MHz, respectively) using $CDCl_3$ as solvent and hexamethyldisiloxane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Shimadzu GCMS-QP5050A instrument (SPB-5 capillary column, 60000×0.25 mm, film thickness 0.25 μm; injector temperature 200°C; carrier gas helium, flow rate 0.7 ml/min; oven temperature programming from 60 to

240°C at a rate of 10 deg/min; detector temperature 200°C; quadrupole mass analyzer, ion source temperature 200°C, a.m.u. range 34–650 D).

Poly[sulfanediyl(3-hydroxypropane-1,2-diyl)sulfanediyl] (V). Elemental sulfur, 1.6 g (50 mmol), was dissolved in a mixture of 8.3 ml (166 mmol) of hydrazine hydrate and 1.0 ml (16.6 mmol) of 2-aminoethanol, heated to 65°C. The mixture was heated under stirring for 2.5 h at 70–73°C and cooled, 2.5 ml (25 mmol) of 2,3-dibromopropan-1-ol (**II**) was added dropwise, the mixture was stirred for 2 h at 45–50°C and cooled, and oligomer **V** separated as a green viscous ductile material. The product was washed with water, ethanol, and diethyl ether, and dried under reduced pressure. Yield 3.4 g (98%). IR spectrum, ν, cm^{-1}: 3408, 2924, 2865, 1619, 1457, 1402, 1262, 1227, 1181, 1143, 1038, 964, 894, 840, 812, 580, 497, 426. Found, %: C 27.20; H 4.90; Br 8.74; S 48.16. $(C_3H_6OS_2)_n$. Calculated, %: C 29.51; H 4.92; S 52.46 (the carbon and sulfur percentages were considerably underestimated due to high concentration of residual bromine). M ~1830.

Poly[sulfanediyl(2-hydroxypropane-1,3-diyl)sulfanediyl] (VI). Elemental sulfur, 9.6 g (300 mmol), was dissolved in a mixture of 45 ml (900 mmol) of hydrazine hydrate and 5.5 g (90 mmol) of 2-aminoethanol, heated to 65°C. 1,3-Dichloropropan-2-ol (**III**), 19.35 g (150 mmol), was slowly added to the resulting solution at 27–35°C, the mixture was stirred for 2.5 h at room temperature, and oligomer **VI** separated as a light yellow viscous ductile material. The product was washed with water, ethanol, and diethyl ether, and dried under reduced pressure. Yield 17.9 g (98%). IR spectrum, ν, cm^{-1}: 3332, 2970, 2813, 2745, 2650, 1608, 1402, 1339, 1250, 1183, 1128, 1070, 1010, 982, 930, 850, 822, 739, 475, 423. Found, %: C 28.40; H 5.22; Cl 3.28; S 50.3. $(C_3H_6OS_2)_n$. Calculated, %: C 29.51; H 4.92; S 52.46. M ~2160.

A 1-g sample of oligomer **VI** was treated with $CDCl_3$ over a period of 18 h. According to the ^1H and ^{13}C NMR data, the transparent extract contained 1,3-dithiolan-4-ol (**XIII**). ^1H NMR spectrum, δ, ppm: 3.17 d.d (2H, CH_2S), 3.95 br.s (1H, OH), 4.94 m (1H, CHO, *ABX* spin system, $^2J_{AB}$ = 11.5, $^3J_{AX}$ = 3.4, $^3J_{BX}$ = 1.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 46.6 (CH_2S), 75.2 (CHO).

2,3-Disulfanylpropan-1-ol (Ia, 1,2-dithioglycerol). A solution of 5.6 g (100 mmol) of potassium hydroxide in 25 ml (500 mmol) of hydrazine hydrate was heated to 80°C, 2.5 g (20 mmol) of oligomer **V** was added, and the mixture was stirred for 2 h at that temperature. The mixture was then cooled to 25°C, poured under argon into a mixture of ice with 55 ml of concentrated hydrochloric acid, and extracted with methylene chloride. The extract was dried over $MgSO_4$, and the solvent was distilled off to leave 1.44 g of almost pure dithiol **Ia**. Yield 58%, bp 106°C (5 mm). IR spectrum, ν, cm^{-1}: 3386 (OH); 2927, 2873 (CH); 2547 (SH). ^1H NMR spectrum (50°C), δ, ppm: 1.68 t (1H, CH_2S**H**, ^3J = 8.4 Hz), 1.74 d (1H, CHS**H**, ^3J = 8.5 Hz), 2.59 s (1H, OH), 2.83 d.d (2H, C**H**$_2$SH, ^3J = 6.24 Hz), 3.02 m (1H, CH), 3.74 d (2H, C**H**$_2$OH, ^3J = 5.8 Hz); in the spectrum recorded at 25°C, the SH proton signals were displaced downfield to δ 1.71 and 1.76 ppm, respectively. ^{13}C NMR spectrum, δ_C, ppm: 29.4 (CH_2S), 44.8 (CHS), 65.2 (CH_2O). Mass spectrum, m/z (I_{rel}, %): 124 $[M]^{+\cdot}$ (1), 106 $[M - H_2O]^{+\cdot}$ (16), 93 (10), 90 $[M - H_2S]^{+\cdot}$ (11), 77 $[M - HSCH_2]^+$ (12), 73 (12), 72 (8), 64 (20), 61 (16), 60 $[C_2H_4S]^{+\cdot}$ (34), 59 (100), 58 (20), 57 (41), 48 (1), 47 $[CH_3S]^+$ (21), 45 $[HCS]^+$ (27) (cf. [12]). Found, %: C 29.15; H 6.48; S 51.81. $C_3H_8OS_2$. Calculated, %: C 29.03; H 6.45; S 51.61.

1,3-Disulfanylpropan-2-ol (Ib, 1,3-dithioglycerol). Oligomer **VI**, 10 g (82 mmol), was dissolved in a mixture of 102 ml (2.05 mol) of hydrazine hydrate and 23 g (0.41 mol) of KOH on heating to 75–80°C (2.5 h). The solution was cooled to room temperature, poured under argon into a mixture of ice with 240 ml of concentrated hydrochloric acid, and extracted with methylene chloride and diethyl ether. The extract was purged with argon and dried over $MgSO_4$. Removal of the solvent left 4.25 g of 1,3-dithioglycerol **Ib** which was purified by vacuum distillation. Yield 47%, light yellow liquid, bp 88–90°C (2 mm). IR spectrum, ν, cm^{-1}: 3386 (OH); 2927, 2873 (CH); 2553 (SH). ^1H NMR spectrum, δ, ppm: 1.48 t (2H, SH), 2.63–2.69 m (4H, CH_2), 3.1 s (1H, OH), 3.70 m (1H, CH). ^{13}C NMR spectrum, δ_C, ppm: 29.55 (CH_2), 72.70 (CH). Mass spectrum, m/z (I_{rel}, %): 124 $[M]^{+\cdot}$ (3), 122 (1), 106 $[M - H_2O]^{+\cdot}$ (39), 77 (48), 76 (11), 73 (23), 72 (20), 64 (1), 61 (13), 59 (100), 58 (14), 57 (7), 49 (12), 48 (26), 47 (62), 46 (20), 45 $[CH_2SH]^+$ (50), 44 (11), 43 (24), 42 (6) (cf. [12]). Found, %: C 28.84; H 6.52; S 52.00. $C_3H_8OS_2$. Calculated, %: C 29.03; H 6.45; S 51.61.

Reaction of 2-(chloromethyl)oxirane (VIII) with a solution of sulfur. Powdered sulfur, 9.6 g (300 mmol), was dissolved in a mixture of 45 ml (900 mmol) of hydrazine hydrate and 5.52 g (90 mmol) of 2-aminoethanol on heating to 60–65°C.

The solution was heated for 2 h at that temperature and cooled to 18–25°C, 13.87 g (150 mmol) of 2-(chloromethyl)oxirane (**VIII**) was slowly added dropwise, the mixture was stirred for 3 h at 22°C, and the precipitate was separated, washed with water, ethanol, and diethyl ether, and dried under reduced pressure. Yield 17.93 g (98%); the product was a goldish viscous material. IR spectrum, ν, cm^{-1}: 3393, 3331, 3195, 2964, 2907, 1606, 1398, 1338, 1250, 1183, 1127, 1068, 1029, 980, 821, 740, 565, 471, 457. Found, %: C 26.94; H 6.00; Cl 1.50; S 45.62. $(C_3H_6OS_2)_n$. Calculated, %: C 29.51; H 4.92; S 52.46.

Reductive cleavage of a 8-g portion of the resulting oligomer under the conditions described above for the synthesis of dithiol **Ia** gave 6.44 g of a mixture of isomers **Ia** and **Ib** containing ~96% of the former (yield 75%).

When the synthesis of oligomer was performed at 65–70°C, a goldish yellow product was obtained (99%). Reductive cleavage of 7.1 g of the product gave 3.5 g of a mixture of dithiols **Ia** and **Ib** at a ratio of 2:3; yield of **Ib** 27%. Apart from dithiols **Ia** and **Ib**, the mixture contained 1,2-dithiolan-4-ol (**XIII**, 3%).

Poly[selanediyl(2-hydroxypropane-1,3-diyl)selanediyl] (IX). Elemental selenium, 7.9 g (100 mmol), was dissolved in a mixture of 15 ml (300 mmol) of hydrazine hydrate and 1.84 g (30 mmol) of 2-aminoethanol, heated to 70–75°C. The solution was heated for 2.5 h at that temperature and allowed to cool down to 40–45°C, 6.45 g (50 mmol) of dichloride **III** was slowly added dropwise, the mixture was stirred for 1 h at about 50°C and cooled to 25°C, and the precipitate was separated, washed with water, ethanol, and diethyl ether, and dried under reduced pressure. Yield 10.2 g (95%). IR spectrum, ν, cm^{-1}: 3420, 3259, 3148, 3052, 2962, 2927, 2705, 2581, 1627, 1494, 1399, 1258, 1190, 1102, 1064, 1005, 947, 792, 518, 478, 447, 413. Found, %: C 16.56; H 3.08; Cl 2.01; Se 71.54. $(C_3H_6OSe_2)_n$. Calculated, %: C 16.67; H 2.78; Se 73.15.

1,3-Diselanylpropan-2-ol (X). A solution of 8.4 g (150 mmol) of potassium hydroxide in 37 ml (750 mmol) of hydrazine hydrate was heated to 55–60°C, 6.4 g (30 mmol) of oligomer **IX** was added, and the mixture was stirred for 2 h at that temperature, cooled to 25°C, and treated as described above in the synthesis of dithiols **Ia** and **Ib**. The oily product was extracted into methylene chloride, the extract was dried over magnesium sulfate, and the solvent was distilled off. According to the GLC data, the residue was almost pure diselenol **X**. Yield 78%, bp 95–97°C (2 mm). The elemental analysis of **X** and its spectral parameters were given in [17].

Reaction of 1,3-dichloropropan-2-ol (III) with a dilute solution of sulfur in hydrazine hydrate–KOH. A solution of 2.8 g (50 mmol) of potassium hydroxide in a mixture of 3.5 ml (70 mmol) of hydrazine hydrate and 15 ml of water was heated to 70–80°C, 1.6 g (50 mmol) of sulfur and 10 ml of water were added, and the mixture was heated for 1 h at 80–85°C. The mixture was then diluted with 105 ml of water, 3.22 g (25 mmol) of dichloride **III** was added dropwise at 55–60°C, the mixture was stirred for 1 h at that temperature and cooled, and viscous sticky oligomer **XI** was separated, washed with water and ethanol, and dried under reduced pressure. Yield 0.89 g (29%). IR spectrum, ν, cm^{-1}: 3429, 2921, 2858, 1658, 1625, 1584, 1452, 1404, 1270, 1224, 1176, 1070, 1009, 743, 589, 466, 442, 412. Found, %: C 30.70; H 4.42; Cl 1.81; S 52.08. $(C_3H_6OS_2)_n$. Calculated, %: C 29.51; H 4.92; S 52.46.

After separation of oligomer **XI**, the aqueous hydrazine solution was extracted with methylene chloride, and the extract was dried over $MgSO_4$ and analyzed by GLC and GC–MS, It contained compounds **XII**–**XIV**.

Thietan-3-ol (XII). Mass spectrum, *m*/*z* (I_{rel}, %): 90 $[M]^{+\cdot}$ (92), 61 $[C_2H_5S]^+$ (46), 47 (8), 46 (100), 45 (62), 44 (96), 43 (66).

1,2-Dithiolan-4-ol (XIII). Mass spectrum, *m*/*z* (I_{rel}, %): 122 $[M]^{+\cdot}$ (100), 80 (11), 79 (35), 78 (57), 76 (13), 64 (11), 61 $[C_2H_5S]^+$ (9), 59 (11), 58 (75), 57 (33), 48 (21), 47 (61), 46 (58), 45 (67), 44 (13), 43 (85), 42 (9).

1,2,3-Trithian-5-ol (XIV). Mass spectrum, *m*/*z* (I_{rel}, %): 154 $[M]^{+\cdot}$ (100), 110 (44), 108 (27), 90 (14), 89 (38), 79 (14), 78 (11), 71 (7), 64 (30), 61 $[C_2H_5S]^+$ (13), 58 (11), 57 (20), 47 (39), 46 (42), 45 (82), 44 (37), 43 (56), 42 (7).

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