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Chalcogenation of 1,3-Dichlorobut-2-ene with Organic Dichalcogenides in the System Hydrazine Hydrate–Alkali

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Abstract—Electron-donor effect of the methyl group in 1,3-dichlorobut-2-ene hampers allylic rearrangement of its primary monochalcogenation products. The use of diphenyl disulfide under harsh conditions (Ph_2S_2–KOH, 1:10, 75–80°C) makes it possible to obtain a mixture of six bis(phenylsulfanyl)butenes, 1,1-bis(phenylsulfanyl)but-1-ene being the major component. No bis(phenylselanyl) derivatives have been formed on heating up to 80°C. Dipotassium ethane-1,2-dithiolate reacts with 1,3-dichlorobut-2-ene to give a linear product of chlorine substitution at the sp^3-carbon atom in two dichlorobutene molecules and a heterocyclic compound, 2-ethylidene-1,4-dithiane (a mixture of *E* and *Z* isomers), whose structure is different from the structure of the product obtained from 1,3-dichloropropene under analogous conditions. Mechanisms have been proposed for the formation of the isolated compounds.

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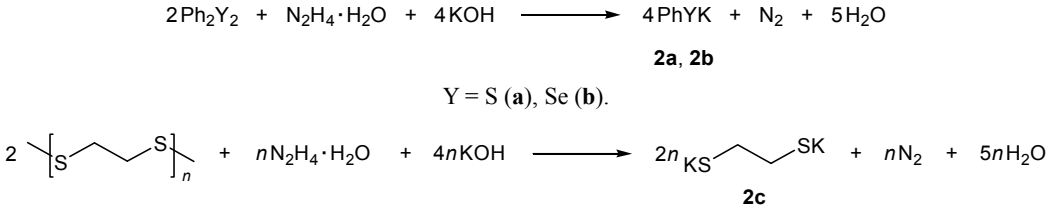
We previously demonstrated the possibility of obtaining promising unsaturated organochalcogen compounds, such as 1-chalcogenyl-3-chloropropenes, 1,3-dichalcogenylpropenes, and 2-ethylidene-1,3-dithiolane, from 1,3-dichloropropene, organic dichalcogenides R_2Y_2 [1], and poly(ethylene disulfide) [2] in the system hydrazine hydrate–alkali. The obtained compounds can be used to synthesize other organochalcogen derivatives [1, 3], as well as highly unsaturated structures containing no chalcogen atoms [4, 5], e.g., prostaglandin $F_{2\alpha}$ [6]. Taking into account the high synthetic potential of compounds obtainable from 1,3-dichloropropene, we examined the chalcogenation of its closest homolog, 1,3-dichlorobut-2-ene (**1**). Anderson et al. [7] reported the sulfanylation of compound **1** with benzenethiol in the presence of anhydrous potassium carbonate in boiling acetone (12 h; **1**–PhSH–K_2CO_3 ratio 0.9:1.0:1.2. Unlike the corresponding propenyl derivative, the product of substitution of the allylic chlorine atom by phenylsulfanyl group (84%) underwent neither thio-Claisen rearrangement nor cyclization.

Compound **1** is a commercial product available as a mixture of *Z* and *E* isomers at a ratio of 6.5:1.0 (according to the ^{1}H NMR data). The chalcogenating agents were generated from organic dichalcogenides, diphenyl disulfide, diphenyl diselenide, and poly(ethylene disulfide), which were preliminarily subjected to reductive cleavage at the S–S or Se–Se bond by the action of hydrazine hydrate–potassium hydroxide. Potassium chalcogenolates **2a** and **2b** or dipotassium ethane-1,2-dithiolate (**2c**) were thus formed [8]

Scheme 1.



(Scheme 1) and brought into reaction with dichlorobutene **1** directly in hydrazine hydrate solution without isolation.

As reported in [1], 1,3-dichloropropene reacted with Ph_2Y_2 under similar conditions to give products of substitution of either allylic chlorine atom or both chlorine atoms by phenylchalcogenyl group. It was convincingly shown [1] that dichalcogenylpropenes are formed via allylic rearrangement of the initially formed monosubstitution product; this rearrangement in the presence of bases involves isomerization of intermediate carbanions [1, 9]. In the reaction of benzenethiolate with 1,3-dichloropropene, the rearrangement occurs even at 25–35°C [1].

We have found that the presence of an electron-donor methyl group in molecule **1** destabilizes the neighboring carbanionic center [9] and dramatically affects the reaction of 1,3-dichlorobut-2-ene (**1**) with organic dichalcogenides.

Only monosubstitution products **3** were obtained in the reactions of 1,3-dichlorobut-2-ene (**1**) with chalcogenolates **2a** and **2b** up to a temperature of 60°C (Scheme 2). The yield of 3-chloro-1-phenylsulfanylbut-2-ene **3a** at 25–35°C was 93%, and at 60°C (5 h), 83%. Bis(phenylsulfanyl)butenes appeared in the reaction mixture when the reaction time was prolonged to 10 h at 60°C; in this case, the yield of **3a** was 70%. Benzeneselenolate **2b** gave rise to only monosubstitution product (yield 92%), while the yields of **3b** at 25–35°C was 94%.

Compounds **3a** and **3b** were isolated as mixtures of *E* and *Z* isomers at the same ratio as in the initial dichlorobutene **1**. Their structure was confirmed by IR, NMR (^1H, ^{13}C, ^{77}Se), and mass spectra.

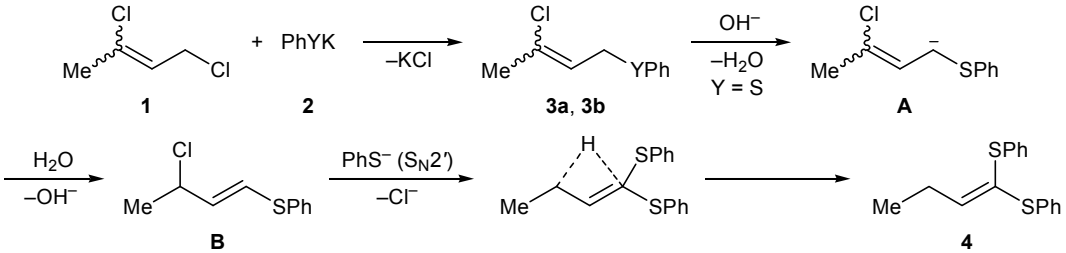
The lack of further transformations that could be expected due to the possibility of allylic rearrangement suggests that the methyl group in the $CH_3CCl=$ fragment hampers migration of the anionic center. The formation of bis(phenylsulfanyl) derivative from compound **3a** became appreciable only at 75–80°C in the presence of a large excess of alkali (Ph_2S_2–KOH ratio 1:10). However, even under these conditions, 10% of monosubstituted product **3a** remains in the reaction mixture after 5 h. The overall yield of bis(phenylsulfanyl) derivatives was 29%. According to the GC/MS data, they were represented by six isomers (*m*/*z* 272 $[M]^+$) at a ratio of 10:1:1:0.5:1:1, i.e., one isomer clearly predominated (yield 19%). By thorough analysis of the isomer mixture by ^1H NMR with the use of two-dimensional NMR techniques, the major isomer was identified as 1,1-bis(phenylsulfanyl)but-1-ene (**4**) (Scheme 2).

Presumably, compound **4** was formed via a series of transformations (Scheme 2), including deprotonation of monosubstitution product **3a** by the action of alkali and allylic rearrangement of the resulting carbanion **A**. Nucleophilic substitution of the chlorine atom in rearrangement product **B** by phenylsulfanyl group is hindered due to steric factor and electron-donor effect of the methyl group which reduces the electrophilicity of the carbon atom in the CHCl fragment. The same factors are likely to determine the S_N2' mechanism [9] of attack of PhS^- anion on molecule **B** with simultaneous 1,3-migration of hydrogen, which leads to the formation of compound **4**. Probably, this migration is favored by the electron-donor effect of the methyl group.

We failed to unambiguously determine the structure of the other bis(phenylsulfanyl)butene isomers. Nevertheless, the formation of a large number of isomers suggests that the presence of a methyl group in the molecule of bielectrophile **1** essentially changes the direction of transformations of primary monosubstitution product **3a** in comparison to analogous transformations of 1,3-dichloropropene.

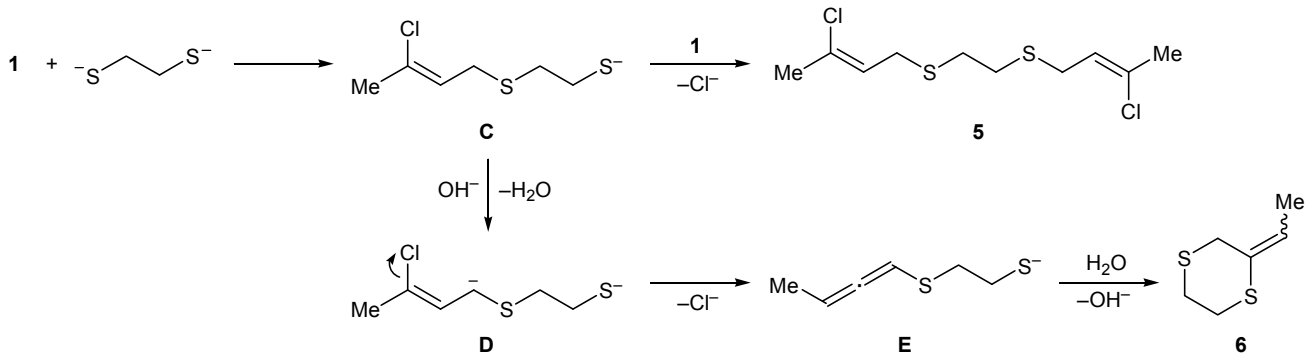
The PhSe substituent reduces the probability of allylic rearrangement in the propenyl fragment as compared to phenylsulfanyl group [1]. Therefore,

Scheme 2.



Y = S (**a**), Se (**b**).

Scheme 3.



monosubstituted product **3b** was formed in a high yield even at 50°C (Scheme 2). Raising the temperature to 80°C led to the formation of a complex mixture of compounds, among which we identified selenide **3b** and diphenyl diselenide; the latter is likely to result from thermal decomposition of bis(phenylselanyl)-butene derivatives under the given conditions.

The reaction of 1,3-dichlorobut-2-ene (**1**) with difunctional dipotassium ethane-1,2-dithiolate gave two products, linear compound **5** (as a result of substitution of the allylic chlorine atom in two molecules **1**) and cyclic 2-ethylidene-1,4-dithiane (**6**) as a mixture of *E* and *Z* isomers (Scheme 3). As expected, linear product **5** contained *Z*- and *E*-chlorobutenyl fragments with the *Z*/*E* ratio corresponding to the isomer ratio in initial compound **1** (~6.5 : 1).

The ratio of compounds **5** and **6** obtained at 25–35°C strongly depended on the concentration of alkali in the reaction mixture. When $(–SCH_2CH_2S–)_n$ was preliminarily treated with a mixture of hydrazine hydrate and potassium hydroxide at a ratio of (1 : 10), compounds **5** and **6** were formed at a ratio of 5 : 1. When the ratio $N_2H_4 \cdot H_2O$/KOH was 1 : 5, the ratio **5**/**6** changed to 9 : 1 (yield 59 and 22 wt %, respectively). Approximately equal amounts of **5** and **6** (**5**/**6** 1.1 : 1.0) were formed at 50°C (5 h). The *Z* isomer of **6** predominated, and the *E*/*Z* ratio 1 : 2 almost did not depend on the reaction temperature.

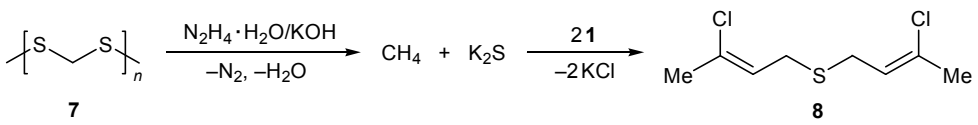
The lower homolog of **1**, 1,3-dichloropropene, reacted with dipotassium ethane-1,2-dithiolate to give a linear product and a five-membered heterocyclic compound, 2-ethylidene-1,3-dithiolane [2]. As shown by quantum chemical calculations, the latter is formed according to a mechanism similar to the formation of bis(phenylsulfanyl) derivative **4** (Scheme 2) via allylic rearrangement and S_N2' reaction [11].

As shown above, the allylic rearrangement of the monosubstitution product in the reaction of dichlorobutene **1** even with $PhS^–$ anion is fairly difficult to occur, though, according to the data of [1], the phenylsulfanyl group is most favorable for allylic rearrangement among the other examined chalcogenyl substituents. Therefore, no allylic rearrangement is observed in the structure containing an $^–SCH_2CH_2S$ substituent, and no dithiolane derivative is formed from dichlorobutene **1**.

Thus, the formation of compounds **5** and **6** can be illustrated by Scheme 3. The primary monosubstitution product **C** either reacts with the second molecule **1** to give linear compound **5** or undergoes dehydrochlorination through intermediate anion **D**; migration of hydrogen in the latter and elimination of chloride ion lead to allene derivative **E**. Intramolecular nucleophilic attack of the free thiolate group on the central carbon atom of the allene fragment of **E** [10] yields cyclic product **6**. Compounds **5** and **6** were not described previously. Their structure was confirmed by IR, ^1H and ^{13}C NMR, and mass spectra.

The reaction of dichlorobutene **1** with poly(methylene disulfide) (**7**) preliminarily treated with hydrazine hydrate–potassium hydroxide (**7**–KOH ratio 1 : 10, 85–90°C) gave 55% of bis(3-chlorobut-2-en-1-

Scheme 4.



n = 10–15.

yl) sulfide (**8**) containing no SCH_2S fragment (Scheme 4). The formation of sulfide **8** could be expected since the corresponding sulfide was obtained in 52% yield in the reaction with more reactive (than **1**) 2,3-dichloroprop-1-ene [12]. In reactions with unsaturated bielectrophiles, compounds like **8** are formed as a result of preferential cleavage of poly(methylene sulfides) at the C–S bond with generation of S^{2-} anions in the system hydrazine hydrate–alkali [13]. The subsequent reaction of K_2S with 1,3-dichlorobutene **1** yields sulfide **8** (Scheme 4). The latter was also obtained in 58% yield by adding compound **1** to a solution of sulfur in $N_2H_4 \cdot H_2O$–KOH (S/KOH ratio 1:2.5) at 25–35°C.

Thus, unsaturated organochalcogen compounds can be synthesized from 1,3-dichlorobut-2-ene (**1**) and organic dichalcogenides. The presence of a methyl group in the MeCCl= fragment strongly inhibits substitution of chlorine at the sp^2-hybridized carbon atom. The reaction of 1,3-dichlorobut-2-ene (**1**) with poly(ethylene disulfide) afforded previously unknown 2-ethylidene-1,4-dithiane as a mixture of *E* and *Z* isomers.

EXPERIMENTAL

The IR spectra were recorded on a Bruker IFS-25 spectrometer from thin films. The ^1H, ^{13}C, and ^{77}Se NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.13, 100.62, and 76.31 MHz, respectively, using $CDCl_3$ as solvent and tetramethylsilane (^1H, ^{13}C) or Me_2Se (^{77}Se) as internal standard. The mass spectra were obtained on a Shimadzu GCMS–QP5050A instrument (SPB-5 column, 60 m×0.25 mm; quadrupole mass analyzer, electron impact, 70 eV, ion source temperature 190°C, a.m.u. range 34–650).

Reductive cleavage of organic dichalcogenides and subsequent reaction with 1,3-dichlorobut-2-ene (1) (*general procedure*). A required amount of the corresponding dichalcogenide was added with vigorous stirring to a solution of potassium hydroxide in hydrazine hydrate heated to 60°C. The resulting solution was stirred for 2 h at 80–85°C and cooled to 25°C, and a required amount of 1,3-dichlorobut-2-ene (**1**) was added dropwise with stirring. The mixture was stirred for 5–10 h at a temperature indicated below, cooled to 25°C, and extracted with diethyl ether (3×50 mL). The combined extracts were washed with water and dried over $MgSO_4$, the solvent was removed, and the residue was subjected to analysis and appropriate treatment.

[(3-Chlorobut-2-en-1-yl)sulfanyl]benzene (3a) was obtained from 4.37 g (0.02 mol) of Ph_2S_2, 5.61 g (0.1 mol) KOH, and 2.5 g (0.02 mol) of **1** in 25 mL of hydrazine hydrate at 25–35°C (5 h). The residue, 3.7 g, was almost pure compound **3a**. Yield 93%, bp 93–95°C (2 mm); published data [7]: bp 84°C (0.75 mm). IR spectrum: ν 1659 cm^{-1} (C=C).

Z Isomer. ^1H NMR spectrum, δ, ppm: 2.06 s (3H, CH_3), 3.65 d (2H, CH_2S, ^3J = 7.2 Hz), 5.57 t (1H, =CH, ^3J = 7.2 Hz), 7.16–7.33 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 26.10 (CH_3), 32.04 (CH_2), 121.76 (=CCl), 126.19 (=CH), 128.93, 129.57, 131.46, 135.86 (Ph). Mass spectrum, *m*/*z* (I_{rel}, %): 200 (16) (^{37}Cl), 198 (40) (^{35}Cl) $[M]^+$.

E Isomer. ^1H NMR spectrum, δ, ppm: 1.84 s (3H, CH_3), 3.46 d (2H, CH_2S, ^3J = 8.5 Hz), 5.74 t (1H, =CH, ^3J = 8.5 Hz), 7.16–7.33 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 20.61 (CH_3), 33.09 (CH_2), 123.06 (=CCl), 127.03 (=CH), 129.00, 130.66, 133.22, 135.20 (Ph). Mass spectrum, *m*/*z* (I_{rel}, %): 200 (17) (^{37}Cl), 198 (43) (^{35}Cl) $[M]^+$. Found (isomer mixture), %: C 60.52; H 5.51; Cl 17.61; S 16.04. $C_{10}H_{11}ClS$. Calculated, %: C 60.44; H 5.58; Cl 17.84; S 16.13. M 198.71.

[(3-Chlorobut-2-en-1-yl)selanyl]benzene (3b) was obtained from 2.5 g (0.008 mol) of Ph_2Se_2, 2.24 g (0.04 mol) of KOH, and 1.0 g (0.008 mol) of **1** in 10 mL of hydrazine hydrate. The residue, 1.84 g, was almost pure compound **3b**. Yield 94%, bp 95–100°C (2 mm). IR spectrum: ν 1654 cm^{-1} (C=C).

Z Isomer. ^1H NMR spectrum, δ, ppm: 2.06 s (3H, CH_3), 3.64 d (2H, CH_2Se, ^3J = 7.9 Hz), 5.67 t (1H, =CH, ^3J = 7.9 Hz), 7.25–7.65 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 25.15 (CH_3), 26.17 (CH_2), 122.32 (=CCl), 127.21 (=CH), 128.97, 129.74, 133.41, 134.96 (Ph). ^{77}Se NMR spectrum: δ_{Se} 337.5 ppm. Mass spectrum, *m*/*z* (I_{rel}, %): 248 (8) (^{37}Cl, ^{80}Se), 246 (18) (^{35}Cl, ^{80}Se) $[M]^+$.

E Isomer. ^1H NMR spectrum, δ, ppm: 1.73 s (3H, CH_3), 3.46 d (2H, CH_2Se, ^3J = 8.7 Hz), 5.83 t (1H, =CH, ^3J = 8.7 Hz), 7.25–7.65 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 25.73 (CH_3), 29.75 (CH_2), 123.86 (=CCl), 127.84 (=CH), 128.35, 129.36, 132.91, 134.18 (Ph). ^{77}Se NMR spectrum: δ_{Se} 349.4 ppm. Mass spectrum, *m*/*z* (I_{rel}, %): 248 (4.6) (^{37}Cl, ^{80}Se), 246 (9) (^{35}Cl, ^{80}Se) $[M]^+$. Found (isomer mixture), %: C 48.71; H 4.53; Cl 14.57; Se 32.19. $C_{10}H_{11}ClSe$. Calculated, %: C 48.90; H 4.51; Cl 14.43; Se 32.15. M 245.61.

1,1-Bis(phenylsulfanyl)but-1-ene (4) was identified in a mixture of isomeric bis(phenylsulfanyl)butenes. ^{1}H NMR spectrum, δ, ppm: 1.05 t (3H, CH_3), 2.42 m (2H, CH_2), 6.39 t (1H, =CH), 7.20–7.44 m (10H, Ph). Mass spectrum: *m*/*z* 272 (I_{rel} 44%) $[M]^+$. Calculated: *M* 272.43.

1,2-Bis(3-chlorobut-2-en-1-ylsulfanyl)ethane (5) was obtained from 2.43 g (0.0264 mol) of poly(ethylene disulfide), 14.8 g (0.264 mol) of KOH, and 3.29 g of **1** in 65 mL of hydrazine hydrate at 25–35°C. The residue, 2.73 g, was a mixture of compounds **5** and **6** (59 and 22%, respectively), which were separated by vacuum distillation.

Compound **5**. bp 150–160°C (2 mm). IR spectrum: ν 1658 cm^{-1} (C=C). ^{1}H NMR spectrum, δ, ppm: *Z* isomer: 2.13 d (3H, CH_3, ^4J = 1.2 Hz), 2.71 s (4H, SCH_2CH_2S), 3.29 d (2H, SCH_2C=, ^3J = 7.5 Hz), 5.54 t.q (1H, =CH, ^3J = 7.5, ^4J = 1.2 Hz); *E* isomer: 2.08 s (3H, CH_3), 2.71 s (4H, SCH_2CH_2S), 3.17 d (2H, SCH_2C=, ^3J = 7.9 Hz), 5.62 t (1H, =CH, ^3J = 7.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: *Z* isomer: 26.02 (CH_3), 29.50 (S**C**H_2C=), 31.10 (SCH_2CH_2S), 122.41 (=CH), 132.82 (=CCl); *E* isomer: 20.86 (CH_3), 29.79 (S**C**H_2C=), 31.37 (SCH_2CH_2S), 123.87 (=CH), 132.82 (=CCl). Mass spectrum (two peaks on the chromatogram): *m*/*z* 270 (^{35}Cl) $[M]^+$. Found, %: C 44.40; H 5.98; Cl 25.81; S 23.58. $C_{10}H_{16}Cl_2S_2$. Calculated, %: C 44.28; H 5.94; Cl 26.14; S 23.64. *M* 271.27.

2-Ethylidene-1,4-dithiane (6). Yield 22%, bp 92–98°C (2 mm). IR spectrum: ν 1632 cm^{-1} (C=C).

Z Isomer. ^{1}H NMR spectrum, δ, ppm: 1.77 d.t (3H, CH_3, ^3J = 6.7, ^5J = 0.8 Hz), 2.99 m (4H, SCH_2CH_2S), 3.41 d.q (2H, SCH_2C=, ^4J = 1.1, ^5J = 0.8 Hz), 5.69 d.q (1H, CH=, ^3J = 6.7, ^4J = 1.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.36 (CH_3), 29.68, 31.19 (SCH_2CH_2S), 36.13 (S**C**H_2C=), 125.37 (CH=), 128.26 (=CS). Mass spectrum: *m*/*z* 146 (I_{rel} 100%) $[M]^+$.

E Isomer. ^{1}H NMR spectrum, δ, ppm: 1.68 d (3H, CH_3, ^3J = 7.0 Hz), 2.99 m (4H, SCH_2CH_2S), 3.48 br.s (2H, SCH_2C=), 5.88 q (1H, CH=, ^3J = 7.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 13.64 (CH_3), 29.85, 33.07 (SCH_2CH_2S), 39.28 (S**C**H_2C=), 126.11 (CH=), 127.52 (=CS). Mass spectrum: *m*/*z* 146 (I_{rel} 100%) $[M]^+$. Found (isomer mixture), %: C 49.19; H 6.80; S 44.12. $C_6H_{10}S_2$. Calculated, %: C 49.27; H 6.89; S 43.84. *M* 146.28.

Bis(3-chlorobut-2-en-1-yl) sulfide (8). *a*. From 2.3 g (0.0294 mol) of poly(methylene disulfide) (**7**), 16.5 g (0.294 mol) of KOH, and 3.68 g (0.0294 mol) of **1** in 70 mL of hydrazine hydrate at 25–35°C. The residue was a yellow liquid, 1.7 g, which was almost pure compound **8**. Yield 55%, bp 89–92°C (2 mm). IR spectrum: ν 1659 cm^{-1} (C=C). ^{1}H NMR spectrum, δ, ppm: *E* isomer: 2.07 s (3H, CH_3), 3.16 d (2H, CH_2S, ^3J = 8.5 Hz), 5.60 t (1H, =CH, ^3J = 8.5 Hz); *Z* isomer: 2.12 s (3H, CH_3), 3.27 d (2H, CH_2S, ^3J = 7.4 Hz), 5.55 t (1H, =CH, ^3J = 7.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: *Z* isomer: 26.18 (CH_3), 29.41 (CH_2S), 122.33 (CH=), 132.84 (=CCl); *E* isomer: 20.85 (CH_3), 29.67 (CH_2S), 123.94 (CH=), 132.84 (=CCl). Mass spectrum: *m*/*z* 210 (I_{rel} 15%) (^{35}Cl) $[M]^+$. Found, %: C 45.32; H 5.72; Cl 33.46; S 15.16. $C_8H_{12}Cl_2S$. Calculated, %: C 45.51; H 5.73; Cl 33.58; S 15.18. *M* 211.15.

b. From 0.8 g (0.025 mol) of sulfur, 3.5 g (0.0625 mol) of KOH, and 4.68 g (0.0374 mol) of **1** in 15 mL of hydrazine hydrate at 25–35°C. The residue, 2.28 g, was almost pure compound **8**. Yield 58%; the product was identical in boiling point and spectral parameters to a sample obtained as described above in *a*.

The main results were obtained using the equipment of the Baikal Joint Analytical Center, Siberian Branch, Russian Academy of Sciences.

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