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Specific Features of the Reactions of 2,3-Dichloro-1-propene with Dibenzylchalcogenides in the System Hydrazine Hydrate–Alkali

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Abstract—Dibenzyldisulfide and -diselenide react with 2,3-dichloro-1-propene in the system hydrazine hydrate–KOH by the domino mechanism: nucleophilic substitution of the allyl chlorine, dehydrochlorination with participation of the chlorine atom at the sp^2-carbon atom, allene–acetylene rearrangement, nucleophilic addition of the chalcogenide reagent to the triple bond. The effect of the nature of the chalcogen atom and the benzyl substituent on the studied domino reaction is discussed.

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2,3-Dichloro-1-propene (**I**) is a reactive species containing two chlorine atoms at the carbon atoms with different hybridization. Compound **I** can react with chalcogen-containing nucleophiles in several ways depending on the nature of the chalcogen in the reactant, reaction conditions and the ratio of the reagents. In the systems hydrazine hydrate–base, in which chalcogenide anions are readily generated from elemental chalcogens or organic dichalcogenides [1], the following possibilities were found: substitution by chalcogenide group only the chlorine atom at the sp^3-carbon atom [2, 3]; elimination of both chlorine atoms with the formation of allene [2, 4]; the sequence of reactions including substitution of the allyl chlorine followed by dehydrochlorination with the formation of allenyl compounds, allene-acetylene rearrangement, and nucleophilic addition of the chalcogenide reagent to the triple bond (the domino reaction) [5, 6]. These domino reactions with the use of diphenyldisulfide or -diselenide are readily controlled and allow to prepare the promising for organic synthesis unsaturated organochalcogen compounds (chloropropenyl, allenyl, acetylene chalcogenides and vinyl bischalcogenides) with rather high selectivity [5, 6].

With the goal of further investigation of the effect of the nature of the chalcogen atom and the substituent in organylchalcogenide ions on the processes of substitution and dehydrohalogenation with participation of chlorine atoms attached to the sp^3- and sp^2-hybridized carbon atoms, we have studied the reactions of 2,3-dichloro-1-propene (**I**) with dibenzyldisulfide **IIa** and -diselenide **IIb** in the system hydrazine hydrate–KOH. The presence of benzylchalcogenide fragments and multiple bonds in the structure of the expected products of these transformations allows to consider them as potential biologically active compounds or their precursors as well as promising synthons. For example, the use of the benzyl group in organic synthesis as a protecting group of thiols is well known [7], in particular, in the synthesis of polypeptides [8].

Dibenzyldichalcogenides Bn_2Y_2 (**IIa**, Y = S; **IIb**, Y = Se), like other dichalcogenides [1] in the system hydrazine hydrate–KOH are reduced to chalcogenolate ions [Eq. (1)].

$$\underset{\textbf{IIa, IIb}}{2Bn_2Y_2} + 4KOH + N_2H_4\cdot H_2O \rightarrow 4BnYK + N_2 + 5H_2O, \quad (1)$$

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

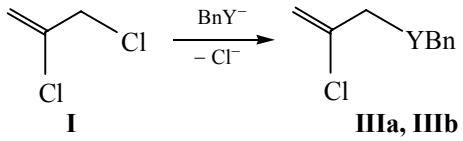
Taking into account the earlier obtained results [1, 5, 6] we have used the molar ratio Bn_2Y_2 : KOH = 1 : 5 for complete reduction of dichalcogenides.

Ratio of the products of the domino reaction of diphenyl- [5, 6] and dibenzyldichalcogenides (**IIa, IIb**) with 2,3-dichloro-1-propene (**I**) (30–35°C, 2 h, R_2Y_2 : KOH = 1 : 5, the yields are given in % in brackets)

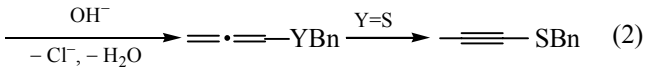
R_2Y_2	Product		
	III	**IV**	**V**
Ph_2S_2	1.0 (5%)	3.3 (23%)	3.3 (23%)
Ph_2Se_2	1.0 (36%)	1.0 (43%)	0.2 (8%)
Bn_2S_2	1.0 (41%)	0.5 (19%)	0.2 (9%)
Bn_2Se_2	1.0 (63%)	0.1 (5%)	–
Ph_2Se_2[a]	–	1.0 (19%)	3.0 (58%)
Bn_2Se_2[a]	1.0 (36%)	1.0 (34%)	0.4 (13%)

[a] R_2Y_2 : KOH = 1 : 10.

Like Ph_2S_2 [5] and Ph_2Se_2 [6], dibenzyldisulfide **IIa** with 2,3-dichloro-1-propene (**I**) in the system hydrazine hydrate–KOH at 30–35°C (2 h) gives three products: 2-chloro-3-benzylsulfanyl-1-propene (**IIIa**) (yield 41%), 1-benzylsulfanylpropadiene (**IVa**) (19%) and 1-benzylsulfanyl-1-propyne (b) (9%). In the same conditions, dibenzyldiselenide (**IIb**) affords only two products: 2-chloro-3-benzylselanyl-1-propene (**IIIb**) (63%) and 1-benzylselanylpropadiene (**IVb**) (5%). The selenium analog of the acetylene derivative **Va** was not detected in the products of the reaction. The formation of the above products can be represented by the sequence of transformations (the domino reaction) depicted in scheme (2).



I **IIIa, IIIb**



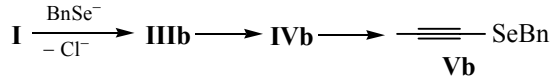
(2)

IVa, IVb **Va**

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

In order to estimate the effect of the nature of the chalcogen atom and the substituent in the organylchalcogenide ion on the course of the domino reaction (2) we have compared the molar ratios of the products formed under similar conditions from Ph_2Y_2 and Bn_2Y_2 (Y = S, Se) (see table).

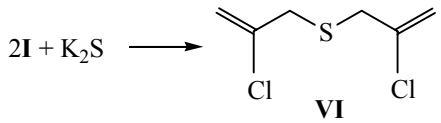
As can be seen from the table, the degree of conversion in the sequence of reactions presented in scheme (2) decreases for the same R on going from the thiolate to selenolate ions, and is substantially decreased from R = Ph to R = Bn. The increase of the alkali concentration in the reaction mixture (R_2Se_2 : KOH = 1 : 10) for Ph_2Se_2 results in complete conversion of the first product of the domino reaction, 2-chloro-3-phenylselanyl-1-propene [6]; for Bn_2Se_2 (**IIb**), the degree of conversion of product **IIIb** is also increased. Only in these conditions (Bn_2Se_2 : KOH = 1 : 10, 30–35°C, 2 h) the formation of the acetylenic derivative **Vb** could be detected (yield 13%) [scheme (3)].



Vb (3)

The reaction of disulfide **IIa** with **I** at –10 to –20°C results in chloropropenyl derivative **IIIa** in 60% yield. Products **IVa** and **Va** were not detected but in the reaction mixture was found bis(2-chloro-1-propen-3-yl)sulfide (**VI**) (yield 7%). Its formation can be due to sulfide ions S^{2-}, which can be generated upon reductive splitting of disulfide **IIa** according to reaction (1). This process occurs at 85–90°C [1], that is, in the conditions in which disulfide **IIa** can be reduced both at the C–S and the S–S bond [scheme (4)].

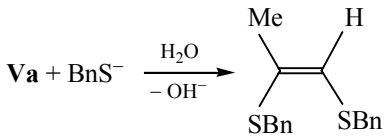
$$Bn–S–S–Bn + N_2H_4·H_2O + 3KOH \rightarrow PhMe + K_2S + BnSK + 4H_2O,$$



VI (4)

Sulfide **VI**, apparently, is formed at higher temperatures, too, (vide supra) but under these conditions it should rapidly suffer further transformations (dehydrochlorination and resinification or decomposition of the formed allylic and acetylenic sulfides).

At 60°C and the ratio Bn_2S_2 : KOH = 1 : 5 after 19 h two compounds are present in the reaction products: acetylenic sulfide **Va** (yield 58%) and *Z*-1,2-bis(benzylsulfanyl)-1-propene **VII** (yield 36%). Apparently, under these conditions the domino reaction goes further (as in the case of Ph_2S_2 [5] and Ph_2Se_2 [6]) [scheme (5)].

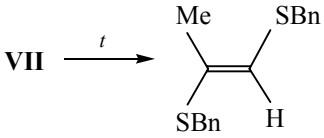


VII (5)

Compound **VII** is formed selectively as the *Z*-isomer, apparently, due to conventional *trans*-addition of the nucleophile to the triple bond [9].

Under the same conditions but for longer duration of the reaction time (26 h) the acetylenic sulfide **Va** was obtained in 52% yield, and product **VII**, in 44% yield.

Vacuum distillation of bissulfide **VII** results in partial *Z*→*E* isomerization and affords the mixture containing 65–75% of the *Z*-isomer **VII** and 25–35% of the *E*–isomer **VIIa** [Eq. (6)].



(6)

VIIa

Proceeding of isomerization [Eq. (6)] in the case of the benzyl derivative **VII** (as compared to its absence in the case of analogous phenyl derivative [5]) can be due to its higher boiling temperature, that facilitates the interconversion of the geometric isomers.

Dibenzyldiselenide **IIb** at 60°C after 30 h forms a complex mixture of products in which it was impossible to unambiguously identify 1,2-bis(benzylselanyl)-1-propene.

Therefore, the readily performed reaction of dibenzyldichalcogenides with 2,3-dichloro-1-propene allows to prepare unsaturated sulfides and selenides (including allylic and acetylenic derivatives) possessing the benzyl group at the heteroatoms.

EXPERIMENTAL

IR spectra were registered in thin layer on a Bruker IFS-25 spectrometer. ^{1}H, ^{13}C, ^{77}Se NMR spectra were registered on a Bruker DPX 400 (400.13, 100.62, 76.31 MHz, respectively) in $CDCl_3$ with internal standards TMS (^{1}H, ^{13}C), Me_2Se (^{77}Se). Mass spectra were obtained on a Shimadzu GCMS–QP5050A chromatomass spectrometer (column SPB-5, 60000 × 0.25 mm), quadruple mass analyzer in the mass range 34-650 Da, electron impact at 70 eV, temperature of ionic source 190°C. The values of *m/z* for Se- and Cl-containing ions are given for ^{80}Se and ^{35}Cl isotopes. The value of I_{rel} is given taking into account total intensity for all isotopes.

The reactions were monitored and the liquid products analyzed on a LKhM 80-MD-2 chromatograph (column 2000 × 3 mm, liquid phase DC-550, 5% on Chromaton N-AW-HMDS, linear temperature programming mode 12 deg/min, gas carrier is helium).

Reaction of 2,3-dichloro-1-propene (I) with dibenzyldisulfide IIa. To the solution of 8.0 g (0.014 mol) KOH in 35 mL of hydrazine hydrate 7.0 g (0.028 mol) of disulfide **IIa** was added portionwise at 40–50°C. The reaction mixture was heated at 85–90°C for 3 h, cooled to room temperature and 6.31 g (0.057 mol) of 2,3-dichloro-1-propene (**I**) was added dropwise. The reaction mixture was stirred for 2.5 h at 25°C, then 2 h at 30–35°C. After cooling to room temperature the mixture was extracted with CH_2Cl_2 (3 × 50 mL), the combined extracts dried over $MgSO_4$ and the solvent removed. The residue (7.34 g), from the GC, GC-MS and ^{1}H NMR analysis contains three products (**IIIa**, **IVa**, and **Va**) in the molar ratio (from ^{1}H NMR) 1.0 : 0.5 : 0.2 (the yields are given in the text).

2-Chloro-3-benzylsulfanyl-1-propene (IIIa). The reduction of 7.0 g of disulfide **IIa** was carried out as above. The reaction with 2,3-dichloro-1-propene (**I**) was performed at –10 to –20°C (10 h). Extraction with CH_2Cl_2 removal of solvent afforded 7.2 g of residue containing 95% (6.84 g) of compound **IIIa** and 5% (0.36 g) of bis(2-chloro-1-propene-3-yl)sulfide (**VI**) (yields are given in the text). Sulfide **IIIa** was isolated by vacuum distillation, bp 89–92°C (1.5 mm Hg). IR spectrum, ν, cm^{-1}: 3104, 3085, 3062, 3029, 2946, 2916, 1627(C=C), 1601, 1494, 1453, 1407, 1233, 1208, 1115, 1071, 1029, 890, 836, 770, 749, 701, 680, 630, 564, 519, 471. ^{1}H NMR spectrum, δ, ppm: 3.16 s (2H, CH_2Ph), 3.65 s (2H, SCH_2CCl=), 5.27 s (2H, CH_2=), 7.17–7.26 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 35.23 ($\underline{C}H_2Ph$), 38.58 ($S\underline{C}H_2CCl$=), 114.38 (CH_2=), 127.05 (C_p), 128.41, 128.93 (C_o, C_m), 138.26 (C_i), 137.30 (=CCl–). Mass spectrum, *m/z* (I_{rel}, %): 198 (4) $[M]^{+\cdot}$, 123 (4) $[BnS]^{+}$, 122 (26) $[PhCHS]^{+\cdot}$, 121 (3) $[PhC{\equiv}S^{+}]$, 92 (6) $[PhMe]^{+\cdot}$, 91 (33) $[C_7H_7]^{+}$, 77 (4) $[Ph]^{+}$, 65 (7) $[C_5H_5]^{+}$, 45 (5) $[HC{\equiv}S^{+}]$. Found, %: C 60.49; H 5.51; S 16.15; Cl 17.68. $C_{10}H_{11}SCl$. Calculated, %: C 60.44; H 5.58; S 16.13; Cl 17.84.

1-Benzylsulfanylpropadiene (IVa) was obtained in 19% yield at the temperature of the reaction of 30–35°C. After vacuum distillation, fraction with bp 90–91°C (2 mm Hg) contain (from ^{1}H NMR) 60% of the allenyl derivative **IVa** and 40% of compound **Va**. IR spectrum, ν, cm^{-1}: 1941 (C=C=C). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.77 s (2H, CH_2Ph), 4.90 d (2H, CH_2=C=, ^{3}J 6.4 Hz), 5.69 t (1H, C=C=CH–, ^{3}J 6.4 Hz),

7.19–7.25 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 36.85 (CH_2S), 80.19 ($\underline{C}H_2$=C=), 86.83 ($\underline{C}$H=C=), 128.10 (C_p), 128.35, 128.85 (C_o, C_m), 137.64 (C_i), 206.28 (=C=). Mass spectrum, *m/z* (I_{rel}, %): 162 (9) $[M]^{+\cdot}$, 161 (3) $[M - H]^+$, 129 (17) $[M - SH]^+$, 128 (3) $[M - H_2S]^{+\cdot}$, 91 (49) $[C_7H_7]^+$, 65 (8) $[C_5H_5]^+$, 51 (3).

1-Benzylsulfanyl-1-propyne (Va) was obtained in 58% yield at the temperature of the reaction of 60°C. bp 90–91°C (2 mm Hg). IR spectrum, ν, cm^{-1}: 2199 (C≡C). ^1H NMR spectrum, δ, ppm: 1.85 s (3H, Me–$\underline{C}$≡), 3.82 s (CH_2S), 7.24–7.28 m (Ph). ^{13}C NMR spectrum, δ_C, ppm: 4.87 (Me), 40.01 (SCH_2), 67.33 (≡C–S), 91.81 (≡$\underline{C}$–Me), 127.48 (C_p), 127.41, 128.88 (C_o, C_m), 136.94 (C_i). Mass spectrum, *m/z* (I_{rel}, %): 162 (7) $[M]^{+\cdot}$, 161 (6) $[M - H]^+$, 129 (15) $[M - SH]^+$, 128 (4) $[M - H_2S]^{+\cdot}$, 92 (3) $[C_7H_8]^{+\cdot}$, 91 (42) $[C_7H_7]^+$, 65 (7) $[C_5H_5]^+$, 51 (3). Found, %: C 73.95; H 6.29; S 19.60. $C_{10}H_{10}S$. Calculated, %: C 74.03; H 6.21; S 19.76.

Bis(2-chloro-1-propen-3-yl)sulfide (VI) was isolated by vacuum distillation of the mixture of products prepared, at the temperature of the reaction of –10 to –20°C. Boiling point and spectral characteristics coincide with those for the earlier prepared product [2].

***Z*-1,2-Bis(benzylsulfanyl)-1-propene (VII)** was prepared in 36–44% yield at the temperature of the reaction of 60°C. IR spectrum, ν, cm^{-1}: 1627 (C=C). ^1H NMR spectrum, δ, ppm: 1.83 s (3H, Me), 3.76 s, 3.86 s (4H, $\underline{CH_2}$Ph), 5.90 s (1H, =C–H), 7.14–7.26 m (Ph). ^{13}C NMR spectrum, δ_C, ppm: 23.90 (Me), 35.67, 37.96 ($\underline{C}H_2$Ph), 124.61 (=C–H); it is difficult to unambiguously assign the ^{13}C signals of the Me–$\underline{C}$= group and benzene rings in the range 127.04–138.15 ppm. Mass spectrum, *m/z* (I_{rel}, %): 286 (21) $[M]^{+\cdot}$, 195 (3) $[M - Bz]^+$, 92 (4) $[C_7H_8]^{+\cdot}$, 91 (56) $[C_7H_7]^+$, 65 (6) $[C_5H_5]^+$. Found, %: C 71.06; H 6.26; S 22.27. $C_{17}H_{18}S_2$. Calculated, %: C 71.28; H 6.33; S 22.38.

***E*-1,2-Bis(benzylsulfanyl)-1-propene (VIIa).** Distillation of compound **VII** [bp 210–217 °C (2 mm Hg)] gave two products (^1H, ^{13}C and GC-MS data). For compound **VIIa**: ^1H NMR spectrum, δ, ppm: 1.87 s (3H, Me), 3.67 s, 3.75 s (4H, $\underline{CH_2}$Ph), 5.79 s (1H, =C–H), 7.14–7.26 m (Ph). ^{13}C NMR spectrum, δ_C, ppm: 19.70 (Me), 36.72, 38.21 ($\underline{C}H_2$–Ph), other signals could not be identified. The mass spectrum is similar to that of the *Z*-isomer of **VII**.

Reaction of 2,3-dichloro-1-propene (I) with dibenzyldiselenide IIb. To the solution of 4.1 g (0.07 mol) of KOH in 18 mL of hydrazine hydrate 5.0 g (0.015 mol) of diselenide **IIb** was added in portions. The reaction mixture was heated at 85–90°C for 3 h, cooled to room temperature and added 3.3 g (0.03 mol) of 2,3-dichloro-1-propene (**I**) dropwise, stirred for 2.5 h at 25°C, then 2 h at 30–35°C and treated as above for diphenyldisulfide (**IIa**). The residue after removal of solvent (4.83 g) contained, according to the GC, GC-MS and ^1H NMR, two products (**IIIb** and **IVb**) in the molar ratio (from ^1H NMR) 1.0 : 0.1 (the yields are given in the table).

2-Chloro-3-benzylselanyl-1-propene (IIIb) was isolated by vacuum distillation of the residue. bp 117–118°C (2 mm Hg). IR spectrum, ν, cm^{-1}: 1624 (C=C). ^1H NMR spectrum, δ, ppm (*J*, Hz): 3.23 s (2H, Se$\underline{CH_2}$–CCl=, $^2J_{H–C–Se}$ 14.3 Hz), 3.75 s (2H, Se$\underline{CH_2}$–Ph, $^2J_{H–C–Se}$ 13.1 Hz), 5.19 d (1H, –CH=C–C, ^2J 1.1 Hz), 5.15–5.20 m (1H, –CH=C–C), 7.14–7.23 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 27.58 (Se$\underline{C}H_2$CCl=, $^1J_{C–Se}$ 61.6 Hz), 30.53 (Se$\underline{C}H_2$–Ph, $^1J_{C–Se}$ 69.0 Hz), 113.70 (CH_2=), 126.73 (C_p), 128.37, 128.92 (C_o, C_m), 138.33 (C_i), 13.43 (–CCl=). ^{77}Se NMR spectrum, δ_{Se}, ppm: 295.1. Mass spectrum, *m/z* (I_{rel}, %): 246 (9) $[M]^{+\cdot}$, 170 (10) $[PhCHSe]^{+\cdot}$, 92 (6) $[C_7H_8]^+$, 92 (60) $[C_7H_7]^+$, 65 (5) $[C_5H_5]^+$. Found, %: C 49.13; H 4.48; Se 32.04; Cl 14.43. $C_{10}H_{11}SeCl$. Calculated, %: C 48.90; H 4.51; Se 32.15; Cl 14.43.

1-Benzylselanylpropadiene (IVb) was isolated as a mixture with 1-benzylselanyl-1-propyne (**Vb**) by distillation. Fraction with bp. 110–112°C (2 mm Hg) contained 45% of compound **IVb**. Selenide **IVb**: IR spectrum, ν, cm^{-1}: 1942 (C=C=C). ^1H NMR spectrum, δ, ppm (*J*, Hz): 3.83 s (2H, Se$\underline{CH_2}$Ph, $^2J_{H–C–Se}$ 11.6 Hz), 4.68 d (2H, CH_2=C=C, $^4J_{H–H}$ 6.4 Hz), 5.78 t (1H, SeCH=C=C, $^4J_{H–H}$ 6.4 Hz), 7.14–7.23 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 29.88 (Se$\underline{C}H_2$Ph, $^1J_{C–Se}$ 60.0 Hz), 76.37 SeCH=C=C, $^1J_{C–Se}$ 110.8 Hz), 77.46 ($\underline{C}H_2$=C=C), 126.76 (C_p), 128.32, 128.85 (C_o, C_m), 138.27 (C_i), 205.77 (=C=). ^{77}Se NMR spectrum, δ_{Se}, ppm: 294.7. Mass spectrum, *m/z* (I_{rel}, %): 210 (6) $[M]^{+\cdot}$, 129 (25) $[M - SeH]^+$, 117 (5) $[M - CHSe]^+$, 92 (2) $[C_7H_8]^+$, 91 (55) $[C_7H_7]^+$, 65 (9) $[C_5H_5]^+$.

1-Benzylselanyl-1-propyne (Vb). ^1H NMR spectrum, δ_C, ppm (*J*, Hz): 1.89 s (3H, Me), 3.91 s (2H, Se$\underline{CH_2}$Ph, $^2J_{H–C–Se}$ 13.6 Hz), 7.14–7.23 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 5.18 (Me), 32.16 (Se$\underline{C}H_2$Ph, $^1J_{C–Se}$ 54.3 Hz), 58.32 (Se$\underline{C}$≡C), 97.38 (Me–$\underline{C}$≡), 127.17 (C_p), 128.31, 128.71 (C_o, C_m), 137.65 (C_i). ^{77}Se NMR spectrum, δ_{Se}, ppm: 241.7. Mass

spectrum, *m/z* (I_{rel}, %): 210 (8) $[M]^{+\cdot}$, 129 (25) $[M - SeH]^+$, 92 (3) $[C_7H_8]^{+\cdot}$, 91 (54) $[C_7H_7]^+$, 65 (9) $[C_5H_5]^+$.

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