Reactions of Dichloroethenes with Sulfur in the System Hydrazine Hydrate–KOH

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Abstract—Vinylidene chloride and 1,2-dichloroethene react with sulfur in the system hydrazine hydrate–KOH with the formation of polyvinylenesulfide oligomer of molecular mass 1750–6120 Da containing also vinylenehydrazine and vinylenechloride links in the *Z*-configuration, and 1,4-dithiine in the yield up to 46%. In aqueous-hydrazine layer polysulfide anions S_n^{2-} (n = 1-4), mainly S_2^{2-} were found. The mechanism of formation of oligomers and 1,4-dithiine is suggested, which includes in the first stage dehydrochlorination of dichloroethenes and generation of chloroacetylene.

Keywords: 1,1(1,2)-dichloroethens, sulfur, unsaturated sulfur-containing oligomers, 1,4-dithiine, hydrazine hydrate

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1,1-Dichloroethene (vinylidene chloride) 1a and 1,2dichloroethene 1b are commercially available monomers and reagents [1, 2] promising for the design of miscellaneous unsaturated structures. Preparation of unsaturated sulfides on their basis opens wide possibilities for the synthesis of polyfunctional ligands for complex formation with transition metal ions, and elaboration on their basis of new catalysts for homogeneous catalysis, promising extractants, and analytical reagents [3]. However, there are no published data on direct reactions of dichloroethenes 1a, 1b with elemental sulfur. Only photochemical reactions of sulfur with 1,2-dichloroethene 1b leading to dichlorothiirane are known [4]. More effectively with compounds 1a, 1b react sulfide anions S^{2-} . Thus, by the reaction of Na₂S·9H₂O with Z-1,2-dichloroethene (Z-**1b**) in acetonitrile during 46 h a mixture of cyclic products (unsaturated thiacrown ethers) was obtained, containing from 6 to 27 atoms in the ring (and, respectively, from 2 to 9 sulfur atoms) in the total yield of 18%, the smallest-size cycle, 1,4-dithiine, being formed in 8.6% isolated yield [5]. The addition of 0.4 equiv. of phase transfer catalyst, 15-crown-5, to the reaction mixture allowed in the same conditions increasing the total yield of cyclic products to 40%

(yield of 1,4-dithiine 17.6%) [5]. The reaction of Na₂S·9H₂O with vinylidene chloride **1a** in the same system at 80°C afforded a mixture of thiacrown ethers in total yield of 40% (yield of 1,4-dithiine 13%) [6]. In [5, 6] the formation of solid insoluble product was also noted, whose structure was not investigated. In spite of relatively low yield of 1,4-dithiine and total yield of cyclic products, these data demonstrate principal possibility of participation of S^{2–} nucleophile in reactions with dichloroethenes **1a**, **1b**.

In the present work we have studied the possibility of synthesis of organosulfur compounds from dichlorides **1a**, **1b** by the reaction with sulfur in a basic reductive system hydrazine hydrate–KOH. In this system, with the molar ratio KOH : S > 2 a selective formation of sulfide anions is observed [7] (Scheme 1).

The formed solution of K_2S in hydrazine hydrate was used in subsequent transformations without isolation in the individual state. Selective reduction of sulfur along Scheme 1 and participation of S^{2-} anions

Scheme 1.

$$1/4S_8 + 4KOH + N_2H_4 \cdot H_2O \rightarrow 2K_2S + N_2 + 5H_2O$$





in subsequent reactions was proved by an independent experiment, the reaction of the obtained solution with 1-bromopropane leading to dipropylsulfide 2 in 85% yield (Scheme 2). The corresponding dipropyldisulfide or -trisulfide were not detected in the reaction mixture.

We have found that sulfur, activated along Scheme 1 to anion S^{2-} , readily reacts with dichlorides **1a** and **1b** at room temperature with the formation of several products. From the reaction mixture oligomer **3** precipitated in 26–75% yield. After its separation 1,4-dithiine **4** was obtained by extraction of the filtrate (yield 16–31% of compound **1a** and 14–46% of compound **1b**). The remaining aqueous-hydrazine layer contained polysulfide anions S_2^{2-} , S_3^{2-} and even traces of S_4^{2-} (Scheme 3).

Oligomer product **3** was obtained as a beige powder. Its yield (calculated on the taken sulfur) varies in a wide range and, apparently, depends not only on the ratio of the reagents but also on the conditions of the experiment (temperature, rate of addition of dichloroethene **1**, intensity of stirring, etc.). A high yield of oligomer **3** (68%) was observed for the molar ratio **1a** : S = 6 : 1 and addition of **1a** in the course of 7 h at 15–20°C. The maximum yield of the oligomer (75%) was obtained when cooling the reaction mixture to 10–15°C and the ratio **1a** : S = 2.5 : 1 (Table 1).

The data of elemental analysis show that all isolated samples of oligomers, apart from carbon, hydrogen and sulfur, contain nitrogen (1-8%) and residual chlorine (from the reaction with **1a**, 1.2-3.8%,

Exp. no.	Amou	int of used reagents,	mmol	Yield, % (calculated on sulfur)						
	S	КОН	1a (1b)	3	4	5 ^a				
1	112.3	449.2	224.6	42	23	16				
2 ^b	25.0	100.0	150.0	68	30	Traces				
3°	31.2	124.8	78.0	75	23	Traces				
4	56.0	224.0	168.0	45	31	16				
5	56.0	224.0	56.0	26	22	24				
6	125.0	500.0	250.0	48	16	14				
7	28.0	280.7	56.1	28	32	17				
8	112.3	449.2	(112.3)	33	14	26				
9	31.2	124.8	(93.6)	44	46	Traces				

Table 1. Yields of the reaction products (Scheme 4, 23°C, 5 h)

^a Yields of polysulfides estimated from the amount of the formed dipropyl polysulfides calculated on Pr₂S₂. ^b Temperature of reaction 15–20°C, time 1 h. ^c Temperature of reaction 10–15°C, time 8.5 h.

Table 2. Elemental analysis data for oligomers 3

	Composition of oligomer 3												Molecular mass		
Exp. no. ^a	found, %					calculated, %							d CI)	ated	
	С	Н	S	Cl	N	formula	С	Н	S	Cl	N	x	У	foun (from	calcula
1	41.34	3.75	44.52	1.20	8.71	$C_{170}H_{196}S_{67}N_{30}Cl_2$	41.85	4.05	44.03	1.45	8.61	66	15	4730	4879
2	40.47	3.88	45.80	5.95	2.44	$C_{112}H_{126}S_{48}N_{14}Cl_2$	41.03	3.87	46.95	5.98	2.16	47	7	3450	3278
3	40.68	4.01	50.38	1.30	4.91	$C_{206}H_{230}S_{90}N_{24}Cl_2$	41.24	3.86	48.10	1.18	5.60	89	12	5460	5999
4	40.87	3.93	43.93	3.27	6.74	$C_{76}H_{88}S_{31}N_{12}Cl_2$	40.85	3.97	44.49	3.17	7.52	30	6	2260	2235
5	41.29	3.76	49.13	3.16	1.26	$C_{186}H_{198}S_{86}N_{12}Cl_2$	41.14	3.68	50.78	3.09	1.30	85	6	5730	5430
6	40.27	3.89	46.53	6.01	1.16	$C_{206}H_{232}S_{89}N_{26}Cl_2$	41.26	3.90	47.59	6.07	1.18	88	13	6120	5997
7	39.44	3.56	44.86	1.28	7.52	$C_{190}H_{222}S_{78}N_{32}Cl_2$	41.30	4.05	45.26	1.28	8.11	77	16	5966	5526
8	40.98	3.31	49.49	1.90	4.51	$C_{50}H_{52}S_{23}N_2Cl_2$	40.32	3.52	49.52	1.88	4.76	22	1	1753	1489
9	41.00	3.93	43.93	3.27	6.74	$C_{64}H_{76}S_{25}N_{12}Cl_2$	40.76	4.06	42.51	3.65	8.91	24	6	2030	1886

^a Corresponds to the exp. no. in Table 1.

with **1b**, 3.2–4.5%). Assuming that the chlorine atoms are located at the ends of macromolecule of the formed oligomer as –CH=CHCl fragments, we can calculate the average molecular mass of the oligomeric product (cf. [8], see Table 2). For the oligomer from vinylidene chloride **1a** it is 2260–6120 Da, and for 1,2-dichloroethene **1b**, 1750–2000 Da. It can be assumed that the principal motif of the oligomer macromolecules forming the polymeric chain is vinylene – CH=CH– to the ends of which are attached the atoms of sulfur, nitrogen and chlorine. Based on this, the structure of the oligomer can be represented as follows: ClCH=CH–S–(CH=CH–S)_x–(CH=CHNHNH–)_vCH=CHCl

In the literature, the synthesis and properties of polyvinylene sulfides were described having the *E* and *Z*-configuration of vinylene fragments by the reaction of polycondensation of sodium *E* or *Z*-ethenedithiolate, respectively, with the *E* and *Z*-1,2-dichloroethene **1b** in DMSO at 40°C [9]. The IR spectra of polyvinylene sulfides with different configuration of the vinylene fragment are substantially different; in particular, in the spectrum of the *E*-isomer an intense band at 910 cm⁻¹ is present δ (=CH), which is lacking for the polymer with the *Z*-configuration [9]. In the IR spectra of all samples of oligomer **3** the absorption in this region is absent, which is indicative of the *Z*-configuration of the vinylene links in the chain. The presence in the IR spectra of oligomers **3** the bands at

3440 cm⁻¹ (v_{NH} , single wide band) and 1615–1625 cm⁻¹ (δ_{NH}) allows assuming the presence of –NH–NH– fragments attached to the double bond in the structure of the oligomer [10] that corresponds to the structure of the oligomer given above.

1,4-Dithiine **4** formed in the course of the reaction (Scheme 3) was the subject of both synthetic and theoretical studies [11, 12]. The earlier elaborated methods for its synthesis, as a rule, were based on the use of difficultly accessible reagents [11]. Thus, it was synthesized in 47% yield along with a unidentified product by the reaction of Z-1,2-dichloroethene with sodium Z-1,2-ethenedithiolate in dry DMSO at 20°C [13]. We have used more available reagents and, besides, product **4** is easily isolated by extraction of the aqueous-hydrazine layer by diethyl ether after separation of oligomer **3**.

The formation of polysulfide anions 5 in the aqueous-hydrazine solution remaining after separation of products 3 and 4 was proved by their reactions of alkylation with 1-bromopropane with identification of the products of alkylation (Scheme 4).

Using the methods of NMR spectroscopy and chromatomass spectrometry, we identified dipropylpoly-





sulfides with n = 1-4. The major product of alkylation was dipropyldisulfide. From ¹H NMR data, the average ratio of products $Pr_2S : Pr_2S_2 : Pr_2S_3 : Pr_2S_4$ was 1 : 3 : 0.5 : traces.

The formation of similar products (3-5) when using vinylidene chloride 1a and the two isomers of 1,2dichloroethene 1b is indicative of the same mechanism of their transformations in the system S/N₂H₄·H₂O-KOH. We believe that the first step of these transformations is dehydrochlorination of compounds 1a, 1b under the action of alkali resulting in the formation of chloroacetylene 6 (Scheme 5). Baseinduced elimination of HCl from dichloroderivatives of the type 1a, 1b with the formation of chloroacetylenes is well known [2, 14]. An indirect confirmation of this in our case was, first, emergence of flashes at the outlet of condenser (cf. [2]) when performing the reaction of vinylidene chloride 1a with the system N₂H₄·H₂O/KOH without introduction of sulfur (blank experiment). Another indication was the absence of reaction of compound 1a with sulfur when activating it by the system hydrazine hydrate-monoethanolamine, which is also used in the synthesis of organosulfur compounds [7, 15] but has much lower basicity, apparently, not sufficient for dehydrochlorination of dichloroethenes 1a, 1b. Chloroacetylene 6 is a highly reactive species [14] where the chlorine atoms are easily replaced by sulfide anions or by excess hydrazine (Scheme 5).

The formation of macromolecules of oligomer **3**, apparently, proceeds with the participation of chloroacetylene **6**, ethynethiolate anions **7**, and substituted hydrazines **9** by addition of thiolate anions **7** to the triple bond of intermediates **6** and **9**. This reaction proceeds as the *trans*-addition with the formation of Z-adducts [16]. The addition of nitrogen-containing nucleophiles and HO⁻ anions to the triple bond occurs, as a rule, in the presence of catalysts, IIB group metal compounds [17] and, therefore, is hardly probable in our case. The earlier shown formation of macrocyclic structures [5, 6] can be prevented by the presence of vinylenehydrazine fragments-CH=CH-NH-NH- in the formed oligomer chain. Sun and Yang [6] suggested the mechanism of formation of unsaturated thia-crown ethers from vinylidene chloride and sodium sulfide, whose key step was substitution of chlorine atoms by NaS⁻ anions with the formation of sodium 1,1-ethenedithiolate, which further isomerizes to the 1,2-isomer. However, this mechanism seems doubtful because it is based on nontrivial and not evident assumptions about the ease of nucleophilic substitution of chlorine atoms at the double bond and about the ready stereoselective migration of the NaS⁻ group in ethenedithiolates.

The most probable route of the formation of 1,4dithiine **4** includes preliminary formation of diethynylsulfide **10**, which suffers cyclization involving sulfide anions (Scheme 6).

The transformation $10 \rightarrow 4$ by the reaction of compound 10 with sodium sulfide in the mixture of liquid ammonia and methanol (2 : 1) at -30°C in 68% yield was described in the literature [18].

In the reaction by Scheme 1 sulfur is reduced to sulfide anions, but in the aqueous-hydrazine layer remaining after the separation of products **3** and **4** anions S_2^{2-} , S_3^{2-} , and even S_4^{2-} are present. Their formation can be due to the oxidation of sulfide anions. The oxidation process with participation of air oxygen is low probable, because polysulfides S_n^{2-} are formed also in an inert atmosphere (in argon), as well as because of the presence in the reaction mixture of a strong reducing agent, hydrazine, which is readily oxidized by oxygen to diimide in alkaline medium [19]. It is known [20] that with lowering pH of the solution the reaction in which hydrazine acts as an oxidant becomes thermodynamically possible. The



Scheme 7.

$$\begin{split} N_2H_4 + 2S^{2-} + 2H_2O &\rightarrow 2NH_3 + S_2^{2-} + 2OH^-, \\ N_2H_4 + S^{2-} + S_2^{2-} + 2H_2O &\rightarrow 2NH_3 + S_3^{2-} + 2OH^-, \\ N_2H_4 + 2S_2^{2-} + 2H_2O &\rightarrow 2NH_3 + S_4^{2-} + 2OH^-. \end{split}$$

possibility of oxidation of some organic and inorganic sulfur compounds by hydrazine was mentioned in the literature [21–23]. Possibly, due to lowering pH of the medium due to dehydrochlorination of dichloroethenes **1a** and **1b** the reactions depicted in Scheme 7 become viable.

Anions S_n^{2-} (n = 2-4) do not enter the reaction with chloroacetylene **6** or ethyne thiolate **7** and remain in aqueous–hydrazine solution.

Therefore, the occurrence of parallel reactions shown in Schemes 5-7 does allow attaining high yields of dithiine 4. Variations of the yield are determined by the ratio of the rates of oligomer 3 formation and that of 1,4-dithiine 4. In turn, the occurrence of these reactions depends on a large number of factors: the nature of dichloroethene, the temperature of the process, the rate of addition of the reagents, hydrodynamic conditions, etc. Nevertheless, the availability of the reagents, mild reaction conditions, and the ease of isolation of 1,4-dithiine allow considering the proposed method of its preparation quite suitable for synthetic purposes.

EXPERIMENTAL

IR spectra were taken on a Bruker IFS–25 spectrometer in KBr (for oligomers) or in thin layer. ¹H, ¹³C NMR spectra were registered on a Bruker DPX-400 spectrometer (working frequencies 400.13 and 100.62 MHz respectively) in CDCl₃, chemical shifts were measured with respect to TMS. Mass spectra were measured on a Shimadzu GCMS-QP505A chromatomass spectrometer (column SPB-5, 60000×0.25 mm), quadruple mass analyzer, electronic ionization, 70 eV, temperature of ionic source 190°C, range of detected masses 34–650 Da.

Dipropylsulfide (2). To a solution containing 26.0 g (0.5 mol) of KOH in 110 mL of hydrazine hydrate, 4.0 g (0.125 mol) of powdered sulfur was added in portions at stirring at 70–85°C. The obtained mixture was stirred for 2 h at 85–90°C, cooled to 23°C, and added dropwise to 30.75 g (0.25 mol) of 1-bromopropane. The reaction mixture was stirred for 5 h at

40–50°C, extracted with ether (5×50 mL). The extract was washed with water, dried over MgSO₄ and the solvent was removed. Yield 12.5 g (85%). ¹H NMR spectrum, δ , ppm: 0.97 t (3H, CH₃, ³*J* = 7.4 Hz), 1.59 sext (2H, CCH₂C, ³*J* = 7.4 Hz), 2.47 t (2H, CH₂S, ³*J* = 7.4 Hz). ¹³C NMR spectrum, δ , ppm: 13.50 (CH₃), 23.05 (C<u>C</u>H₂C), 34.21 (CH₂S). Mass spectrum, *m/z*: 118 [*M*⁺].

General procedure for the reaction of dichloroethenes 1a, 1b with sulfur in the system hydrazine hvdrate-alkali. To the solution of KOH in hvdrazine hydrate the required amount of powdered sulfur (see Table 1) was added at stirring. The mixture was stirred for 2 h at 80-85°C, cooled to 23°C and dichloroethene 1a or 1b was added at continued stirring at 23°C for another 3-6 h (Table 1 shows total time of addition and stirring). The formed precipitate of oligomer 3 was filtered off, washed with water, ether and dried. The vields calculated from the sulfur content in the product, molecular mass, composition and the data of elemental analysis for oligomers 3 are shown in Table 2. On heating to 150-175°C, oligomers 3 become dark and decompose. IR spectrum, v, cm⁻¹: 3442, 3024, 1619, 1559, 1384, 1271, 1145, 1013, 805, 771, 640, 539. ¹H NMR spectrum (DMSO- d_6), δ , ppm: 6.68 d $(=CH-Cl, {}^{3}J = 4.3 Hz), 6.52-6.66 m (=CH-S, =CH-N).$

1,4-Dithiine (4). The reaction mixture after separation of oligomer **3** was extracted with ether (3×50 mL), combined extracts were dried over MgSO₄. After removal of the solvent, the residue was practically pure 1,4-dithiine **4**. The yields calculated on sulfur taken for the reaction are given in Table 1. The product is a red liquid of bp 78°C (20 c) {68°C (12 mmHg) [18], 77°C (17 mmHg) [24]}. ¹H and ¹³C NMR spectra were given earlier [25].

Dipropylpolysulfides. Aqueous-hydrazine layer of green or green-yellow color remaining after separation of oligomer **3** and extraction of 1,4-dithiine **4** was treated with excess 1-bromopropane at 40–50°C. After addition of propylbromide the reaction mixture discolored. By extraction the mixture with ether a mixture of dipropylpolysulfides was isolated. ¹H NMR spectrum, δ , ppm: 0.82–1.02 group of triplets (3H, CH₃), 1.50–1.80 group of multiplets (2H, CCH₂C), 2.40–2.73 group of triplets (2H, CH₂S). ¹³C NMR spectrum, δ_{C} , ppm: 11.58, 12.94, 13.5, and 13.34 (CH₃); 20.14, 22.32, 22.53 and 22.85 (C<u>C</u>H₂C); 29.35, 33.98, 35.27 and 40.95 (CH₂S). Mass spectrum, *m/z*: 118 [Pr₂S], 150 [Pr₂S₂], 182 [Pr₂S₃], 185 [*M* – C₂H₅]⁺.

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