

Synthesis of Polydentate Chalcogen-Containing Ligands Using the System Hydrazine Hydrate–Base

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Abstract—The reaction of dichlorodiethyl ether or dichlorodiethylamine hydrochloride with potassium or ethanolammonium dichalcogenides prepared in situ from elemental sulfur or selenium in the system hydrazine hydrate–alkali results in oligomeric dichalcogenides, whose further splitting at the chalcogen–chalcogen bond in the same system and subsequent alkylation affords bis(organylsulfanyl)- or bis(organylselanyl)-substituted derivatives of diethyl ether or diethylamine. Ligands with aryl substituents at the chalcogens have been prepared in 48–59% yield by splitting of diaryldichalcogenides in the system hydrazine hydrate–alkali and subsequent reaction with dichlorodiethyl ether or dichlorodiethylamine. A ditopic tetradentate ligand has been synthesized by the use of dichlorodiethylformal.

Keywords: sulfur, selenium, dichalcogenides, polydentate ligands, hydrazine hydrate

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The chalcogen atoms in organic molecules are capable of formation of strong coordination bonds with heavy metal ions [1]. Especially stable chelate complexes are formed when two or more donor centers are present in the ligand [1]. Complexes of this type are of interest for coordination chemistry [2, 3], for the design of analytical reagents [4] and selective sensors [5–7] and are used for the analysis and investigation of coordination compounds of noble metals [8]. Especially promising is the use of metal complexes with chalcogen-containing ligands in homogeneous catalysis [9, 10]. Thus, palladium complexes with organochalcogen ligands were proposed as catalysts in the Suzuki reaction [11]. An important feature of complexes used in homogeneous catalysis is their ability to reduce the coordination number of the complex-forming metal [12]. As a result, coordinatively unsaturated complexes are formed, which can interact with the molecules of the reagents to continue the catalytic cycle. Since the chalcogen atoms, as soft bases, form strong bonds with many transition metal ions (soft acids), for the formation of

labile metal–ligand bonds the presence of hard donor centers in the structure of the latter is necessary, for example, the oxygen or nitrogen atoms.

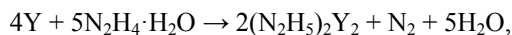
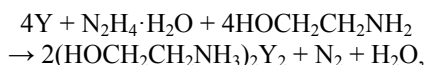
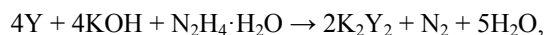
These requirements are fulfilled in polydentate ligands containing between the donor atoms the chain of two carbon atoms, which favors the formation of the energetically stable five-membered chelate cycles upon the formation of complexes with metals. Within this group of ligands the derivatives of diethyl ether and diethylamine containing additional chalcogenide substituents are intensively studied [3, 4, 6–11]. The data on the synthesis of oxa- and aza-substituted bischalcogenide ligands of this type are scarce. The known methods are mostly based on the use of 2,2'-dichloroethyl ether (chlorex) **1a** and the salts of 2,2'-dichlorodiethylamine **1b** as the reagents. A multistep synthesis of bis(2-methylsulfanyl)diethylether in 61% yield from chlorex via the steps of formation of the double isothiuronium salt with its subsequent transformation to the corresponding dithiol and methylation of the latter with methyl iodide has been described

[13]. For the synthesis of dibenzylsulfanyl derivative instead of chlorex diethylene glycol ditosylate is used, whose reaction with benzylthiuronium chloride in the presence of solid alkali and a phase transfer catalyst gives the target product in 30–55% yield [14]. Dichalcogen-substituted derivatives of diethylamine were prepared in 69–89% yield by the reaction of dichlorodiethylamine with thiulates or selenolates of alkali metals [5, 6, 10, 11, 15–21]. For the synthesis of bisphenylselenanyl derivative of 3-azapentylene diphenyldiselenide was used, which was reduced to phenylselenolate by the reaction with NaBH₄ [6]. All reactions with thiulates and selenolates were carried out in inert atmosphere. The above brief inventory of the methods of synthesis of bischalcogenyl-substituted derivatives of diethyl ether and diethylamine shows that they are laborious and that elaboration of synthetically efficient methods for preparation of polydentate ligands containing oxa- or aza-groups in the molecule, as well as with two organysulfanyl or -selenanyl moieties is actual.

In the present work two methods of the synthesis of 1,5-diorganylchalcogenide-3-oxa(3-aza)pentylenes **2a–2j** are worked out based on the use of chlorex **1a** or dichlorodiethylamine hydrochloride **1b**, elemental sulfur, selenium or diorganylchalcogenides in the system hydrazine hydrate–base.

Elemental chalcogens and diorganylchalcogenides in the system hydrazine hydrate–base are reduced to polychalcogenides or potassium organylchalcogenolates [22]. The reduction of sulfur or selenium to disulfide or diselenide anion proceeds rather selectively using either the system hydrazine hydrate–monoethanolamine (molar ratio N₂H₄·H₂O : H₂NCH₂CH₂OH = 10 : 1) or the system hydrazine hydrate–KOH with the molar ratio Y : KOH = 1 : 1 [22] (Scheme 1).

Scheme 1.



Y = S, Se.

The obtained anionic forms of chalcogens were used in further syntheses without isolation as individuals.

The reductive splitting of diorganylchalcogenides proceeds by Scheme 2. For effective proceeding of the

reaction an excess of alkali was used, R₂Y₂ : KOH = 1 : 5. Chalcogenolates **3a–3c** either were not isolated as individuals.

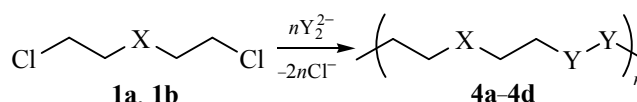
Scheme 2.



R = Ph, Y = S (**a**), Se (**b**); R = Bn, Y = S (**c**).

Alkylchalcogenide tridentate ligands **2a–2f** were obtained using active forms of chalcogens prepared as in Scheme 1. Polycondensation of dihaloderivatives **1a** and **1b** with dichalcogenide anions gives oligomeric dichalcogenides **4a–4d** (Scheme 3).

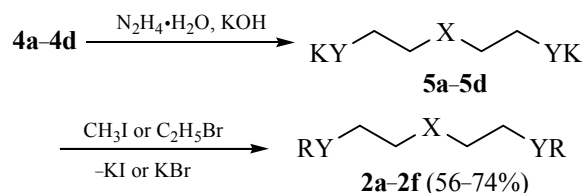
Scheme 3.



1, X = O (**a**), NH (**b**); **4**, X = O, Y = S (**a**), Se (**b**); X = Nh, Y = S (**c**), Se (**d**).

The reductive splitting of oligomers **4a–4d** at the Y–Y bond using the system hydrazine hydrate–KOH results in the formation of dichalcogenolates **5a–5d**, which were further alkylated with alkyl halides affording the target products **2a–2f** (Scheme 4).

Scheme 4.



5, X = O, Y = S (**a**), Se (**b**); X = NH, Y = S (**c**), Se (**d**); **2**, X = O, Y = S, R = CH₃ (**a**), C₂H₅ (**b**), Y = Se, R = CH₃ (**c**), C₂H₅ (**d**); X = NH, Y = S, R = CH₃ (**e**), Y = Se, R = CH₃ (**f**).

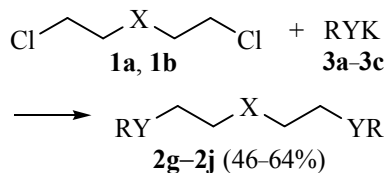
Oligomeric dichalcogenides **4a, 4b** we obtained earlier [23, 24] when studying the reactions of dielectrophiles (including chlorex) with elemental chalcogens in the system hydrazine hydrate–base. Oligomer **4a** was obtained in 62% yield and had the average molecular mass equal to 1770, as defined by the method of terminal groups [23]. Oligomer **4b** was isolated in 75% yield and had molecular mass of 1730 [24]. The structure of the obtained oligomers was proved by the method of reductive splitting. The synthesis and reductive splitting of oligomers **4c, 4d** is performed for the first time. The compounds are viscous black substances. Oligomer **4c** was obtained in

Table 1. Yields, boiling points, elemental analysis, and IR spectra of compounds **2a–2j**

Comp. no.	Yield, %	bp, °C (mmHg)	Found, %				Formula	Calculated, %				IR spectrum, ν , cm^{-1}
			C	H	N	Y		C	H	N	Y	
2a	60	87–90 (3) {97 (4) [13]}	43.19	8.51	–	38.89	$\text{C}_6\text{H}_{14}\text{OS}_2$	43.33	8.49	–	38.56	2970, 2916, 2860
2b	67	93–96 (2) [23]	49.54	9.27	–	32.47	$\text{C}_8\text{H}_{18}\text{OS}_2$	49.44	9.34	–	32.99	2985, 2936, 2914, 2886, 2854
2c	64	96–97 (1.5) [24]	27.60	5.39	–	60.81	$\text{C}_6\text{H}_{14}\text{OSe}_2$	27.71	5.42	–	60.72	2994, 2970, 2925, 2858
2d	64	112–116 (1.5)	33.59	6.12	–	55.00	$\text{C}_8\text{H}_{18}\text{OSe}_2$	33.35	6.30	–	54.80	2973, 2956, 2945, 2865
2e	74	85–87 (1.5) [3]	43.60	9.00	8.32	39.08	$\text{C}_6\text{H}_{15}\text{NS}_2$	43.59	9.15	8.47	38.79	3283, 2962, 2921, 2815
2f	56	119–122 (2)	27.91	5.87	5.56	60.66	$\text{C}_6\text{H}_{15}\text{NSe}_2$	27.81	5.83	5.41	60.95	3287, 2994, 2962, 2922, 2820
2g	57	203–206 (1.5)	65.80	6.02	–	21.15	$\text{C}_{16}\text{H}_{18}\text{OS}_2$	66.17	6.25	–	22.08	3072, 3056, 3019, 2958, 2925, 2864
2h	58	120 (0.02) [3, 14]	67.64	6.97	–	19.69	$\text{C}_{18}\text{H}_{22}\text{OS}_2$	67.88	6.96	–	20.13	3083, 3060, 3027, 2952, 2917, 2859
2i	64	–	50.45	4.51	–	41.28	$\text{C}_{16}\text{H}_{18}\text{OSe}_2$	50.02	4.72	–	41.10	3069, 3054, 3015, 2978, 2934, 2860
2j	46	213–215 (2) [5]	66.37	6.82	4.93	21.88	$\text{C}_{16}\text{H}_{19}\text{NS}_2$	66.39	6.62	4.84	22.15	3300, 3056, 3018, 2922, 2832

70% yield with molecular mass 1460, oligomer **4d** in 96% yield and molecular mass 6400. Therefore, we have realized the preparative synthesis of compounds **2a–2f** via the intermediate dichalcogenide oligomers.

For the synthesis of arylchalcogenide and benzylchalcogenide ligands **2g–2j** we have used an alternative approach based on the reaction of chalcogenolates **3a–3c** with chlorex **1a** or with dichlorodiethylamine hydrochloride **1b** (Scheme 5). The yields, data of elemental analysis and IR spectroscopy of compounds **2a–2j** are given in Table 1, the data of ^1H and ^{13}C NMR spectra, in Table 2.

Scheme 5.

1, X = O (**a**), NH (**b**); **2**, X = O, Y = S, R = Ph (**g**), Bn (**h**), Y = Se, R = Ph (**i**), X = NH, Y = S, R = Ph (**j**); **3**, R = Ph, Y = S (**a**), Se (**b**); R = Bn, Y = S (**c**).

Using the approach represented in Schemes 1, 3, and 4, we have first synthesized a ditopic tetradentate ligand, 1,10-dithia-5,7-dioxadecane **5** by the reaction of 2,2-dichlorodiethylformal **6** and sulfur preliminary reduced in the system hydrazine hydrate–monoethanolamine via the stage of formation of oligomeric disulfide **7** (Scheme 6). Oligomer **7** was prepared in 94% yield as a highly viscous gray substance; its

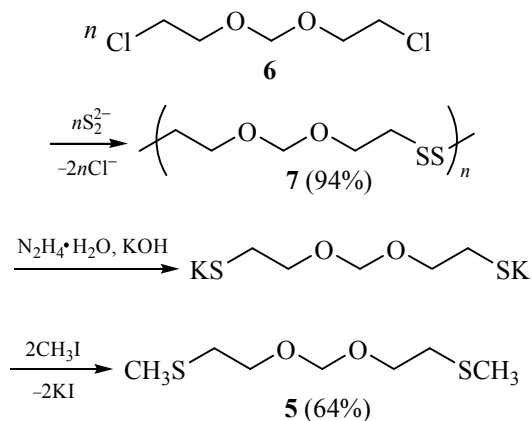
Scheme 6.

Table 2. ^1H , ^{13}C , ^{77}Se NMR spectra of compounds **2a–2j**, chemical shifts, δ , ppm

Comp. no.	δ_{H} , ppm				δ_{C} , ppm			δ_{Se} , ppm
	XCH ₂	YCH ₂	NH	R	XCH ₂	YCH ₂	R	
2a	3.64 t $^3J = 6.8$ Hz	2.67 t	—	2.14 s (3H, CH ₃)	70.15	33.40	15.90 (CH ₃)	—
2b	3.62 t $^3J = 7.0$ Hz	2.71 t	—	2.57 q (2H, CH ₂), 2.49 t (3H, CH ₃ , $^3J = 7.4$ Hz)	70.54	30.87	26.26 (CH ₂), 14.73 (CH ₃)	—
2c	3.69 t $^3J = 7.0$, $^2J_{\text{SeH}} = 12.5$ Hz	2.70 t	—	2.03 s (3H, CH ₃)	70.68	24.07	4.48 d (CH ₃ , $^1J_{\text{SeC}} = 61.9$ Hz)	60.24
2d	3.68 t $^3J = 7.3$ Hz	2.72 t	—	2.60 q (2H, CH ₂ , $^3J = 7.5$ Hz), 1.39 t (3H, CH ₃ , $^3J = 7.5$ Hz)	71.00	22.06 d ($^1J_{\text{SeC}} = 65.0$ Hz)	17.53 d (CH ₂ , $^1J_{\text{SeC}} = 58.5$ Hz), 15.73 (CH ₃)	175.16
2e	2.84 t $^3J = 6.6$ Hz	2.66 t	1.56 br.s	2.10 s (3H, CH ₃)	47.55	34.33	15.27 (CH ₃)	—
2f	2.83 t $^3J = 6.7$ Hz	2.64 t	1.75 br.s	1.94 s (3H, CH ₃)	48.39	25.79 d ($^1J_{\text{SeC}} = 60.8$ Hz)	3.92 d (CH ₂ , $^1J_{\text{SeC}} = 62.3$ Hz)	49.4
2g	3.60 t $^3J = 6.9$ Hz	3.06 t	—	7.13–7.33 m (5H, C ₆ H ₅)	69.69	33.23	135.92 (C _i), 129.53, 128.97 (C _o , C _m), 126.25 (C _p)	—
2h	3.51 t $^3J = 6.7$ Hz	2.57 t	—	3.71 s (2H, CH ₂ Ph), 7.21–7.30 m (5H, C ₆ H ₅)	70.40	30.60	138.32 (C _i), 128.88, 128.46 (C _o , C _m), 126.98 (C _p)	—
2i	3.64 t $^3J = 7.1$ Hz	3.00 t	—	7.20–7.50 m (5H, C ₆ H ₅)	70.40	26.59 d ($^1J_{\text{SeC}} = 65.0$ Hz)	129.44 (C _i), 132.58, 128.98 (C _o , C _m), 126.90 (C _p)	270.2
2j	2.61 t $^3J = 6.4$ Hz	3.03 t	1.80 br.s	7.33 m (2H, H _o), 7.24 m (2H, H _m), 7.17 m (1H, H _p)	47.73	34.19	135.28 (C _i), 129.78, 128.92 (C _o , C _m), 126.26 (C _p)	—

molecular mass was about 1000. The splitting of oligomer **7** at the S–S bonds and the subsequent alkylation results in the formation of compound **5**.

Therefore, the use of basic-reductive systems on the basis of hydrazine hydrate allows synthesizing dichalcogenide polydentate ligands of linear structure, promising reagents for the design of chelate complexes with participation of heavy metal ions.

EXPERIMENTAL

IR spectra were recorded on a Bruker IFS-25 spectrophotometer from thin layers. ^1H , ^{13}C , ^{77}Se NMR spectra were registered on a Bruker DPX-400 spectrometer (400.13, 100.62, and 76.31 MHz respectively) in DMSO-*d*₆ or CDCl₃ solution, internal reference TMS (^1H , ^{13}C) and Me₂Se (^{77}Se). Mass spectra were taken on a Shimadzu GCMS–QP5050A

chromatomass spectrometer (column SPB-5, 60000×0.25 mm, quadruple mass analyzer, electron ionization, 70 eV, temperature of ion source 190°C, the range of detected masses 34–650 Da). The purity of the products and the course of the reactions were monitored by GC on a LKhM 80-MD-2 chromatograph (column 2000×3 mm, liquid phase DC-550, 5% on Chromaton N-AW-HMDS, linear programming of the temperature with the heating rate 12 deg/min, gas carrier helium).

Oligomers **4a,b** were synthesized as described earlier [23, 24]. The oligomers were also prepared by the use of the system hydrazine hydrate–monoethanolamine [25].

Poly(3-azapentylene disulfide) (4c). To the solution of 1.26 g (0.0224 mol) of KOH and 1.1 mL of hydrazine hydrate in 7 mL of water 0.72 g (0.0224 mol) of elemental sulfur was added by portions, the reaction mass was stirred for 5 h at 85–90°C, cooled, 6 mL of 10% KOH and 2.0 g (0.0112 mol) of dichlorodiethylamine hydrochloride **1b** was added. The obtained mixture was stirred for 2 h at 60–65°C, cooled, the formed oligomer was separated by decanting, washed with water, ethanol, ether, dried in a vacuum to a constant mass. 1.13 g (75%) of oligomer **4c** was obtained as a viscous compound of black color. IR spectrum, ν , cm^{-1} : 3291, 2920, 2820, 1455, 1358, 1285, 1122, 1003, 752. Found, %: C 34.96, H 6.67, Cl 4.86, N 10.61, S 43.63. $\text{C}_{44}\text{H}_{98}\text{N}_{11}\text{Cl}_2\text{S}_{20}$. Calculated, %: C 35.41, H 6.57, Cl 4.76, N 10.33, S 42.92. The molecular mass determined by the residual chlorine was 1460, which corresponded to $n = 11$.

Poly(3-azapentylene diselenide) (4d) was prepared similarly. Yield 3.28 g (96%) of oligomer **4g** as a viscous mass of black color. IR spectrum, ν , cm^{-1} : 3283, 2921, 2815, 1455, 1407, 1356, 1261, 1221, 1179, 1116, 975, 797, 735, 643, 547. Found, %: C 20.61, H 4.11, Cl 1.11, N 5.90, Se 69.39. $\text{C}_{112}\text{H}_{252}\text{N}_{28}\text{Cl}_2\text{Se}_{54}$. Calculated, %: C 20.98, H 3.96, Cl 1.12, N 6.12, Se 68.94. The molecular mass determined by the residual chlorine was 6400, which corresponded to $n = 28$.

Compounds 2a–2f (general procedure). 20% KOH solution in hydrazine hydrate and the calculated amount of oligomer **4a–4d** (molar ratio $n : \text{KOH} = 1 : 5$) was stirred for 3.5 h at 85–90°C, the reaction mass was cooled to 25°C and MeI or EtBr was added dropwise [$n : \text{AlkHlg} = 1 : (2.2–3)$] and the mixture was stirred for 2.5 h at 30–35°C and 4 h at 25°C. The product was

extracted with ether (2×50 mL), the extract was dried over MgSO_4 , the solvent was removed, the residue was distilled in a vacuum.

Compounds 2g–2j (general procedure). 20% KOH solution in hydrazine hydrate and the calculated amount of diorganyl dichalcogenide R_2Y_2 ($\text{R}_2\text{Y}_2 : \text{KOH} = 1 : 5$) was stirred for 4 h at 85–90°C. the reaction mass was cooled to 60°C, 1 equivalent of chlorex **1a** or salt **1b** was added, the mixture was stirred for 5 h at 60–70°C, cooled to 25°C, the product was extracted with ether (2×50 mL), the extract was dried over MgSO_4 , the solvent was removed. Compounds **2g, 2j** were distilled in a vacuum, compounds **2h, 2i** were analyzed without distillation.

Poly(3,5-dioxaheptylenedisulfide) (7). 3 mL of hydrazine hydrate, 0.34 g (0.0056 mol) of ethanolamine, and 1.12 g (0.035 mol) of elemental sulfur was stirred for 2.5 h at 60–65°C. The reaction mass was cooled to 25°C, 3.0 g (0.0173 mol) of dichlorodiethylformal was added dropwise and the mixture was stirred for 4 h at 75–80°C. After cooling, oligomer **7** was separated by decanting as a viscous mass. Yield 2.7 g (94%). IR spectrum, ν , cm^{-1} : 2921, 2870, 2801, 1466, 1412, 1377, 1282, 1197, 1154, 1115, 1072, 1029, 951, 812, 791, 758, 666, 620, 477. ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm: 2.93 m (4H, CH_2S), 3.73 m (4H, CCH_2O), 4.66 s (2H, OCH_2O). ^{13}C NMR spectrum ($\text{DMSO}-d_6$), δ , ppm: 38.14 (CH_2S), 65.56 (CCH_2O), 94.62 (OCH_2O). Found, %: C 35.50, H 6.27, S 29.05, Cl 7.79. $\text{C}_{27.5}\text{H}_{55}\text{Cl}_2\text{S}_9$. Calculated, %: C 36.26, H 6.04, S 31.65, Cl 7.80. The molecular mass determined by the residual chlorine was 910, which corresponded to $n = 5–6$.

5,7-Dioxa-2,10-dithiaundecane (5). A mixture of 2.97 g (0.053 mol) of KOH, 13 mL of hydrazine hydrate, and 1.76 g (0.0106 mol) of oligomer **7** was stirred for 3 h at 85–90°C. The reaction mass was cooled to 25°C, 4.52 g (0.0318 mol) of methyl iodide was added dropwise, the mixture was stirred for 2 h at 30–35°C and 4 h at 25°C. The product was extracted with ether (2×50 mL), the extract was dried over MgSO_4 and evaporated. The residue was distilled in a vacuum. Yield 1.33 g (64%), colorless liquid, bp 102–105 (2 mmHg). IR spectrum, ν , cm^{-1} : 2918, 2873, 2792, 1463, 1428, 1380, 1287, 1203, 1155, 1112, 1074, 1027, 954, 775, 726, 698, 659, 618, 475. ^1H NMR spectrum (CDCl_3), δ , ppm: 2.14 s (6H, CH_3), 2.71 t (4H, 3J 6.6 Hz, CCH_2O), 4.72 s (2H, OCH_2O). ^{13}C NMR spectrum (CDCl_3), δ , ppm: 15.88 (CH_3),

33.85 (CH₂S), 66.88 (CCH₂O), 95.31 (OCH₂O). Mass-spectrum, *m/z*: 196 [M]⁺, 75 [CH₃SCH₂CH₂]⁺. Found, %: C 42.67, H 8.42, S 32.10. C₇H₁₆O₂S₂. Calculated, %: C 42.86, H 8.21, S 32.66.

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