Heteroatom Chemistry
Volume 16, Number 3, 2005

Sulfur-Containing Derivatives of 1,4-Naphthoquinone, Part 1: Disulfide Synthesis

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Received 7 December 2004; revised 5 February 2005

ABSTRACT: *Disulfides of 1,4-naphthoquinone were synthesized, and different methods of their synthesis were investigated. High yields and purity of disulfides were obtained from the oxidation of thiol derivatives. The latter were prepared in high yields and purity from isothiuronic salts. The obtained disulfides are synthones for compounds with a wide spectrum of biological activity.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:205–211, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20112

INTRODUCTION

Disulfides are found in natural products frequently. They are included as binding elements into secondary structure of fibers. It provides rather stable position of the peptide circuits, which formed at the expense of weaker binding forces, in particular of the hormones (somathostatin, insulin, vasopresin, oxytocin, calcitonin). Disulfide bonds are found in the structures of alkaloids (cassipurin, zherardin) and antibiotics. Disulfides are used as antiradiation preparations, nootropic psychostimulators, and as drugs for the treatment of the alcoholism. The compounds that have high biological activity (for example, gliotoxin and sporidesmin), which have antifungal, antibacterial, and antivirus activities, are represented among disulfides [1]. Some disulfides are widely used as accelerators of the rubbers' vulcanization [2].

Due to the lability of S—S bond, its ability to be broken under action of the nucleophilic and electrophilic reagents, disulfides are widely used in multistage organic synthesis.

Investigations during the last 10–15 years were dedicated to the further development of already known disulfides syntheses and to the search of new methods (synthesis of the base of alkylthiotrimethylsilanes, thiocyanates, sulfenylthiocyanates etc.).

The combination of disulfide bond and naphthoquinone fragment will allow different chemical conversions. Only bis-[2-amino-1,4-naphthoquinone]-3-disulfide (**4a**) is described in the literature at present [3]. We investigated various methods of synthesis of new disulfides from 3-chloro-, -thiolo-, and -thiocyano-1,4-naphthoquinone and unsubstituted 1,4-naphthoquinone.

RESULTS AND DISCUSSION

Interaction of 2-Amino-3-chloro-1,4-naphthoquinones with Sodium Disulfide

As an initial compound for synthesis of 2-amino-3-chloro-1,4-naphthoquinones **2** was used 2,3-dichloro-1,4-naphthoquinone (**1**). The preparation

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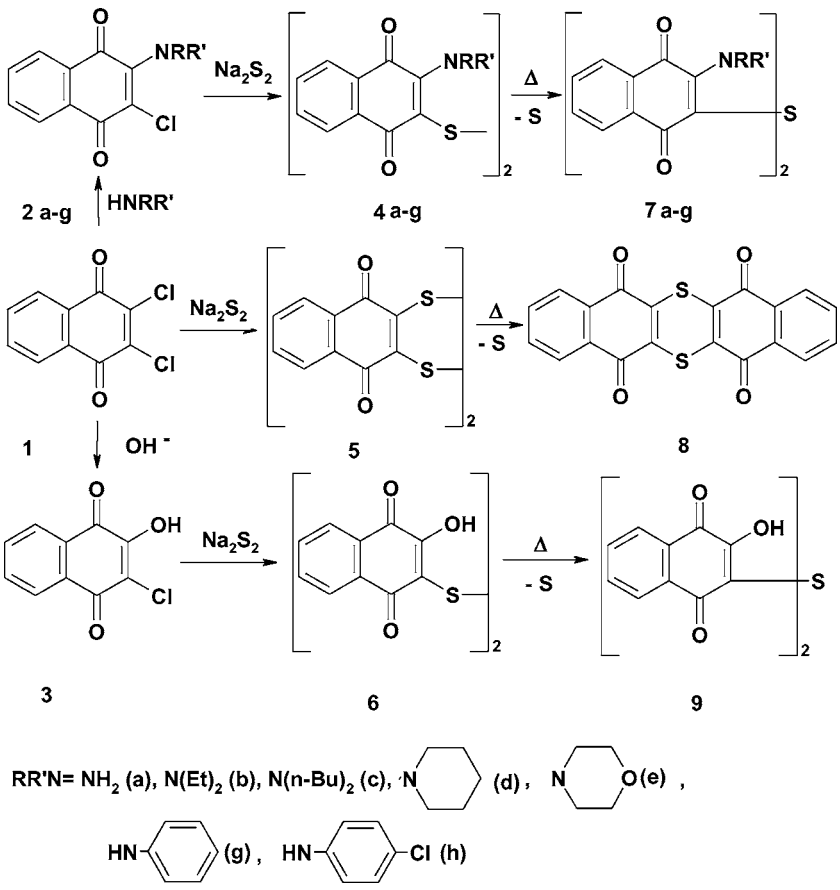


SCHEME 1

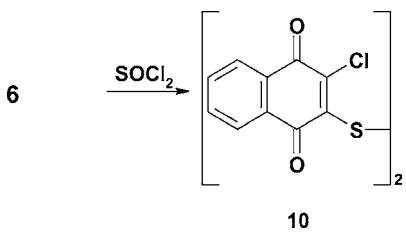
of **2** from **1** was modernized, and the yields and purity of products were improved [4].

Synthesis of disulfides **4a–g** and bis-disulfide **5** was carried out similarly to the method described [5]. Long boiling of the reaction mixture leads to desulfurization of disulfides with the formation of the sulfides **7a–g,8,9** (Scheme 1). Structures of bis-(2-amino-1,4-naphthoquinone)sulfide (**7a**), bis-(2-anilino-1,4-naphthoquinone)sulfide (**7f**), and cyclic sulfide **9** were confirmed by NMR, IR, the element analysis and were identified as described earlier [3,20]. It is established that bis-[2-hydroxy-1,4-naphthoquinone]-3-disulfide (**6**) turns into bis-(2-hydroxy-1,4-naphthoquinone)-sulfide (**9**), described in the the literature [3]. The desulfurization to **9** takes place at weak heating of reaction mixture. Therefore, reaction carried below at room temperature.

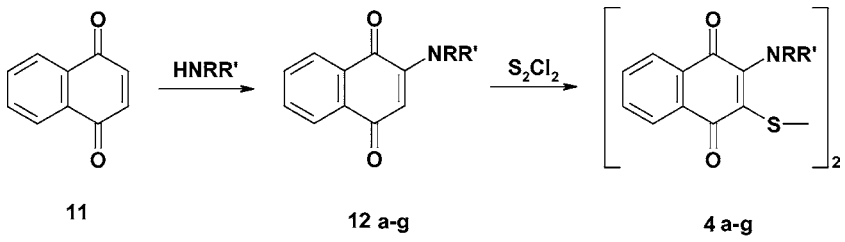
6 was processed in bis-[2-chloro-1,4-naphthoquinone]-3-disulfide **10** by thionyl chloride in dry carbon tetrachloride (Scheme 2).

Interaction of 2-Amino-1,4-Naphthoquinones and 1,4-Naphthoquinone with Disulfur Dichloride

Synthesis of disulfides by the previous method at nonconstant temperature led to by-products, which reduced yields of disulfides. Therefore, we carried out the interaction of 2-amino-1,4-naphthoquinones **12a–g**, obtained similarly to the methods [6,7], and



SCHEME 2



RR'N as in Scheme 1

SCHEME 3

of 1,4-naphthoquinone **11** with disulfur dichloride in inert solvents in the presence of the base (Scheme 3) [3]. The reaction passes easily, but with formation of by-products.

Interaction of **11** with disulfur dichloride led to formation of a product mixture (Scheme 4).

Attempts to separate the mixture by chromatography on silica gel (benzene/ethyl acetate, 80:20) gave the individual compounds, three of which were structures **13**, m = 2 (2.5%); **5** (15%); and **8** (50%). Other compounds, isolated from a reaction mixture, are impossible to identify. Formation of the reaction products can be explained by a mechanism similar to [8].

Hydrolysis of Thiocyanate Derivatives of 1,4-Naphthoquinone

Two previous methods of disulfide synthesis did not increase the purity and yields of products. Therefore, we considered method of disulfide preparation from thiocyanate derivatives. It is known that disulfides are obtained from thiocyanates by hydrolysis [9]. We synthesized disulfides from 2-amino-, 2-hydroxy-3-thiocyanate-1,4-naphthoquinone and 2,3-dichloro-1,4-naphthoquinone by a modified method [10]. Disulfides were obtained with higher yields and purity as compared to the above-mentioned methods (Scheme 5).

Synthesis of Disulfides from 2-Amino-, 2-Chloro-, 2-Hydroxy-3-thiolo- 1,4-naphthoquinones

2-Amino-, 2-chloro-, 2-hydroxy-3-thiolo-1,4-naphthoquinones were prepared similar to [11,12] by the interaction of **2,3**:

A—with sodium sulfide in water, followed by acidification of the reaction mixture;

B—with thiourea in alcohol, alkaline hydrolysis of isothiuronic salts, and acidification of the reaction mixture.

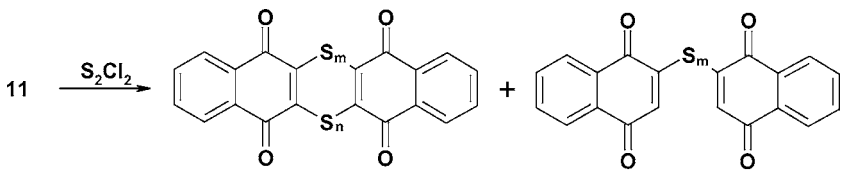
18a,f obtained by the method A were described in the literature [13,14] earlier.

It is known that thiols are easily oxidized to disulfides [9,15–17]. We carried out oxidation of **18–20** by nitric acid and hydrogen peroxide [9]. Disulfides were obtained with high yields and purity (Scheme 6).

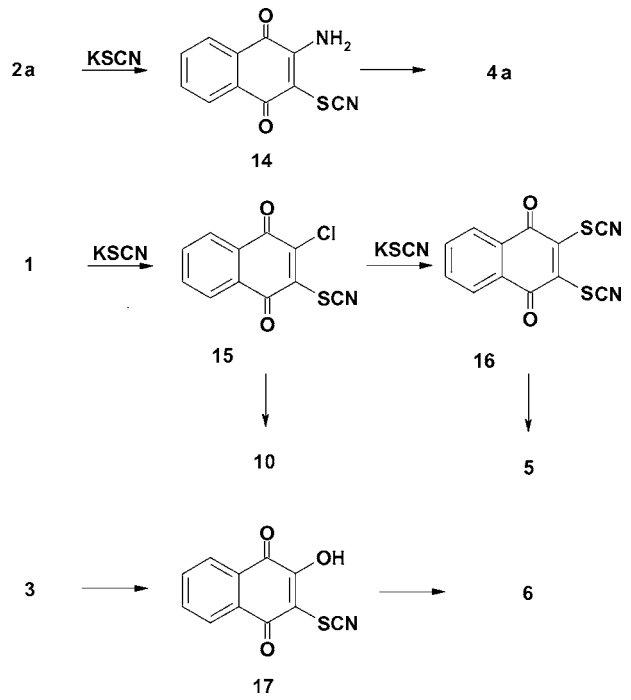
Attempts to obtain disulfides from thiols by chlorine, sulfuryl chloride, or phosphorus pentachloride in inert solvents failed because products cannot be separated.

The structures of the obtained compounds were confirmed by NMR, IR, and the element analysis.

Obtained disulfides are poorly soluble and high melting point compounds. They are synthones for the further chemical transformations with the purpose of preparation of new biologically active compounds.



SCHEME 4



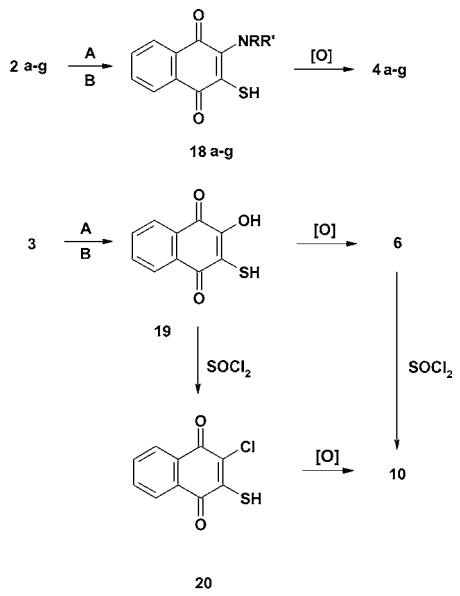
SCHEME 5

EXPERIMENTAL

Melting points were measured on a Nagema melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded on Varian VXR (300 MHz) spectrometer as solutions in DMSO-d_6 with TMS as a internal standard. IR spectra were recorded on Specord M80 in tablets KBr.

Materials. 2-Amino-3-chloro-1,4-naphthoquinones (**2a–g**) [4], 2-piperidyl-1,4-naphthoquinone (**12d**) [6,7], 2-hydroxy-3-chloro-1,4-naphthoquinone (**3**) [18], 2-chloro-3-thiocyano-1,4-naphthoquinone (**15**) from **1**, and 2,3-dithiocyano-1,4-naphthoquinone (**16**) from **1** [19].

2-Amino-3-chloro-1,4-naphthoquinone (**2a**). Ammonia was slowly passed through dense suspension of **1** in DMSO at stirring. After 5 min the reaction mixture begins to redden, **1** dissolves. Ammonia passed for 3 h; reaction mixture was left for night, and then warmed up to 50°C to remove excess ammonia. A mixture was filtered. Orange crystals, which do not require clearing, separated from the filtrate at cooling. All constants for **2a** match with those described in [14].



SCHEME 6

General Procedure of Synthesis of Bis-[2-R-1,4-naphthoquinone]-3-disulfides **4a–g** *from* **2a–g**

2a–g (0.07 mol) was suspended in 60 mL of ethanol. Mixture was brought to boiling and sodium disulfide (0.05 mol) added. Boiling was continued for 3 h. The reaction mixture was chilled and the product collected; residue was washed by water till water did not become colorless (see Table 1).

Preparation of the bis-[2-hydroxy-1,4-naphthoquinone]-3-disulfide (**6**) is carried out at <15°C. The

TABLE 1 Disulfides **4a–g,5,6** from 3-chloro-1,4-naphthoquinones **2a–g,1,3**, and Na_2S_2

		Calculated Found (%)				
	Yield (%)	*C*	*H*	*N*	*S*	*Cl*
4a	61	58.82	2.94	6.86	15.69	
		59.01	2.85	6.91	15.65	
4b	64	64.62	5.38	5.38	12.30	
		64.54	5.42	5.35	12.35	
4c	58	66.67	6.25	4.86	11.11	
		66.85	6.11	4.93	11.25	
4d	52	66.18	5.15	5.15	11.76	
		66.25	5.01	5.23	11.82	
4e	53	65.63	4.69	5.47	12.50	
		65.59	4.45	5.69	12.25	
4f	56	68.57	3.57	5.01	11.43	
		68.25	3.78	5.12	11.45	
4g	59	61.05	2.86	4.45	10.17	11.29
		61.12	2.94	4.12	10.05	11.40
6	45	58.54	2.44		15.60	
		58.75	2.36		15.78	
5	42	54.55	1.82		29.09	
		54.21	2.02		29.21	

reaction mixture was filtered, filtrate was acidified, and orange brown crystals were collected. Residue was washed by diluted hydrochloric acid and dried.

General Procedure of Synthesis of Bis-[2-R-1,4-naphthoquinone]-3-disulfides **4b–g** *from* **12b–g**

The mixture of **12b–g** (0.06 mol), triethylamine (0.12 mol), and disulfur dichloride (0.06 mol) was stirred in benzene, heated to 40–50°C for 2 h. The reaction mixture was cooled, product collected, washed by water, and dried (see Table 2).

General Procedure of Synthesis of Bis-[2-R-1,4-naphthoquinone]-3-disulfides **4a–g** *from 2-R-3-Thiolo-1,4-naphthoquinones* **18a–g**

Method A. 7.1 mmol of 30% of a solution of hydrogen peroxide was added to a water solution 9.7 mmol of 2-amino-3-thiolo-1,4-naphthoquinone. The reaction mixture was left at constant stirring for 2 days. Then mixture was filtered, the precipitate was washed by water, and dried.

Method B. 2-Amino-3-thiolo-1,4-naphthoquinone was suspended in 3 N solution of nitric acid and left at stirring for 4 days, then reaction mixture was filtered and washed by water, and dried (see Table 3).

General Procedure of Synthesis of Bis-[2-R-1,4-naphthoquinone]-3-disulfides **4a,5,6,10** *from Thiocyanates Derivatives* **14–17**

0.01 mol of **14** and 0.02 mol of ammonium acetate was suspended in 20 mL of acetic acid, and mixture was heated at 100–105°C. The reaction mixture was left at room temperature for 6 h and filtered. The precipitate was washed by small amount of water and dried (see Table 4).

TABLE 2 Disulfides **4b–g,5** from 1,4-Naphthoquinones **12b–g,11**, and S_2Cl_2

		Found (%)				
	Yield (%)	*C*	*H*	*N*	*S*	*Cl*
4b	73	64.52	5.51	6.01	11.99	
4c	75	66.42	6.11	4.75	11.01	
4d	81	65.98	5.01	5.16	11.12	
4e	74	65.12	4.35	5.53	12.12	
4f	72	68.12	4.12	5.12	11.27	
4g	78	60.91	2.74	4.53	9.92	11.11
5	15	55.01	1.96		29.15	

TABLE 3 Disulfides **4a–g,6,10** from 3-Thiolo-1,4-naphthoquinones **18a–g, 19, 20**, and H_2O_2(A) or HNO_3(B)

		Found (%)				
	Yield (%) A/B	*C*	*H*	*N*	*S*	*Cl*
4a	88/86	58.80	2.95	6.84	15.72	
4b	87/88	64.59	5.40	5.36	12.35	
4c	89/86	66.63	6.20	4.90	11.07	
4d	87/89	66.25	5.20	5.11	11.69	
4e	85/87	65.57	4.72	5.52	12.43	
4g	86/88	68.50	3.64	5.06	11.52	
4h	87/85	61.10	2.81	4.50	10.01	11.35
6	89/87	58,65	2,50		15,71	
10	83/81	53.73	1.73		14.39	15.92

TABLE 4 Disulfides **4a, 10, 5, 6** from the hydrolysis of 3-Thiocyanato-1,4-naphthoquinones

		Found (%)				
	Yield (%)	*C*	*H*	*N*	*S*	*Cl*
4a	80	58.93	2.84	6.94	15.61	
10	68	53.47	1.95	14.21		15.73
5	54	54.49	1.90	29.12		
6	72	58.65	2.50	15.71		

TABLE 5 3-Thiolo-1,4-naphthoquinones **18a–g,19** from 3-Chloro-1,4-naphthoquinones **2a–g,3**, and Na_2S (A) or $(NH_2)_2CS$ (B)

		Calculated Found (%)			
	Yield (%) A/B	*C*	*H*	*N*	*S*
18a	88/81	58.54	3.41	6.83	15.61
		58.47	3.35	6.78	15.59
18b	75/84	64.37	5.75	5.36	12.26
		64.42	5.82	5.31	12.34
18c	69/87	66.44	6.57	4.84	11.07
		66.37	6.48	4.91	11.02
18d	78/85	65.93	5.49	5.13	11.72
		65.85	5.52	5.21	11.68
18e	75/83	61.09	4.72	5.09	11.64
		61.12	4.65	6.99	11.71
18f	78/85	68.33	3.91	4.98	11.38
		68.42	3.87	4.87	11.45
18g	77/87	77.41	4.03	5.65	12.90
		77.52	3.98	5.52	12.82
19	85/91	58.25	2.91		15.53
		58.32	2.98		15.64

TABLE 6 3-Chloro-1,4-naphthoquinones **20, 10** from 3-Hydroxy-1,4-naphthoquinones **19,6**, and $SOCl_2$

		Calculated Found (%)			
	Yield (%)	*C*	*H*	*S*	*Cl*
20	88	53.45	2.22	14.25	15.81
		53.54	2.13	14.34	15.96
10	74	53.45	2.01	14.45	15.97

TABLE 7 Data of the Synthesized Compounds

	Formula mp (°C)	*^{1}H NMR (δ, ppm)*	*IR (cm^{-1})*
4a[d]	$C_{24}H_{12}N_2S_2O_4$ 309–310	δ 7.63–8,29 (6H, m, ArH and NH_2)	1655 (C=O), 1358 (*tert*-N)
4b[a]	$C_{28}H_{28}N_2S_2O_4$ >300	1.08 (12H, t, CH_3), 3.41 (8H, q, CH_2), 7.66; 7.56 (4H, td, CH_{Ar}), 8.12;8.05 (4H, dd, CH_{Ar})	1650 (C=O), 1350 (*tert*-N)
4c[a]	$C_{32}H_{36}N_2S_2O_4$ >300	0.84 (12H, t, $^3J_{HH} = 9.3$ Hz, CH_3), 1.32 (16H, m, CH_2), 3.06 (8H, m, CH_2), 8.03; 8.04 (4H, td, CH_{Ar}), 8.12;8.17 (4H, dd, CH_{Ar})	1655 (C=O), 1360 (*tert*-N)
4d[a]	$C_{30}H_{28}N_2S_2O_4$ >300	1.31 (12H, m, CH_2), 3.71 (8H, m, CH_2), 7.46; 7.78 (4H, td, CH_{Ar}), 7.98; 8.17 (4H, dd, CH_{Ar})	1620, 1656 (C=O), 1340 (*tert*-N)
4e[a]	$C_{28}H_{24}N_2S_2O_6$175	δ 3.49 (16H, m, $CH_{2morpholine}$), 7.56; 7.67 (4H, td, CH_{Ar}), 8.02;8.05 (4H, dd, CH_{Ar})	1612, 1648 (C=O), 1355 (*tert*-N)
4f[a]	$C_{32}H_{20}N_2S_2O_4$ >300	δ 7.05 (10H, m, Ar), 7.33 (2H, s broad, NH), 7.62; 7.82 (4H, td, CH_{Ar}), 7.66; 8.05 (4H, dd, CH_{Ar})	3160 (–NH–), 1610, 1650 (C=O)
4g[a]	$C_{32}H_{18}S_2O_4Cl_2$ 270	6.94 (4H, d, $^3J_{HH} = 8,4$ Hz, CH_{Ar}), 7.1 (4H, d, $^3J_{HH} = 8.4$ Hz, CH_{Ar}), 7.41 (2H, s broad, NH), 7.63; 7.70 (4H, td, CH_{Ar}), 8.16; 8.19 (4H, dd, CH_{Ar})	3160 (–NH–), 1612, 1648 (C=O)
5[a]	$C_{20}H_8S_4O_4$ >300	7.65 (4H, tm, CH_{Ar}); 8.22 (4H, dd, CH_{Ar})	1650 (C=O)
6[b]	$C_{20}H_{10}S_2O_6$ 165–167	7.45; 7.79 (4H, td, CH_{Ar}), 7.92; 8.17 (4H, dd, CH_{Ar}), 8.5 (2H, s broad, OH)	3535 (OH), 1652 (C=O)
10[b]	$C_{20}H_8S_2Cl_2O_4$ 163–165	7.7 (4H, m, CH_{Ar}), 8.2, 8.16 (4H, dd, CH_{Ar})	1660 (C=O)
14[c]	$C_{11}H_6SN_2O_2$ 210	δ 7.63–8.29 (6H, m, ArH and NH_2)	1655 (C=O)
17[b]	$C_{11}H_5SNO_2$ 191	δ 7.79; 7.81 (2H, td, CH_{Ar}), 8.05; 8.15 (2H, dd, CH_{Ar}), 8.71 (1H, s broad, OH)	3540 (OH), 2100 (SCN), 1662 (C=O)
18a[d]	$C_{12}H_{12}SNO_2$ 290–292	δ 7.63–8.29 (6H, m, ArH and NH_2); 8.51 (1H, s, SH)	2850 (SH), 1660 (C=O), 1351 (*tert*-N)
18b[d]	$C_{14}H_{15}SNO_2$ 245–247	δ 1.08 (6H, t, CH_3), 3.42 (4H, q, CH_2), 7.56; 7.68 (2H, td, CH_{Ar}), 7.99; 8.03 (2H, dd, CH_{Ar}), 8.52 (1H, s, SH)	2850 (SH), 1664 (C=O), 1350 (*tert*-N)
18c[d]	$C_{16}H_{19}SNO_2$ 278	δ 0.89 (6H, t, $^3J_{HH} = 9,2$ Hz, CH_3), 1.28 (8H, m, CH_2), 3.07 (4H, m, CH_2), 7.56;7.65 (2H, td, CH_{Ar}), 7.98;8.08 (2H, dd, CH_{Ar}), 8.53 (1H, s, SH)	2858 (SH), 1658 (C=O), 1360 (*tert*-N)
18d[d]	$C_{15}H_{15}SNO_2$ 197	δ 1.31 (6H, m, CH_2), 3.72 (4H, m, CH_2), 7.6; 7.74 (2H, td, CH_{Ar}), 7.98; 8.03 (2H, dd, CH_{Ar}), 8.48 (1H,s,SH)	2852 (SH), 1654 (C=O), 1362 (*tert*-N)
18e[d]	$C_{14}H_{13}SNO_3$ 285	δ 3.51 (8H, m, $CH_{2morpholine}$), 7.47;7.67 (2H, td, CH_{Ar}), 8.03;8.06 (2H, dd, CH_{Ar}), 8.49 (1H,s,SH)	2855 (SH), 1658 (C=O), 1340 (*tert*-N)
18f[d]	$C_{16}H_{11}SNO_2$ 281	δ 7.11 (5H, m, Ar), 7.35 (1H, s broad, NH), 7.59; 7.77 (2H, td, CH_{Ar}), 7.61;8.11 (2H, dd, CH_{Ar}), 8.49 (1H, s, SH)	3175 (–NH–), 2860 (SH), 1650 (C=O)
18g[d]	$C_{16}H_{10}SNO_2$ 178–179	δ 6.98 (2H, d, $^3J_{HH} = 8.3$ Hz, CH_{Ar}), 7.2 (2H, d, $^3J_{HH} = 8.4$ Hz, CH_{Ar}), 7.42 (2H, s broad, NH), 7.66; 7.74 (2H, td, CH_{Ar}), 8.13; 8.16 (2H, dd, CH_{Ar}), 8.47 (1H, s, SH)	3175 (–NH–), 2850 (SH), 1665 (C=O)
19[d]	$C_{10}H_6SO_3$ 175–177	δ 7.65; 7.79 (2H, td, CH_{Ar}), 8.05; 8.11 (2H, dd, CH_{Ar}), 8.52 (1H, s, SH), 8.7 (1H, s broad, OH)	3540 (OH), 2864 (SH), 1662 (C=O)
20[d]	$C_{10}H_5SClO_2$ 180	δ 7.79 (2H, m, CH_{Ar}), 8.08; 8.16 (2H, dd, CH_{Ar}), 8.51 (1H,s,SH)	2860 (SH), 1650 (C=O)

Recrystallization solvents: [a]chlorobenzene, [b]benzene/hexane, [c]benzene, [d]DMF/ethanol.

*2-Amino-3-thiocyano-1,4-naphthoquinone (***14***) from* **2a**. To a chloroform solution of 0.034 mol 2-amino-3-chloro-1,4-naphthoquinone was added potassium thiocyanate. Mixture was warmed up for 3 h, cooled, filtered, the filtrate was left for night at cooling then orange red crystals separated from it.

Compound **17** was transferred to potassium salt of it in the ratio 1:1 and then was hydrolyzed similarly to the method described for **14**.

General Procedure of Synthesis of 2-R-3-Thiolo-1,4-naphthoquinone **18a–g**

Method A. To a water–ethanol solution of 2-amino-3-chloro-1,4-naphthoquinone (9.6 mmol) added a water solution of sodium sulfide (0.026 mol). Reaction mixture was warmed up for 3 h, was cooled, filtered; a filtrate was acidified and brown crystals of thiol separated from it and which was washed by water and dried.

Method B. 0.024 mol of 2-amino-3-chloro-1,4-naphthoquinone and 0.024 mol of thiocarbamide were suspended and then brought to boil during 12 h. Then solvent was evaporated in vacuo. Water solution of 0.109 mol of potassium hydroxide was added to isothiuronic salt and was boiled for 6 h, filtered the reaction mixture, a filtrate was acidified, crystals was filtered and washed by diluted hydrochloric acid, and dried (see Table 5).

The constants of compounds **18a,f** obtained by both methods match with those described earlier in the literature [19].

*Synthesis of Bis-[2-chloro-1,4-naphthoquinone]-3-disulfide (***10***) and 2-Chloro-3-thiolo-1,4-naphthoquinone (***20***)*

0.005 mol of **6** (or **19**) was suspended in 100 mL of benzene, 0.01 mol of thionyl chloride, and few drops of DMF were added. Mixture was left at heating for day. Gradually disulfide passed in solution. The filtrate was evaporated and oil residue was treated with benzene for full dissolution; and then **10** (or **20**) was precipitated by hexane, filtered, and dried (see Table 6), Data of all synthesized compounds are shown in Table 7.

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