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Electrochemical Synthesis, Structure, Magnetic and Tribochemical Properties of Metallochelates of New Azomethine Ligands, Bis-[2-(*N*-tosylaminobenzylidenealkyl(aryl)]disulfides

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Abstract—On the basis of new ligand systems, bis-azomethines of *N*-tosylaminobenzaldehyde and alkyl(aryl) diaminodisulfides, mono- and binuclear chelates with 4N- and 2N2S-ligand arrangement were chemically (from metal acetates) or electrochemically (from metals in the zero oxidation state) first synthesized and characterized. Mononuclear chelates have a tetrahedral structure. In binuclear copper complexes the magnetic exchange interaction of the antiferromagnetic type was observed. Bisazomethines of disulfides were used as additives to lubricant oils.

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Electrochemical synthesis based on the anode dissolution of the complex-forming metals in the zero oxidation state is an important method of preparation of coordination compounds [1–15]. In summary, the process of electrolysis of proton-donor ligating compounds can be represented as follows:

$$\text{Cathode (Pt): } n\,\text{LH} + n\,\text{e} \rightarrow n\,\text{L}^- + n/2\,\text{H}_2,$$

$$\text{Anode (M}^0\text{): M} - n\,\text{e} \rightarrow \text{M}^{n+},$$

$$\text{Solution: } n\text{L}^- + \text{M}^{n+} \rightarrow [\text{MLn}].$$

Advantages of electrosynthesis over conventional chemical synthesis of coordination compounds consist in the possibility of the purposeful preparation of metal complexes containing no anions and in carrying out synthetic reactions under mild conditions (usually at room temperature) with a high yield of final products [10, 15].

The method of electrochemical synthesis was widely used for preparation of metal chelates, in particular, the Schiff bases complexes [9–13, 15]. An important place in this research occupies electrosynthesis of coordination compounds of azomethines containing in the coordnation sphere the chalcogene atoms, for example [10, 16–20].

In the development of research in this field we have first synthesized and studied the ligating azomethines: bis[2-(*N*-tosylaminobenzylidenealkyl(aryl))]disulfides **I**, their mono- **II** and binuclear **III** complexes.

The synthesis of bisazomethine disulfide **I** was performed by classical method for preparation of the Schiff bases [21] by the reaction of *o*-tosylaminobenzaldehyde (**IV**) [22] with 2,2'-dithiodianiline (**V**) [23].

Electrosynthesis of chelates **II** and **III** was performed from bisazomethine ligating compounds under standard conditions [10, 12, 15] upon anodic dissolution of the complex-forming metals (Co^0, Ni^0, Cu^0, Zn^0, Cd^0) with a platinum cathode in acetonitrile at 20°C with $[\text{Et}_4\text{N}]\text{ClO}_4$ as a conducting additive. Charac-

Table 1. Data of elemental analysis of azomethines **Ia–Ic** and their complexes

Comp. no.	R	X	M	Composition	mp, °C	Found, %				Formula	Calculated, %			
						C	H	N	M		C	H	N	M
Ia	$(CH_2)_2$	Ts	H		90–91	57.93	5.32	8.62	–	$C_{32}H_{34}N_4O_4S_4$	57.63	5.14	8.40	–
IIa	$(CH_2)_2$	Ts	Cu	CuL	229–230	52.45	4.32	7.90	9.32	$C_{32}H_{32}N_4O_4S_4Cu$	52.76	4.42	7.69	8.72
IIa[a]	$(CH_2)_2$	Ts	Cu	CuL[a]	229–230	52.43	4.12	7.80	9.10	$C_{32}H_{32}N_4O_4S_4Cu$	52.76	4.42	7.69	8.72
IIb	$(CH_2)_2$	Ts	Ni	NiL	265–266	53.11	4.25	7.63	8.21	$C_{32}H_{32}N_4O_4S_4Ni$	53.15	4.45	7.74	8.11
IIb[a]	$(CH_2)_2$	Ts	Ni	NiL[a]	245–246	53.25	4.32	7.56	8.30	$C_{32}H_{32}N_4O_4S_4Ni$	53.15	4.45	7.74	8.11
IIc	$(CH_2)_2$	Ts	Co	CoL·MeOH	>250	52.45	4.17	7.70	17.00	$C_{33}H_{36}N_4O_5S_4Co$	52.44	4.80	7.42	16.97
IId	$(CH_2)_2$	Ts	Zn	ZnL	220–221	52.38	4.36	7.28	8.89	$C_{35}H_{35}N_4O_4S_4Cu$	52.63	4.41	7.67	8.59
IId[a]	$(CH_2)_2$	Ts	Zn	ZnL[a]	215–216	52.45	4.52	7.83	9.10	$C_{32}H_{32}N_4O_4S_4Zn$	52.63	4.41	7.63	8.95
IIe	$(CH_2)_2$	Ts	Cd	CdL	205–207	50.12	4.24	7.32	14.76	$C_{32}H_{32}N_4O_4S_4Cd$	49.44	4.14	7.20	14.46
Ib	$(CH_2)_2$	O	H	H_2L	79–80	59.29	5.85	7.23	–	$C_{18}H_{20}N_2O_2S_2$	59.97	5.59	7.77	–
IIf	$(CH_2)_2$	O	Cu	CuL	175	51.70	4.67	6.40	14.86	$C_{18}H_{18}N_2O_2S_2Cu$	51.23	4.29	6.64	15.05
IIf[a]	$(CH_2)_2$	O	Cu	CuL[a]	169	51.43	4.42	6.70	15.5	$C_{18}H_{18}N_2O_2S_2Cu$	51.23	4.29	6.64	15.05
IIg	$(CH_2)_2$	O	Ni	NiL	193	51.43	4.10	6.32	14.58	$C_{18}H_{18}N_2O_2S_2Ni$	51.79	4.35	6.71	14.07
IIg[a]	$(CH_2)_2$	O	Ni	NiL[a]	180–181	52.21	4.55	6.32	14.58	$C_{18}H_{18}N_2O_2S_2Ni$	51.79	4.35	6.71	14.07
IIh	$(CH_2)_2$	O	Co	CoL	190–191	51.70	4.16	7.10	14.62	$C_{18}H_{18}N_2O_2S_2Co$	51.79	4.34	6.71	14.12
IIi	$(CH_2)_2$	O	Zn	ZnL	230–231	51.12	4.41	6.56	15.58	$C_{18}H_{18}N_2O_2S_2Zn$	51.00	4.28	6.61	15.42
IIi[a]	$(CH_2)_2$	O	Zn	ZnL[a]	220–221	51.32	4.18	6.84	15.12	$C_{18}H_{18}N_2O_2S_2Zn$	51.00	4.28	6.61	15.42
IIj[a]	$(CH_2)_2$	O	Cd	CdL[a]	210–211	45.31	3.82	5.34	24.10	$C_{18}H_{18}N_2O_2S_2Cd$	45.91	3.85	5.95	23.87
Ic	Ph	Ts	H	H_2L	155–156	62.67	4.31	7.42	–	$C_{40}H_{34}N_4O_4S_4$	62.97	4.49	7.30	–
IIk	Ph	Ts	Cu	$CuL·H_2O$	>250	57.32	4.25	6.84	8.20	$C_{40}H_{34}N_4O_5S_4Cu$	57.02	4.06	6.64	7.54
IIk[a]	Ph	Ts	Cu	CuL[a]$·H_2O$	>250	54.68	4.28	6.94	14.76	$C_{40}H_{34}N_4O_5S_4Cu$	54.10	3.64	6.31	14.4
IIl	Ph	Ts	Ni	NiL[a]$·H_2O$	>250	56.85	3.88	6.26	7.61	$C_{40}H_{34}N_4O_5S_4Ni$	57.36	4.09	6.69	7.00

[a] Complex compounds prepared by the electrochemical synthesis.

teristics of the products of the electrochemical reaction are presented in Table 1.

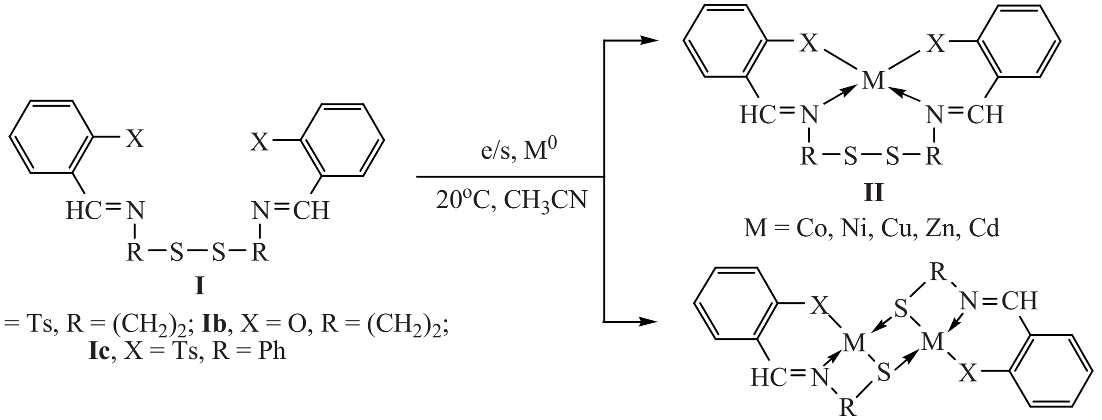
To compare the characteristics of the previously unknown complexes, their chemical synthesis was performed by direct reaction [12, 13] of ligand **I** with acetates of the aforementioned d-metals in boiling methanol.

It is known [10, 16–20] that under the conditions of electrosynthesis based on disulfide ligands (**I**, X = O) complex compounds with both retention and rupture of the S–S bond can be obtained. The latter result is based on the data of the Tuck's studies [23–25]. Judged from the ^1H NMR spectra, the ligand systems of the type **I** display the amino–imine tautomerism: the signals of the HC=N (8.40–8.50 ppm) and HNTs (12.80–12.90 ppm) groups are observed.

According to the elemental analysis (Table 1), all prepared compounds have ML composition regardless of the method of their synthesis. This can result from the formation of mono- (ML–**II**) or binuclear (M_2L_2–**III**) complexes [12, 13, 26–28]. The chelate type of the described structures **II** and **III** follows from comparison of the data of IR and ^1H NMR spectra.

The C=N bond stretching vibration frequencies in the IR spectra decrease on going from ligands **I** to complexes **II, III** (1630–1640 cm^{-1} for ligands, 1620–1630 cm^{-1} for complexes) (Table 2), which, according to the data of [27] and references therein proves participation of the azomethine nitrogen atom in the coordination.

Comparison of the ^1H NMR spectra of ligands **I** and zinc complexes **II** (Table 2) shows the disappearance in the products of complex formation the signals of protons of the HNTs fragments and a downfield shift of the signals of the HC=N group (8.50–8.60 ppm), which is in compliance with the formation of metallocycles including 4N (**II**) or 2N2S (**III**) donor centers.



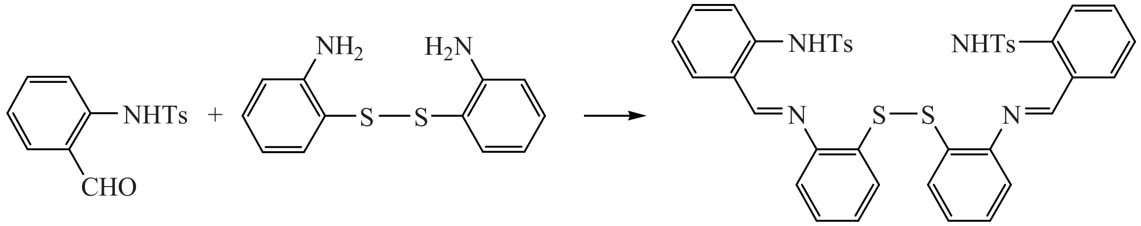
I

Ia, X = Ts, R = $(CH_2)_2$; **Ib**, X = O, R = $(CH_2)_2$;
Ic, X = Ts, R = Ph

II

M = Co, Ni, Cu, Zn, Cd

III



IV **V** **I**

The choice between mono- **II** and binuclear **III** structures has been made on the basis of the ESR and magnetochemical studies at variable temperature (295–77 K).

The electrochemically synthesized copper complexes of the type (**II**, X = O, NTs) have the following parameters of the ESR spectra taken in $CHCl_3$: $g_{\|} = 2.229$; $g_{\perp} = 2.069$; A = 167.4 E. [**II**, R = $(CH_2)_4$, X = O]; $g_1 = 2.219$; $g_2 = 2.134$; $g_3 = 2.080$ [**II**, R = $(CH_2)_4$, X = NTs]. A notable increase in the degree of tetrahedral distortion in complexes [**II**, R = $(CH_2)_4$, X = NTs] is due to steric hindrances caused by the tosyl fragment. Replacement of the hydroxyl oxygen by the NTs group results in an increase in the anisotropy of *g*-factors and to such a sharp decrease in the hyperfine splitting constant (HSC) on copper that in complex (**II**, X = NTs) it practically cannot be seen.

Similar data have been obtained by ESR study of the copper complexes synthesized by chemical method from ligand **I** (X = NTs, R = C_6H_4). In the ESR spectrum of this complex one unresolved signal with $g = 2.1$ is observed. This can be due to the formation of a substantially distorted tetrahedral structure of the monomer complex [29, 30]. In this case the signals g_1, g_2, g_3, cannot be seen, as in complex **II** (X = NTs, R = C_6H_4) because of a large width of the line.

It is interesting to note that in the ESR spectrum of the same complex prepared by electrochemical method the signals $g_1 = 2.268$; $g_2 = 2.131$; $g_3 = 2.086$ are superimposed on the wide signal $g = 2.1$. These results can be explained by the formation upon electrosynthesis of a mixture of the mononuclear **II** and binuclear **III** structures with the rupture of the disulfide groups.

This conclusion is definitely proved by the magnetochemical data (Table 3). For copper complexes **II** [X = O, NTs, R = $(CH_2)_2$], irrespective of the method of preparation, metallochelates are formed whose magnetic properties practically do not vary with temperature [μ_{eff}=2.09 (294.5 K) – 1.94 (82.9 K), 1.89 (295 K) – 1.86 (82.3 K) BM]. This is indicative of their monomeric structure of the type (**II**, L = Cu) with retention of the S–S bond. A similar magnetochemical result was obtained for the synthesized from copper acetate complex (**II**, L = Cu, X = NTs, R = C_6H_4). The value of its μ_{eff} = 1.82 BM does not change when measured even at 77 K, which points to the formation of mononuclear complex.

For the copper complex prepared under the conditions of electrosynthesis from the ligand (**I**, X = NTs, R = C_6H_4), as distinct from the chemically synthesized copper chelate, a temperature dependence of μ_{eff} = 1.92 MB (295 K) and 1.59 MB (82.2 K) was observed, suggesting the presence of magnetic exchange of antiferromagnetic type. This result, according to the literature data [31, 32], can be related to the formation

Table 2. Magnetic moments, IR and ^{1}H NMR spectra of compounds **Ia–Ic** and their complexes **IIa–IIl**

Comp . no.	μ_{eff}., BM	IR spectrum, ν, cm^{-1}						^{1}H NMR spectrum (DMSO-d_6), δ, ppm
		M	OH	C=N	SO$_2$, as	SO$_2$, s	Ph–O	
Ia	–	H	–	1620 o. c	1320 vs	1146 vs	–	2.21 s (6H, 2CH$_3$), 3.13 t (4H, 2CH$_2$), 3.93 t (4H, 2CH$_2$), 7.07–7.67 m (16H, H$_{arom}$), 8.48 s (2H, CH=N), 12.88 br.s (2H, 2NH)
IIa	2.09–1.94 (294–82.9 K)	Cu	–	1617 vs	1288 vs	1121 vs	–	
IIa[a]	1.76–1.71 (293–83 K)	Cu[a]	–	1617 vs	1286 vs	1126 vs	–	
IIb	3.12 (296 K)	Ni	–	1613 vs	1300 vs	1116 vs	–	
IIb[a]	3.2 (296 K)	Ni[a]	–	1613 vs	1300 vs	1116 vs	–	
IIc		Co	–	1606 vs	1286 vs	1120 vs	–	
IId	dia	Zn	–	1620 vs	1288 vs	1126 vs	–	2.55 s (6H, 2CH$_3$), 3.80 br.s (2H, CH$_2$), 4.30 br.s (2H, CH$_2$), 6.68–7.94 m (16H, H$_{arom}$), 8.56 s (2H, 2CH=N)
IId[a]	dia	Zn[a]	–	1620 vs	1288 vs	1126 vs	–	
IIe	dia	Cd	–	1609 vs	1286 vs	1126 vs	–	
Ib	–	H	–	1620 vs	–	–	1275 s	
IIf	1.89–1.86 (295–82.3 K)	Cu	–	1606 vs	–	–	1340 s	
IIf[a]	1.90–1.89 (295–82 K)	Cu[a]	–	1600 vs	–	–	1340 s	
IIg	1.76 (296 K)	Ni	–	1606 vs	–	–	1342 s	
IIg[*]	1.76 (296 K)	Ni[*]	–	1606 vs	–	–	1342 s	
IIh	0.76 (296 K)	Co	–	1607 vs	–	–	1333 s	
IIi		Zn	–	1622 vs	–	–	1332 s	
IIi[a]		Zn[a]	–	1622 vs	–	–	1333 s	
IIj[a]		Cd[a]	–	1608 vs	–	–	1330 s	
Ic	–	H	3433 br	1600 s	1326 s	1142 vs	–	
IIk	1.82–1.82 (295–82 K)	Cu	–	1600 s	1296 s	1142 vs	–	
IIk[a]	1.91–1.59 (295–82 K)	Cu[a]	3340 br	1600 s	1296 s	1124 vs	–	
IIl	3.11 (296 K)	Ni	–	1593 s	1296 s	1120 vs	–	

[a] Complex compounds prepared by the electrochemical synthesis.

of binuclear complexes of the type **III** formed upon electrochemical rupture of the S–S bond of the ligand system (**I**, X = NTs, R = C_6H_4).

A similar situation was earlier observed in complexes of the ligand (X = O) prepared from zero-valent metals under the conditions of electrosynthesis [8].

Effective magnetic moments for nickel and cobalt complexes of the type (**II**, X = O, R = C_6H_4) with tetradentate ligands lacking a tosyl fragment are equal at room temperature to 1.77 and 0.76 MB, respectively. They are in agreement with the structure of four-coordinated low-spin metallochelates with planar coordination node for the cobalt and tetrahedrally distorted nickel complex [34]. Thus, planar chelate node CKC Co(2+) was proved by X-ray analysis for the earlier studied [*N,N'*-ethylenebis(2-amino-5-chlorobenzylideneiminate)]cobalt(2+) [2].

Introduction of the tosyl group into the molecule of ligand (X = NTs) substantially affects the distortion of the planar chelate node and favors the formation of nonplanar structures. For the nickel chelate of this type, μ_{eff} = 3.12 (296 K); 3.20 (296 K) BM, and for the cobalt chelate it is 4.34 (296 K) BM, which is indicative of a distorted tetrahedral structure of the coordination node [34, 35].

Therefore, when studying the complex formation of bis[2-*N*-tosylaminobenzimidene-2'-aminoalkyl(aryl)]-disulfides (**I**, X = NTs, R = $(CH_2)_4$, C_6H_4) with metals it was established that both ligand systems irrespective of the method of synthesis are capable of formation of metallochelates (with Cu, Co, Zn, Ni, Cd). Bis[2-(*N*-tosylaminobenzimidene)-2'-aminophenyl]disulfide under the conditions of electrosynthesis with the use of copper anode suffers the rupture of the disulfide bond and, as a result, the metallochelates on its basis have binuclear structure with spin-spin antiferromagnetic interaction between the copper atoms.

Azomethine compounds and complexes on their basis are effective additives to lubricant oils [36–40]. In this connection, we have studied compounds **I** as additives to lubricant oils. Lubricant properties of different oils can be improved by addition into the lubricant composition 0.1–0.5 wt % of **I**. With this, the maximum loading increases as compared to other compositions by 20–40%, the weight wear intensity decreases by 15–30% for bronze and by 40–95% for steel, and the friction coefficient decreases by 40–70%. Therefore, the obtained and studied in the present work azomethines and their complexes can be practical

Table 3. Temperature dependence of magnetic properties of complexes **IIa, IIf**, and **IIe**

Comp. no.	T, K	$\chi M_{exp} \times 10^6$, $cm^3 mol^{-1}$	$M_{eff, exp}$, BM
IIa	82.9	5682.4	1.94
	100	4685.4	1.94
	118	3978.1	1.94
	140	3381.7	1.95
	162	3126.0	2.01
	186	2819.3	2.05
	217	2444.3	2.06
	256	2103.5	2.08
	294.5	1847.9	2.09
IIa[a]	83.1	4382.8	1.71
	100	3646.7	1.71
	120	2997.2	1.70
	142	2564.2	1.71
	164	2239.5	1.71
	181.5	2077.1	1.74
	203	1914.7	1.76
	232	1671.2	1.76
	293	1319.4	1.76
IIf	82.3	5249	1.86
	100.5	4195.9	1.84
	120	3478.8	1.83
	148.5	2873.8	1.85
	164	2649.8	1.86
	188	2358.5	1.88
	222	1955.1	1.86
	245	1787.1	1.87
	295	1507.0	1.89
IIe	82.1	5044.3	1.82
	99.6	4133.3	1.81
	119	3384.3	1.79
	144	2745.8	1.78
	156	2522.3	1.77
	183	2235.0	1.81
	226	1788.1	1.80
	253	1564.1	1.78
	295	1405.0	1.82
IIe[a]	82.9	8299.8	1.66
	99.6	6902.3	1.66
	120	5903.9	1.69
	143	5305.6	1.75
	162	4905.6	1.74
	188	4506.4	1.85
	241	3841.5	1.93
	258	3574.2	1.93
	259	3306.0	1.98

[a] Complex compounds prepared by the electrochemical synthesis.

additives to lubricant oils and used for development of new effective lubricant compositions for different machines and mechanisms.

EXPERIMENTAL

IR spectra were taken on a Specord IR 72 instrument, samples prepared as pastes in mineral oil. ^{1}H NMR spectra were registered on a Varian Unity 300 instrument (300 MHz) in the regime of internal stabilization of the polar resonance line of ^{2}H in $CDCl_3$. ESR spectra were recorded on a FR 9 (Zeiss) instrument from powders. Magnetic susceptibilities in solid phase were measured by the Faraday method in the range 80–300 K using the device made in the Rostov State University [41].

The azomethine compounds Ia–Ic were prepared by the following general procedure: To the solution of 1.22 g of salicylaldehyde or 2.75 g of 2-(*N*-tosylaminobenzal-dehyde) [22] in 50 ml of methanol the filtered solution of 1.12 g of bis(aminoethyl)disulfidehydrochloride and 1.36 g of anhydrous sodium acetate in 30 ml of methanol or 1.24 g of 2,2'-dithioaniline in 20 ml of methanol was added. The mixture was refluxed for 1 h, the precipitated azomethines were filtered off and washed with methanol (2×5 ml), crystallized from the mixture chloroform−methanol (1:2), and dried under a vacuum. Yield 85–87%.

Metal complexes IIa–IIl were prepared by the reaction of equimolar amounts of the corresponding azomethines **Ia–Ic** and acetates of the corresponding metals in methanol (chemical method). The mixture was refluxed for 1 h. The complexes precipitated after cooling were filtered off, washed with methanol, crystallized from the mixture chloroform−methanol (2:1), and dried under a vacuum. Yield 80–85%.

Electrochemical synthesis of complex compounds **IIa, IIb, IId, IIf, IIg, IIi, IIj,** and **IIk** was performed by anodic dissolution of the metal (Cu, Ni, Zn, Cd) in the methanol solution of the ligand by the routine procedure [10, 11, 42] $Pt(-)/MeOH/H_2L/M(+)$. $[Et_4N]ClO_4$ was used as a conducting additive. Electrosynthesis was carried out at constant current of 40 mA and voltage 20 V during 2 h at 23°C. Charge of the electrolyzer: 0.5 g of the ligand was dissolved in 30 ml of methanol at heating. After completion of electrosynthesis the precipitate formed was filtered off, washed with boiling methanol (3×5 ml), dried under a vacuum at 150°C. The data of elemental analysis and characteristics of complexes are given in Table 1.

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