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COORDINATION COMPOUNDS

Copper N-(4'-Benzo-15-crown-5)-2-(amino-Ntosyl)phenylaldiminate: Complexing and Ion-Selective Properties

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Abstract—Copper(II) chelate with N-(4'-benzo-15-crown-5)-2-(amino-N-tosyl)phenylaldiminate CuL₂ and its coordination compounds with Li, Na, and K salts have been synthesized. Their IR spectra have been studied and assignment of the key vibration frequencies of CuL₂ and complexes based on it. On the basis of spectral, thermogravimetric, and elemental analysis datasassumptions concerning the compound structure are made. The ion-selective properties of CuL₂ are studied by potentiometry.

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In recent years, enhanced attention has been paid to ligand systems containing simultaneously several donor centers, in particular, azomethine derivatives of crown ethers and coordination compounds based on them [1–7]. Previously, we reported the results of studies of the ZnL_2 chelate and its complexes with

alkali metals [8, 9]. To continue the studies of the complexing properties of ambidentate azomethine derivatives of benzo-15-crown-5, we investigated the complexing properties and potentiometric selectivity of the metal chelate CuL_2 .



EXPERIMENTAL

CuL₂ was prepared by the following procedure. Copper acetate (0.00001 mol) was added to a solution of N-(4'-benzo-15-crown-5)-2-(amino-N-tosyl)phenyl-aldimine (HL) (0.1 g, 0.00002 M) in methanol (10 mL) and the mixture was refluxed for 2 h on a water bath. The precipitate was filtered off and recrystallized from alcohol. The heteronuclear CuL_2 complexes with lithium, sodium, and potassium salts were synthesized by dissolving a CuL_2 sample in acetonitrile and adding the appropriate portion of an alkali metal salt in the ratio CuL_2 : M = 1 : 2 (for Li and Na salts) or 1 : 1 (for K salts). The solution was left in air for 5–7 days. The precipitate formed upon solvent evaporation was washed with alcohol and dried over CaCl₂.

Compound	Content (found/calculated), %					
Compound	Н	С	Ν	S		
$\overline{CuL_{2}C_{56}H_{62}N_{4}O_{14}S_{2}Cu_{1}}$	4.8/5.5	59.4/58.9	4.9/4.9	not analyzed		
$\begin{array}{l} 2LiClO_4 \cdot CuL_2 \cdot 2H_2O\\ C_{56}H_{66}N_4O_{24}S_2Cl_2Li_2Cu_1 \end{array}$	4.8/4.7	44.8/48.2	4.0/4.0	3.6/4.6		
$\begin{array}{l} 2NaI \cdot CuL_2 \cdot 2H_2O \\ C_{56}H_{66}N_4O_{24}S_2I_2Na_2Cu_1 \end{array}$	4.0/4.5	43.7/45.4	3.7/3.8	5.2/4.3		
$\begin{array}{l} 2NaSCN\cdot CuL_{2}\cdot 2H_{2}O\\ C_{58}H_{66}N_{4}O_{24}S_{4}I_{2}Na_{4}Cu_{1} \end{array}$	5.6/5.1	49.6/51.9	6.6/6.3	not analyzed		
$\begin{array}{l} KI \cdot CuL_2 \cdot H_2O \\ C_{56}H_{64}N_4O_{15}S_2I_1K_1Cu_1 \end{array}$	5.1/4.8	48.7/50.7	4.2/4.2	5.7/4.8		
$\begin{array}{l} KSCN \cdot ZnL_{2} \\ C_{57}H_{62}N_{5}O_{14}S_{3}K_{1}Zn_{1} \end{array}$	4.9/5.0	57.6/55.2	6.8/5.6	not analyzed		

Table 1. Results of elemental analysis of CuL₂ and its complexes with alkali metals

The elemental analysis of the obtained compounds was carried out on a Carlo Erba analyzer. According to elemental analysis (Table 1), trinuclear complexes are formed with Li⁺ and Na⁺ and binuclear complexes are obtained with K⁺: 2 MX \cdot ZnL₂ \cdot *n*H₂O (M = Li, Na; X = ClO₄, NCS, I; *n* = 0; 2) and KX \cdot ZnL₂ \cdot *n*H₂O (X = I, NCS; *n* = 0; 1).

The IR absorption spectra were recorded on a Nicolet NEXUS FT IR spectrophotometer in the range 4000–400 cm⁻¹ (as mineral oil and hexachlorobutadiene mulls).

Thermogravimetric measurements were carried out on a Q-1500D derivatograph in the temperature range of 20–500°C in platinum crucibles; the heating rate was 10 K/min.

Plasticized polymeric membranes containing HL, ZnL₂, or CuL₂ were prepared by a known procedure [10] (a solution in the active component in an organic solvent, a plasticizer, was introduced into the polyvinyl chloride matrix). As the plasticizer, *o*-nitrophenyl octyl ether (dielectric constant $\varepsilon = 23$) was used. The membranes had the following composition (wt %): neutral transport agent, 1; poly(vinyl chloride), 33; plasticizer, 66.

The electroanalytical parameters of the membranes were studied by means of the galvanic circuit

Ag|AgCl, KCl(1 M)|sample solution|membrane|internal solution|AgCl|Ag

The e.m.f. was measured by an OR-300 pH ionometer (Radelkis, Hungary).

The electroanalytical parameters of ion selective electrodes were measured according to IUPAC recommendations [11] at pH 5–7. The selectivity coefficients were determined by the biionic potential method.

RESULTS AND DISCUSSION

In studies [9, 12] devoted to complexation of azomethine derivatives HL and ZnL_2 with some alkali

metal salts, it was shown that the complexation in both cases is due to coordination of the oxygen atoms of the macrocyclic moiety to the alkali metal. X-ray diffraction and vibrational spectroscopy showed analysis data for [5, 6, 8] that the macrocycle conformations in HL, ZnL_2 and HL^1 are different, being dependent not only on the nature of the substituent in the benzene ring but changing appreciably upon coordination of zinc cation to the azomethine moiety. In addition, the ion-selective properties of HL and ZnL_2 as neutral transport agents are markedly different.

The vibrational frequencies of CuL_2 and the derived complexes (Table 2) were assigned resorting to the spectral data for HL, ZnL_2 , HL¹, B15C5, 4'-nitrobenzo-15-crown-5, and their complexes [5, 6, 8, 9, 12–14].

The IR spectrum of CuL₂ has changed with respect to the spectrum of HL similarly to the spectrum of ZnL₂, which changes upon coordination to the azomethine moiety. First, the δ (NH) band (1572 cm⁻¹ in HL) disappears upon the replacement of the proton by a copper ion; the v(C=N) band shifts to lower frequencies by 7 cm⁻¹ (or 4 cm⁻¹ for ZnL₂) from its position in the spectrum of HL. Second, the v_{as}(SO₂) and v_s(SO₂) stretching vibration frequencies considerably decrease (1303, 1141 cm⁻¹ for CuL₂ and 1338, 1156 cm⁻¹ for HL, respectively) (Table 2).

The stretching vibrations involving the anisole oxygen atoms, v_s (PhO) and v_{as} (PhO), occur in the spectrum of CuL₂ at 1263 and 1234 cm⁻¹ as medium-intensity bands (for ZnL₂, 1269 and 1244 cm⁻¹).

The region of 1140–1080 cm⁻¹ is known [15, 16] to contain conformationally sensitive v_{as} (COC) modes of the macrocycle. In the spectrum of CuL₂, this region contains two strong bands at 1131 cm⁻¹ (with an inflection at 1106 cm⁻¹) and 1082 cm⁻¹ (Table 2). Their positions and intensity ratio are similar to those observed in the spectrum of ZnL₂ (1136 and 1082 cm⁻¹). We attrib

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Assignment	HL [6]	ZnL ₂ [9]	CuL ₂	$\begin{array}{c} 2\text{LiClO}_4 \\ \cdot \text{CuL}_2 \\ \cdot 2\text{H}_2\text{O} \end{array}$	$\begin{array}{c} 2NaNCS\\ \cdot \ CuL_2\\ \cdot \ 2H_2O \end{array}$	$\begin{array}{c} \text{KNCS} \\ \cdot \text{CuL}_2 \end{array}$	$2NaI \cdot CuL_2 \\ \cdot 2H_2O$	$\begin{array}{c} \text{KI} \cdot \text{CuL}_2 \\ \cdot \text{H}_2\text{O} \end{array}$
$\overline{\nu(H_2O)}$			-	3570 3522	3403		3406	3448
v(C≡N)					2056	2052		
v(C=N)	1613	1609	1606	1611	1610	1607	1609	1608
$\overline{\nu(CC)_{Ph}}$	1595	1594 1555	1591 1544	1597 1522	1596	1594	1596	1596
		1520			1552	1552	1553	1553
$v(CC)_{Ph}$	1502	1497	1509	1511	1507	1508	1508	1508
	1490	1488		1478	1480	1476	1478	1476
δ(CH ₂)		1473						
_	1453	1457	1463	1456		1457		
		1447		1444	1448	1442	1443	1443
		1436					1431	
	1423	1418	1405					1426
		1396		1401	1396	1401	1400	1402
$\omega(CH_2),$	1386							
v(CN)	1365	1362	1364	1358		1360	1358	1359
		1334 1319		1344	1344	1334	1344	
$v_{as}(SO_2)$	1338	1300	1303	1299	1295	1300	1299	1302
τ(CH ₂)	1298							
× 2/	1286	1256	1280	1285				
$\overline{v_s(PhO)}$	1265	1269	1263	1264	1261	1262	1261	1265br
$\overline{v_{as}(PhO)}$	1249	1244	1234	1231	1204	1204	1206	1203
δ(CH)	1212	1208	1200	1206	1204	1203	1203	
$\overline{\delta(CH)_{Ph}}$		1108						
	1167		1172				1162	1167
		1155		1158sh	1160	1158		
$\overline{\nu_s(SO_2)}$	1156	1136	1141	1131	1129	1126	1131	1138
$\overline{v_{as}(\text{COC})}$	1134	1136	1131	1131			1131	1138
	1128				1129	1126		1128
	1112	1111sh	1106sh					
	1100				1103		1104	
δ(CH) _{Ph}	1092			1099sh				
	1079	1083	1082	1082	1082	1081	1081	1082
$\nu(ClO_4)$				1084				
v(CO) + v(CC),	1063	1063						
ð(CH) _{Ph}	1053	1053	1052 1029		1050	1050	1055	1053
	1013	1018	1020					
	991							1001
	978	980	981	985	983		982	989

Table 2. Assignment of key vibration frequencies (cm^{-1}) in the IR spectra of CuL_2 and its complexes

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Table 2. (Contd.)

Assignment	HL [6]	ZnL ₂ [9]	CuL ₂	$\begin{array}{c} 2LiClO_4\\ \cdot CuL_2\\ \cdot 2H_2O\end{array}$	$\begin{array}{c} 2NaNCS\\ \cdot \ CuL_2\\ \cdot \ 2H_2O \end{array}$	$\begin{array}{c} KNCS \\ \cdot \ CuL_2 \end{array}$	$2NaI \cdot CuL_2 \\ \cdot 2H_2O$	$\begin{array}{c} \text{KI} \cdot \text{CuL}_2 \\ \cdot \text{H}_2\text{O} \end{array}$
v(CO) + v(CC)	951	947	945	945	943		945	943
$+ \rho(CH_2)$	943		938			936		938
		912	920			917		919
		901		905	902	903	903	906
$\overline{\rho(CH_2) + \nu(CO)},$	896		891					
$v_{puls}(M)$	878	877	876	880	880	872	882	872
	853	848						
	835		840	838	838	826	839	828
	812	815	815	816	816	814	815	
		800	802					
$\overline{ \begin{matrix} \nu_{puls}(A), \\ \delta(CH)_{Ph} \end{matrix} }$	796	795	795					
	764	762	769					
			755	756	758	755	760	758
	737		739	737				
	722	722	723	721		721	722	723
	704		708	707	707			708
δ(ClO ₄)				623				
Other vibrations	659	663	664	662	661	660	661	660
	636	645	642	648		642	642	641
	602	628			602			
		569	571	575	574	574	576	575
	565					563		
	549	556	551	553	549	551	551	552
	540	544					544	
		525			519		521	524
	509	511						
			490					
	474		481		478			
			415					

uted the band at 1136 cm⁻¹ in the spectrum of ZnL₂ to an $S_+T_-S_-$ conformation unit [8]. The somewhat lower position of the high-frequency component in the spectrum of CuL₂ (1131 cm⁻¹) suggests that CuL₂ macrocycles incorporate no ethylene glycol units with this configuration. The band at 1082 cm⁻¹ is probably due to the bending vibrations of the benzene rings.

Generally, the IR spectrum of CuL_2 is more complicated than the spectrum of ZnL_2 over the whole spectral region. Some bands are split. This is especially true for the range 1050–400 cm⁻¹ (Table 2). This may be caused by low symmetry of the molecule or specific molecular packing in the crystal and by different conformations of both macrocycles of the molecule. Thus this range in the spectrum of CuL_2 contains a split band (945 and 938 cm⁻¹) instead of a single band (947 cm⁻¹) present in the spectrum of ZnL_2 . Two new bends appear: a medium-intensity band (~920 cm⁻¹) and a strong band (~840 cm⁻¹). In the region of out-of-plane bending vibrations of the benzene rings (800–700 cm⁻¹), the number, the intensity ratio, and the positions of IR bands of CuL_2 and ZnL_2 are also different.

Analysis of the spectroscopic data suggests that the macrocycle conformation in CuL_2 differs from that in ZnL_2 .

The complexation of CuL_2 with alkali metal salts induces certain changes in the vibrational spectrum. However, in the IR spectra of alkali metal complexes with CuL₂, as in the spectra of complexes with ZnL₂, the bands due to vibrations of the benzene ring and SO₂ retain their positions and intensity ratios observed in the spectrum of uncoordinated CuL₂. The v(C=N) frequency of the azomethine group slightly increases as was also observed for ZnL₂ coordination (Table 2).

The most pronounced changes in the vibrational spectrum of CuL₂ upon complex formation are observed in conformationally sensitive regions and in the region of v(PhO) vibrations. For example, the v_{as} (PhO) frequency substantially decreases upon complexation, while the v_s (PhO) frequency remains almost unchanged. In the spectra of potassium and sodium complexes, v_{as} (PhO) decreases by ~30 cm⁻¹ and for 2LiClO₄ · CuL₂ · 2H₂O, by 18 cm⁻¹. This is somewhat greater than the decrease in v_{as} (PhO) following the formation of similar complexes with ZnL₂.

In the conformationally sensitive region of $v_{as}(COC)$ (1145-1080 cm⁻¹), the IR spectra of potassium complexes somewhat differ from the spectra of the sodium or lithium complexes. The spectrum of CuL_2 in the presence of potassium exhibits in this region two strong bands, at 1130 and 1083 cm⁻¹, like the spectrum of CuL_2 . In the case of potassium iodide complex, the high-frequency component has two peaks, 1138 and 1128 cm⁻¹. In the spectrum of the potassium thiocyanate complex, this region comprises an asymmetric singlet band at 1126 cm⁻¹. The IR spectra of the sodium complexes exhibit three bands in the $v_{as}(COC)$ region: two strong bands at 1130 and 1082 cm⁻¹ and one weak band at 1103 cm⁻¹ (Table 2). In the spectrum of the lithium complexes, a perchlorate stretching band is present in this region at ~1099 cm⁻¹, in addition to the $v_{as}(COC)$ bands at 1131 and 1082 cm⁻¹.

On the basis of correlations reported in the literature [9] for ZnL_2 , the bands in the range 1138–1131 cm⁻¹ in the spectra of all of the described complexes can be presumably assigned to ethylene glycol units with *trans*-configured C–C bonds in the macrocycles, and the bands near 1127 cm⁻¹ may be due to the presence of units with approximate *TGG* or *SGG* conformations.

In the other conformationally sensitive region (950– 800 cm⁻¹), the IR spectra of potassium complexes also differ somewhat from the spectra of sodium and lithium complexes. The spectra of potassium complexes exhibit in this region bands at 938 and 920 cm⁻¹, like the spectra of uncoordinated CuL₂. No such bands are present in the spectra of sodium or lithium complexes (Table 2).

The regions of out-of-plane bending vibrations of the benzene rings (800–690 cm⁻¹) in the spectra of complexes and CuL₂ are very similar (Table 2). Only the intensity ratios of some bands do change but the positions of bands are retained. An exception is the spectrum of $2NaNCS \cdot CuL_2 \cdot 2H_2O$, which contains only two bands at 758 and 707 cm⁻¹ in this region.

Note that the spectra of all complexes are simpler and smoother over the whole spectral region than the spectrum of CuL_2 , which may attest indirectly to a polymeric structure of complexes or to the presence of strong hydrogen bonds in the complexes or higher conformational uniformity of the OCCO units in the macrocycles.

In the complex $2\text{LiClO}_4 \cdot \text{CuL}_2 \cdot 2\text{H}_2\text{O}$, the $\text{ClO}_4^$ anion is not bound to the lithium ion, because its spectrum, like the spectrum of lithium perchlorate complex with ZnL₂, exhibits only two bands typical of this ion, $\nu(\text{ClO}_4)$ at ~1099 cm⁻¹ and $\delta(\text{ClO}_4)$ at ~623 cm⁻¹.

The NCS⁻ ion occupies an outer-sphere position in the sodium and potassium complexes, as $v(C\equiv N)$ is 2051 and 2056 cm⁻¹ for potassium and sodium complexes, respectively. In analogous ZnL₂ complexes, the thiocyanate group is most likely coordinated to the metal (2071 cm⁻¹ for 2NaNCS \cdot ZnL₂ and 2075 cm⁻¹ for KNCS \cdot ZnL₂).

It was shown [17] that, for alkali metal complexes with cyclic polyethers, the stretching frequencies of coordinated water are higher than v(H₂O) for the water involved in hydrogen bonding to anions. The pattern of absorption in the region of stretching vibrations of water molecules in the spectra of 2NaNCS \cdot CuL₂ \cdot 2H₂O, 2NaI \cdot CuL₂ \cdot 2H₂O, and KI \cdot CuL₂ \cdot H₂O (broad asymmetric bands at 3403, 3406, and 3448 cm⁻¹, respectively) indicates that water molecules in these complexes are apparently inner-sphere ligands or are involved in hydrogen bonds. The conclusions based on analysis of vibrational spectra are confirmed by thermogravimetric data. It was found that water is removed at a rather high temperature with simultaneous decomposition of complexes:

 $\begin{array}{l} \text{KI} \cdot \text{CuL}_2 \cdot \text{H}_2\text{O} - 360^\circ\text{C}, 2\text{NaNCS} \cdot \text{CuL}_2 \cdot 2\text{H}_2\text{O} - 275^\circ\text{C}, \\ 2\text{NaI} \cdot \text{CuL}_2 \cdot 2\text{H}_2\text{O} - 300^\circ\text{C}. \end{array}$

Analysis of the results leads to the assumption that coordination of lithium and sodium cations to the macrocycle oxygen atoms is accompanied by incorporation of the metal in the macrocycle cavity. This results in a change in the conformation, apparently, of only several ethylene glycol units. In the case of potassium complexes, sandwich structures are probably formed.

Macrocyclic polyethers are efficient ion transport agents, in particular, in polymer membranes [18]. This feature of crown compounds can be used for the design of ion-selective electrodes. Previously [9], we studied HL and ZnL₂ as the active components of plasticized polymeric (poly(vinyl chloride)) membranes. In this work, we carried out similar studies for CuL₂. The potentiometric selectivity sequence for CuL₂ in comparison with the data for HL and ZnL_2 is presented below:

HL:
$$Pb^{2+} > Ag^+ > K^+ > Rb^+ > Cu^{2+} > Na^+ \approx Cs^+ \approx Sr^{2+}$$

> $NH_4^+ > Mg^{2+}$ [9],

$$ZnL_2: Ag^+ > Pb^{2+} > K^+ > Cu^{2+} > Zn^{2+} \approx Rb^+ > Cs^+ \approx Ba^{2+}$$
$$\approx Cd^{2+} > Li^+ \approx Na^+ \approx Ca^{2+} \approx NH_4^+ [9],$$

$$CuL_{2}: Cu^{2+} > K^{+} > Mn^{2+} > NH_{4}^{+} > Li^{+} \approx Na^{+} > Rb^{+} \approx Cs^{+} > Pb^{2+} > Cd^{2+} > Co^{2+} \approx Ni^{2+} > Ba^{2+} > Ag^{+}$$

(the selectivity decreases in these sequences from left to right, i.e., the ions toward which the ligands show the highest selectivity are situated in the beginning of the sequence).

It can be seen from analysis of the above selectivity sequences that coordination of HL by transition metals through the azomethine moiety entails a pronounced change in its selective properties, the selectivity being dependent on the nature of the metal in the chelate. The replacement of zinc by copper results in a significant change in the selectivity sequence: ZnL_2 is most selective toward Ag⁺ and Pb²⁺ ions, whereas CuL₂ is selective toward Cu²⁺ and is not selective toward Pb²⁺. This is apparently related to the difference in the geometry of ZnL₂ and CuL₂ molecules and in the conformation of their macrocycles. Thus, by changing the nature of the metal in the chelate, it is possible to change deliberately the selectivity of neutral transport agents.

The studies have shown that the complexation of CuL_2 with alkali metal salts form complex heteronuclear structures similar to those formed with ZnL_2 complexes where lithium and sodium ions reside in the macrocycle cavity of the chelate, while the potassium ions coordinate the oxygen atoms of two macrocycles, apparently, of neighboring CuL_2 molecules to give brickwork-type sandwich structures typical of potassium complexes of crown-containing phtalocyanines [19].

The change in the nature of the metal incorporated in the chelate ring on HL coordination through the azomethine moiety virtually does not affect the complexing properties of the macrocyclic donor center but somewhat changes the macrocycle conformation. The selectivity of these chelates with respect to ion transport in plasticized poly(vinyl chloride) membranes markedly changes.

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