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DIFORMAZYL DERIVATIVES OF DIBENZO-18-CROWN-6

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New derivatives of the macrocyclic polyether dibenzo-18-crown-6 that contain formazyl groupings in the aromatic rings were synthesized. The complexing ability of the polyether ring and the formazyl groupings was studied. Complexes of two types, viz., those that contain a transition metal and those that simultaneously contain a transition metal and an alkali or alkaline earth metal, were isolated in crystalline form.

Macrocyclic polyethers occupy a special place among complexing compounds as a consequence of their ability to form ion–dipole complexes with salts of various metals, including alkali and alkaline earth metals. Representatives of this class of compounds have found ex-

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TABLE 1. Results of Elementary Analysis of Diformazyl Derivatives of Dibenzo-18-crown-6

Compound	Found, %			Empirical formula	Calc., %		
	C	H	N		C	H	N
I	68,8	5,4	13,6	$C_{44}H_{44}N_8O_6$	68,6	5,5	13,9
II	58,8	4,8	17,2	$C_{40}H_{38}N_{10}O_6S_2$	58,7	4,6	17,1
III	58,0	4,8	13,4	$C_{40}H_{40}N_8O_{10}$	58,2	4,8	13,5
IV	49,3	4,6	12,0	$C_{38}H_{40}N_8O_{16}S_2$	49,1	4,3	12,1
V	57,3	4,6	17,3	$C_{38}H_{34}N_{10}O_{10}$	57,7	4,3	17,7
VI	51,8	5,1	11,0	$C_{44}H_{50}Cl_2N_8O_{16}$	51,7	5,3	11,0
VII	59,2	5,4	14,2	$C_{38}H_{40}N_8O_{10}$	59,4	5,2	14,6

TABLE 2. Complexes of Diformazyl Derivatives of Dibenzo-18-crown-6 with Nickel and Cobalt Salts

Compound	Starting substan		Composition of the complex L:M	Found, %			Empirical formula	Calc., %		
	compound	salt of transition metal		C	H	N		C	H	N
VIII	I	$Co(CH_3COO)_2$	1 : 2	57,8	4,9	10,9	$C_{44}H_{44}N_8O_6 \cdot Co(CH_3COO)_2$	57,8	4,7	10,8
IX	I	$Ni(CH_3COO)_2$	1 : 2	57,3	5,1	10,4	$C_{44}H_{44}N_8O_6 \cdot Ni(CH_3COO)_2$	57,8	4,7	10,7
X	I	$Ni(NO_3)_2$	1 : 1	63,6	5,0	12,7	$C_{44}H_{44}N_8O_6 \cdot Ni(NO_3)_2$	63,7	5,0	12,8
XI	II	$Ni(CH_3COO)_2$	1 : 2	49,9	3,8	13,8	$C_{40}H_{38}N_{10}O_6S_2 \cdot Ni(CH_3COO)_2$	50,2	4,1	13,3
XII	III	$Ni(CH_3COO)_2$	1 : 2	50,3	4,0	10,1	$C_{40}H_{40}N_8O_{10} \cdot Ni(CH_3COO)_2$	49,9	4,2	10,6
XIII	III	$Ni(NO_3)_2$	1 : 1	53,3	4,6	12,4	$C_{40}H_{40}N_8O_{10} \cdot Ni(NO_3)_2$	53,4	4,5	12,5
XIV	IV	$Co(CH_3COO)_2$	1 : 2	43,0	3,5	9,3	$C_{38}H_{40}N_8O_{16}S_2 \cdot Co(CH_3COO)_2$	43,4	3,8	9,6
XV	V	$Ni(NO_3)_2$	1 : 1	53,7	4,1	16,2	$C_{38}H_{34}N_{10}O_{10} \cdot Ni(NO_3)_2$	53,8	3,9	16,5
XVI	VI	$Co(CH_3COO)_2$	1 : 2	45,3	4,4	8,6	$C_{44}H_{50}Cl_2N_8O_{16} \cdot Co(CH_3COO)_2$	45,6	4,7	8,9
XVII	VII	$Co(CH_3COO)_2$	1 : 2	50,3	4,4	11,2	$C_{38}H_{40}N_8O_{10} \cdot Co(CH_3COO)_2$	50,3	4,4	11,2
XVIII	VII	$Ni(CH_3COO)_2$	1 : 2	50,3	4,2	11,4	$C_{38}H_{40}N_8O_{10} \cdot Ni(CH_3COO)_2$	50,4	4,4	11,2
XIX	VII	$Ni(NO_3)_2$	1 : 1	54,5	4,5	13,9	$C_{38}H_{40}N_8O_{10} \cdot Ni(NO_3)_2$	54,7	4,56	13,8

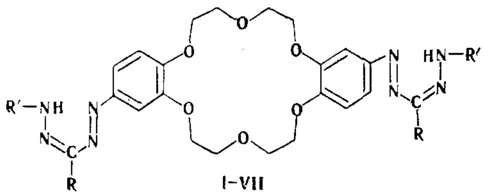
TABLE 3. Complexes of Diformazyl Derivatives with Two Metals

Compound	Starting substance		Composition of the complex, L:M:M'	Found, %			Empirical formula	Calc., %		
	compound	alkali metal or alkaline earth		C	H	N		C	H	N
XX	VIII	$Ba(NO_3)_2$	1 : 2 : 1	45,9	4,0	—	$C_{44}H_{44}N_8O_6 \cdot Co(CH_3COO)_2 \times$ $\times Ba(NO_3)_2$	46,2	3,7	—
XXI	XI	KCNS	1 : 2 : 1	46,9	3,9	13,2	$C_{40}H_{38}N_{10}O_6S_2 \cdot Ni(CH_3COO)_2 \times$ $\times KCNS$	47,0	3,7	13,4
XXII	XIII	KCNS	1 : 1 : 1	50,4	4,0	12,7	$C_{40}H_{40}N_8O_{10} \cdot Ni(NO_3)_2 \cdot KCNS$	50,3	3,9	12,9
XXIII	XIV	KI	1 : 2 : 1	37,5	3,2	8,5	$C_{38}H_{40}N_8O_{16}S_2 \cdot Co(CH_3COO)_3 \times$ $\times KI$	37,9	3,4	8,4
XXIV	XV	KI	1 : 1 : 1	44,6	3,5	13,7	$C_{38}H_{34}N_{10}O_{10} \cdot Ni(NO_3)_2 \cdot KI$	45,0	3,3	13,8
XXV	XVI	KI	1 : 2 : 1	40,7	4,4	7,7	$C_{44}H_{50}Cl_2N_8O_{16} \cdot Co(CH_3COO)_2 \times$ $\times KI$	40,5	4,3	7,9
XXVI	XVII	KI	1 : 2 : 1	43,2	3,8	9,4	$C_{38}H_{40}N_8O_{10} \cdot Co(CH_3COO)_2 \cdot KI$	43,2	4,2	9,7

tensive application in preparative organic and analytical chemistry, as catalysts for chemical processes and extractants of metals, and in other fields of chemistry [1]. The testing of the biological properties of macrocyclic polyethers is of equal significance. It is known that dibenzo-18-crown-6 derivatives that contain thiourea and azomethine groupings in the aromatic rings display tuberculostatic activity [2].

In the present paper we describe the synthesis of new dibenzo-18-crown-6 derivatives with formazyl complexing groupings in the aromatic rings and the results of a study of their ability to undergo complexing simultaneously with two different metals. Up until recently, information regarding compounds of this sort was not available in the literature [3].

Macrocyclic polyethers form complexes with salts of metals of primarily alkali and alkaline earth metals, while formazans form complexes with salts of transition metals. The combination of two complexing centers in a single molecule made it possible to propose the possibility of the simultaneous complexing by one molecule of two different metals, viz., a transition metal and an alkali or alkaline earth metal.



I-VII

I $R=R'=C_6H_5$; II $R=C_6H_5$, $R'=$ 4-thiazolyl III $R=COCH_3$, $R'=o\text{-}C_6H_4COOH$; IV $R=COCH_3$, $R'=$2-hydroxy-5-sulfophenyl V $R=CN$, $R'=o\text{-}C_6H_4COOH$; VI $R=$D-galacto-$C_5H_{11}O_5$, $R'=o\text{-}ClC_6H_4$; VII $R=COCH_3$, $R'=o\text{-}C_6H_4OH$

Dibenzo-18-crown-6 derivatives with formazyl groupings in the aromatic rings were synthesized by diazotization of diaminodibenzo-18-crown-6 hydrochloride with subsequent coupling of the diazo compound with various hydrazones in an alkaline medium. The reaction products were obtained in the form of bright red to dark cherry-red crystals in 45-73% yields after acidification of the reaction mixtures with acetic acid.

The formation of formazyl groupings is confirmed by the development of a blue coloration when the compounds obtained are treated with concentrated sulfuric acid and by decolorization when they are treated with a solution of lead tetraacetate. The presence of formazyl substituents in the dibenzo-18-crown-6 molecule was also proved by the IR spectra, which contain absorption bands at 1460-1470 (N=N) and 1495-1510 cm^{-1} (C=N). The band at 3100-3400 cm^{-1}, which corresponds to stretching vibrations of NH groups, is absent in the spectra of these compounds. This is explained by the fact that the derivatives with formazyl substituents, like the analogous formazans without a polyether macrocycle, have an intramolecular hydrogen bond.

Complexes of dibenzo-18-crown-6 derivatives with formazyl groupings of two types were obtained: those that contain only a transition metal, and those that simultaneously contain a transition metal and an alkali or alkaline earth metal. Both types of complexes were isolated in crystalline form. Their synthesis was carried out as follows. Complexes with transition metals involving the formazyl groups were obtained initially by adding a metal salt to a solution of the corresponding diformazyl derivative of dibenzo-18-crown-6 (I-VII). The complexes involving the formazyl groupings are dark brown to dark cherry-red or dark blue crystals with a metallic luster that are soluble in dimethylformamide (DMF), only slightly soluble in methylene chloride and methanol, and insoluble in water and ether. Complexes of the diformazyl derivatives with two metals (XX-XXVI) were then synthesized. The same changes in the intensities and the shift of the absorption maxima of the ether bonds of the macrocyclic ring as those described in [4] are observed in the IR spectra as a result of complexing.

Thus the introduction of formazyl groupings in the aromatic rings of dibenzo-18-crown-6 results in the simultaneous retention of the complexing ability of the polyether ring and the formazyl groupings. These compounds may find application as complexing agents in, for example, analytical and organic chemistry and medicine and as catalysts and dyes.

EXPERIMENTAL

The IR spectra of mineral oil pastes and KBr pellets of the compounds were recorded with a UR-20 spectrometer. The UV spectra were recorded with an SF-4 spectrophotometer.

3,3'-Bis(3,5-diphenylformazyl)dibenzo-18-crown-6 (I). A 0.5-ml sample of concentrated hydrochloric acid was added to 0.39 g (0.001 mole) of 3,3'-diaminodibenzo-18-crown-6 [2] in 1 ml of water, and the compound was diazotized with a solution of 0.14 g of sodium nitrite in 2 ml of water at −5°C. The diazo solution was added with stirring to 0.5 g (0.002 mole) of benzaldehyde phenylhydrazone in 40 ml of methanol at pH 8-9 and −8°C, during which the color of the reaction mixture changed from yellow to bright red. The mixture was then stirred with cooling for 2 h, after which it was acidified to pH 6-7 with acetic acid. The resulting dark red precipitate was removed by filtration, washed with 500 ml of water and 20 ml of hot methanol, and dried *in vacuo* over P_2O_5 and paraffin. The reaction product was soluble in chloroform, ethanol, and DMF but insoluble in ether and water. The yield was 0.57 g (73%). IR spectrum: 1003, 1240, 1065, 1140 (C—O—C); 770, 1465, 1510 (C_6H_5); 1465 (N=N); 1510 cm^{-1} (C=N). UV spectrum in chloroform, λ_{max}: 279, 306, and 345 nm.

3,3'-Bis[(3-phenyl-5-thiazolyl)formazyl]dibenzo-18-crown-6 (II). A 0.39-g (0.001 mole) sample of 3,3'-diaminodibenzo-18-crown-6 diazotized as described above was added at pH 8-9 to a cooled (to −8°C) solution of 0.4 g (0.002 mole) of benzaldehyde 2-thiazolylhydrazone in 30 ml of methanol, after which stirring was continued for 2 h, and 100 ml of water was added gradually. The precipitate was removed by filtration and washed to neutrality with water. Crystallization from methanol gave dark brown crystals that were soluble in alcohol, chloroform, and DMF but insoluble in water and ether. They were dried *in vacuo* to give 0.5 g (61%) of product. IR spectrum: 1010, 1240, 1065, 1140 (C—O—C); 1460 (N=N); 1510 cm^{-1} (C=N). UV spectrum in chloroform, λ_{max}: 267 and 306 nm.

3,3'-Bis[3-acetyl-5-(2-carboxyphenyl)formazyl]dibenzo-18-crown-6 (III). A 0.39-g (0.001 mole) sample of diazotized 3,3'-diaminodibenzo-18-crown-6 was added with vigorous stirring at −8°C to a solution of 0.5 g (0.002 mole) of methylglyoxal 2-carboxyphenylhydrazone in 50 ml of 30% alkali (pH 8-9), during which the reaction mixture turned bright red. Stirring was continued for 2 h, after which the reaction mixture was acidified to pH 6-7, and the reaction product was isolated as in the case of VII and dried *in vacuo* to give dark red crystals that were soluble in ethanol but insoluble in water and ether. The yield was 0.48 g (63%). IR spectrum: 1025, 1240, 1065, 1140 (C-O-C); 1460, 1590 (C_6H_4); 1460 (N=N); 1495 (C=N); 1685 (C=O); 1460 (OH in COOH); 1685 cm^{-1} (C=O in COOH). UV spectrum in ethanol, λ_{max}: 263 and 319 nm.

3,3'-Bis[3-acetyl-5-(2-hydroxy-5-sulfophenyl)formazyl]dibenzo-18-crown-6 (IV). A solution of 0.66 g(0.002 mole) of the trisodium salt of methylglyoxal 2-hydroxy-5-sulfophenylhydrazone in 40 ml of 30% alkali was cooled to −8°C, and 0.39 g (0.001 mole) of diazotized 3,3'-diaminodibenzo-18-crown-6 was added during which the reaction mixture turned bright red. Stirring was continued for 2 h, and the mixture was acidified to pH 6-7 with acetic acid. Workup was carried out as in the preparation of VII, and the product was dried *in vacuo* over P_2O_5 and paraffin to give dark cherry-red crystals that were quite soluble in water and DMF but insoluble in ethanol, ether, and benzene. The yield was 0.65 g (70%). IR spectrum: 1003, 1240, 1140 (C—O—C); 1460, 1510, 1600 (C_6H_3); 1465 (N=N); 1510 (C=N); 1670 cm^{-1} (C=O). UV spectrum in water, λ_{max}: 274 and 338 nm.

3,3'-Bis[3-cyano-5-(2-carboxyphenyl)formazyl]dibenzo-18-crown-6 (V). A 0.39-g (0.001 mole) sample of 3,3'-diaminodibenzo-18-crown-6 was diazotized and coupled with 0.37 g (0.002 mole) of cyanoglyoxal 2-carboxyphenylhydrazone in 50 ml of 30% alkali (pH 8-9) at −5°C. The mixture was then stirred in the cold for 2 h, after which it was acidified to pH 6-7 with acetic acid. The precipitate was removed by filtration and washed with water and methanol to give dark red crystals, which were reprecipitated from DMF by the addition of ether and dried *in vacuo*. The product was soluble in DMF but insoluble in water, ether, and ethanol. The yield was 0.47 g (58%). IR spectrum: 1015, 1240, 1065, 1140 (C—O—C); 770, 1460, 1510, 1590 (C_6H_4); 1460 (N=N); 1510 (C=N); 3410, 1675 cm^{-1} (OH and C=O in COOH). UV spectrum in ethanol, λ_{max}: 275 and 296 nm.

3,3'-Bis[3-(D-galacto-pentahydroxypentyl)-5-(4-chlorophenyl)formazyl]dibenzo-18-crown-6 (VI). Water (6 ml) was added to a solution of 0.61 g (0.002 mole) of galactose 4-chlorophenylhydrazone in 6 ml of pyridine, and the reaction mixture was cooled to −5°C and coupled with 0.39 g (0.001 mole) of diazotized 3,3'-diaminodibenzo-18-crown-6. Stirring was continued for 3 min in the cold, and the mixture was poured into 100 ml of ice water. After 2 h, the precipitate was removed by filtration and washed with water and methanol. The dark red crystals were dried *in vacuo*. The product was soluble in ethanol, chloroform, dichloroethane, and DMF but insoluble in water, ether, and methylene chloride. The yield was 0.46 g (45%). IR spectrum: 1015, 1240, 1065, 1140 (C—O—C); 1465 (N=N); 1510 (C=N); 750 cm^{-1} (C-Cl). UV spectrum in ethanol, λ_{max}: 268 and 305 nm.

3,3'-Bis[3-acetyl-5-(2-hydroxyphenyl)formazyl]dibenzo-18-crown-6 (VII). A 0.39-g (0.001 mole) sample of 3,3'diaminodibenzo-18-crown-6 diazotized as described above was added at −8°C to a solution of 0.45 g (0.002 mole) of methylglyoxal 2-hydroxyphenylhydrazone in 50 ml of water and 30% alkali (pH 8-9), and the bright red reaction mixture was stirred in the cold for 2 h. It was then acidified to pH 6-7 with acetic acid, and the precipitate was removed by filtration, washed with 2 liters of water and 20 ml of methanol, and dried *in vacuo*. The dark-red crystals were soluble in alcohol and DMF but insolbule in water. The yield was 0.63 g (65%). IR spectrum: 1025, 1240, 1065, 1140 (C—O—C); 1470, 1510, 1600 (C_6H_4); 1470 (N=N); 1510 (C=N); 1690 (C=O); 3400 cm^{-1} (OH). UV spectrum in ethanol, λ_{max}: 243, 263, and 321 nm.

The results of elementary analysis of I-VII are presented in Table 1.

Complexes of Diformazyl Derivatives of Dibenzo-18-crown-6 (I-VII) with Transition Metals (VIII-XIX) (Table 2). A 0.005-mole sample of the transition metal salt in 3 ml of water was added to a hot solution of 0.002 mole of I-VII in 200 ml of methanol, and the reaction mixture was heated on a water bath at 65-70°C for 1 h and allowed to stand overnight. The precipitate was removed by filtration, washed with 3% acetic acid and water, and dried *in vacuo*, The products were obtained in 56-75% yields.

Complexes of Diformazyl Derivatives of Dibenzo-18-crown-6 with Transition and Alakli or Alkaline-Earth Metals (XX-XXVI) (Table 3). A 0.002-mole sample of complex VIII-XIX and 0.002 mole of the salt of an alkali or alkaline earth metal were dissolved by heating in DMF, and the reaction mixture was heated on a water bath for 10-15 min. The solvent was removed by distillation, and the residue was dried *in vacuo*. The yields were quantitative.

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CRYSTAL AND MOLECULAR STRUCTURE OF CHELATE COMPOUNDS OF Co(II) AND Ni(II) WITH (2-MERCAPTO-5-ETHYL-3-THENYLIDENE)CYCLOHEXYLAMINE

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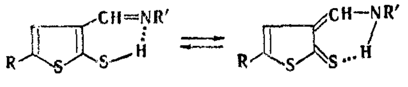
Chelate compounds of Co(II) and Ni(II) with (2-mercapto-5-ethyl-3-thenylidene)-cyclohexylamine were subjected to x-ray diffraction analysis. It was established that the investigated chelates are isostructural and that the metal atoms are tetrahedrally coordinated. It is shown that an intermediate distribution of the multiple bonds is realized in the examined chelate compound, just as in the starting ligand, and that their structures cannot be described within the framework of canonical structures.

A study of the tautomerism of mercaptoaldimines of the thiophene series — new ligands of the I type — has shown that in solutions of these compounds the equilibria are shifted virtually completely to favor the thionamine form with an intramolecular NH...S hydrogen bond [1], whereas in the crystalline state the distribution of the bond lengths constitute evidence for the contribution of both tautomeric forms and the absence of preponderance of one of them [2].



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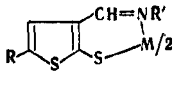

II,III

R=H, alkyl R'=*cyclo*-C_6H_{11}; II M=Co(II); III M=Ni(II)

In this connection, it seemed of interest to study the structures of the chelate compounds formed by these ligands to obtain a solution to the problem as to the presence or absence of tautomeric forms in the metal complexes and the effect of complexing on the stereochemistry of the ligand.

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