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Zn Complex with *N*-(4'-Benzo-15-crown-5)-2-(amino-*N*-tosyl)-phenylaldimine: Synthesis, Crystal Structure, and Vibration Spectrum

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Abstract—The ZnL_2 complex (**I**) (HL = *N*-(4'-benzo-15-crown-5)-2-(amino-*N*-tosyl)phenylaldimine) is synthesized and studied by IR spectroscopy and X-ray diffraction analysis. The crystals are monoclinic: $a = 23.139(2)$ Å, $b = 19.931(2)$ Å, $c = 14.604(2)$ Å, $\beta = 121.20(1)°$, $Z = 4$, space group $C2/c$, $R = 0.751$ for 766 reflections with $I > 2\sigma(I)$. The Zn atom in a molecule of the complex lies on a twofold axis and is coordinated at the vertices of a distorted tetrahedron by four N atoms of two HL ligands (average Zn–N distance 2.024 Å).

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Previously, we reported the results of the study of crystal structure and vibration spectra of *N*-(4'-benzo-15-crown-5)-2-(amino-*N*-tosyl)-phenylaldimine HL (**II**) [1] and *N*-(4'-benzo-15-crown-5)-5-bromo-2-hydroxyphenylaldimine (**III**) [2]. This work continues our studies of the structure, complexing and physicochemical properties of azomethin derivatives of benzo-15-crown-5 and describes the synthesis, IR and X-ray diffraction data for Zn complex with *N*-(4'-benzo-15-crown-5)-2-(amino-*N*-tosyl)-phenylaldimine, ZnL_2 (**I**).

EXPERIMENTAL

Synthesis of I. Zinc acetate (0.01 mol) was added to a solution of *N*-(4'-benzo-15-crown-5)-2-(amino-*N*-tosyl)-phenylaldimine (0.1 g, 0.02 mol) in 10 ml of methanol, and the mixture was refluxed for 2 h on water bath. The precipitate formed was filtered off and recrystallized from an alcohol.

For $C_{56}H_{62}N_4O_{14}S_2Zn$

anal. calcd. (%):	C, 58.76;	H, 5.46;	N, 4.89.
Found (%):	C, 58.54;	H, 4.84;	N, 4.72.

IR spectra were recorded on a FT-IR NEXUS Fourier spectrometer (Nicolet) (with essential oil and hexachlorobutadiene mulls) at 4000–400 cm^{-1}.

X-ray diffraction analysis. Crystals **I** are monoclinic ($M = 1144.59$): $a = 23.139(2)$, $b = 19.931(2)$, $c = 14.604(2)$ Å, $\beta = 121.20(1)°$, $V = 5760.9(5)$ Å^3, ρ(calcd.) = 1.320 g/cm^3, $\mu(MoK_\alpha) = 0.565$ mm^{-1}, $F(000) = 2400$, $Z = 4$, space group $C2/c$.

The experimental material (5175 reflections including 5048 independent reflections) was collected on automated Enraf-Nonius CAD4 diffractometer (room temperature, graphite monochromator, ω-scan mode, $\theta_{max} = 25.0°$) from very fine plate-like crystal $0.053 \times 0.053 \times 0.049$ mm in size. Reflections were collected in a range of indices $0 \le h \le 27$, $0 \le k \le 23$, $-17 \le 1 \le 14$. The experimental data were corrected for the Lorentz and polarization effects. The absorption anisotropy was not taken into account because of the low absorption coefficient.

The structure was solved by the direct methods (SHELXS93 [3]) and refined by the least-squares method on F^2 (SHELXL97 [4]) in a full-matrix anisotropic approximation for all non-hydrogen atoms. Benzene rings in a ligand were refined by a heavy body method. The hydrogen atoms were located geometrically ($C(sp^2)$–H 0.93, $C(sp^3)$–H 0.97 Å) and refined in a rider model with isotropic thermal parameters U_H, which are 1.2 times (1.5 times for the H atoms (CH_3)) as high as U_C for the C atom to which this H atom is bonded. The refinement was restrained by the tendency of some O–C and C–C bonds in crown ether to have standard lengths. It should be noted that the crystal quality was very poor and the presence of a twinning admixture was not excluded (the high value of R_{int} = 0.5397), while the number of reflections with $I > 2\sigma(I)$ was low (15.2% of the total number of independent reflections) as compared to a total number of the refined parameters.

The final values of R-factors were: $R_1 = 0.0751$, wR_2 = 0,0922, GOOF = 0.846 on 766 reflections with $I > 2\sigma(I)$; $R_1 = 0.5859$, $wR_2 = 0.1713$ on all independent reflections. The total number of the refined parameters was 313, the values of $\Delta\rho_{\max}$ and $\Delta\rho_{\min}$ were 0.339 and –0.966 e A^{-3}, respectively.

The coordinates of atoms and their thermal parameters in structure **I** are given in Table 1, the bond lengths and bond angles are listed in Table 2.

RESULTS AND DISCUSSION

Crystal **I** consists of individual monomer molecules ZnL_2 (Fig. 1). The Zn atom lies on a twofold axis and is coordinated at the vertices of a distorted tetrahedron by two imine N atoms and by two N atoms, bonded to tosyl groups, of two deprotonated bidentate L^- ligands. The lengths of two independent Zn–N bonds are equal to 2.00(1) and 2.05(1) Å. The substantial angular distortions of a tetrahedron occur mainly as a result of coordination of the ligand L^- and a closure of a six-membered metal ring (the chelate angle M(1)ZnN(2) 95.2(4)°, the remaining NZnN angles 100.8°–128.5°). The ZnN(2)C(15)C(16)C(21)N(1) chelate ring is almost planar (the average deviation of atoms ($\boldsymbol{\Delta}_{\mathbf{av}}$) from the chelate plane being 0.019 Å).

The geometries of a deprotonated ligand L^- in **I** and of the HL molecule in **II** slightly differ in the arrangement of phenyl rings about the crown ether plane. In complex **II**, the dihedral angles between mean planes of a substituted benzo-15-crown-5 (**B15C5**) (Δ_{av} = 0.307 Å) and of the phenyl rings C(16)–C(21) (1, Δ_{av} = 0.000 Å), C(22)–C(27) (2, Δ_{av} = 0.000 Å), C(22)–C(27) (2, Δ_{av} = 0.000 Å) are equal to 30.5° and 113.0°, respectively (in **II**, they are 54.1° and 119.7°). The mutual arrangement of the Ph-rings in the two structures is similar: the dihedral angle 1/2 is equal to 85.5° and 83.3° in **I** and **II**, respectively.

A microcycle in complex **I**, as in **II**, in **III** with close composition, and in a free B15C5(**IV**) [5], is of an alternate type. In complex **I**, the average deviations of the O atoms of a substituted B15C5 from its mean plane (Δ_{av} = 0.231 Å) are equal to (figures given in parentheses refer to complex **II**): O(1) 0.137 (0.027), O(2) –0.271 (0.250), O(3) 0.314 (–0.407), O(4) –0.306 (0.326), O(5) 0.127 (–0.196) Å.

Table 1. The coordinates of atoms ($\times10^4$) and their thermal parameters U_{eq} ($\times10^3$) in structure **I**

Atom	x	y	z	U_{eq}, Å^2
Zn	5000	4642(1)	7500	49(1)
S	6095(2)	3711(2)	9030(3)	52(1)
O(1)	2806(5)	6128(5)	5149(7)	64(3)
O(2)	1541(6)	5567(6)	4024(12)	130(5)
O(3)	642(9)	6833(10)	2042(18)	394(17)
O(4)	1866(8)	7435(6)	2122(11)	112(5)
O(5)	2991(5)	7114(5)	4165(8)	72(3)
O(6)	5518(4)	3749(4)	9170(6)	60(3)
O(7)	6745(4)	3871(4)	9947(6)	64(3)
N(1)	5892(5)	4207(5)	7995(7)	34(3)
N(2)	5064(6)	5298(6)	6475(7)	46(3)
C(1)	3543(6)	6705(5)	4747(7)	42(4)
C(2)	4156(7)	6763(4)	4792(7)	77(6)
C(3)	4667(5)	6299(6)	5369(8)	47(5)
C(4)	4565(5)	5777(5)	5900(7)	40(4)
C(5)	3952(7)	5718(4)	5855(7)	57(5)
C(6)	3441(4)	6182(6)	5278(8)	44(4)
C(7)	2613(7)	5556(8)	5451(11)	72(5)
C(8)	1922(8)	5606(10)	5165(12)	122(8)
C(9)	1032(11)	6074(16)	3512(16)	330(20)
C(10)	1038(14)	6293(12)	2500(20)	460(30)
C(11)	970(10)	7482(11)	2357(15)	296(18)
C(12)	1177(11)	7649(10)	1557(17)	217(18)
C(13)	2348(10)	7906(10)	2823(16)	115(8)
C(14)	3019(8)	7570(8)	3444(12)	84(6)
C(15)	5571(7)	5242(7)	6337(9)	46(4)
C(16)	6192(4)	4847(4)	6897(8)	43(4)
C(17)	6658(6)	4990(4)	6596(6)	44(4)
C(18)	7262(5)	4636(5)	7048(7)	51(4)
C(19)	7400(4)	4139(5)	7802(7)	50(4)
C(20)	6934(6)	3996(4)	8104(6)	52(5)
C(21)	6330(5)	4350(5)	7652(8)	54(5)
C(22)	6143(7)	2884(5)	8624(12)	56(5)
C(23)	6580(5)	2410(9)	9355(7)	100(6)
C(24)	6583(6)	1758(7)	9022(12)	123(8)
C(25)	6149(8)	1578(4)	7958(14)	112(8)
C(26)	5713(6)	2052(7)	7228(7)	72(6)
C(27)	5710(5)	2704(6)	7560(10)	59(5)
C(28)	6184(11)	878(7)	7602(15)	187(11)

Table 2. The main bond lengths and bond angles in structure **I***

Bond	*d*, Å	Bond	*d*, Å
Zn–N(1)	1.997(10)	Zn–N(2)	2.050(11)
S–O(7)	1.438(8)	S–O(6)	1.453(8)
S–N(1)	1.657(9)	S–C(22)	1.776(9)
O(1)–C(6)	1.39(1)	O(1)–C(7)	1.38(1)
O(2)–C(8)	1.43(1)	O(2)–C(9)	1.43(2)
O(3)–C(10)	1.34(2)	O(3)–C(11)	1.45(2)
O(4)–C(13)	1.41(2)	O(4)–C(12)	1.43(2)
O(5)–C(1)	1.38(1)	O(5)–C(14)	1.42(1)
N(1)–C(21)	1.37(1)	N(2)–C(15)	1.29(1)
N(2)–C(4)	1.39(1)	C(7)–C(8)	1.43(1)
C(9)–C(10)	1.56(2)	C(11)–C(12)	1.51(2)
C(13)–C(14)	1.49(2)	C(15)–C(16)	1.46(1)
C(25)–C(28)	1.51(1)		
Angle	ω, deg	Angle	ω, deg
N(1)$^{\#1}$ZnN(1)	128.5(6)	N(1)$^{\#1}$ZnN(2)$^{\#1}$	95.2(4)
N(1)$^{\#1}$ZnN(2)	117.7(4)	N(2)$^{\#1}$ZnN(2)	100.8(6)
O(7)SO(6)	116.8(5)	O(7)SN(1)	112.5(6)
O(6)SN(1)	104.5(5)	O(7)SC(22)	107.5(6)
O(6)SC(22)	108.4(6)	N(1)SC(22)	106.6(6)
C(6)O(1)C(7)	121(1)	C(8)O(2)C(9)	115(2)
C(10)O(3)C(11)	117(2)	C(13)O(4)C(12)	117(2)
C(1)O(5)C(14)	117(1)	C(21)N(1)S	122.8(8)
C(21)N(1)Zn	126.6(8)	SN(1)Zn	110.0(5)
C(15)N(2)C(4)	120(1)	C(15)N(2)Zn	118(1)
C(4)N(2)Zn	121.7(8)	O(5)C(1)C(2)	125(1)
O(5)C(1)C(6)	115(1)	C(5)C(4)N(2)	119(1)
C(3)C(4)N(2)	122(1)	O(1)C(6)C(5)	123(1)
O(1)C(6)C(1)	117(1)	O(1)C(7)C(8)	110(1)
O(2)C(8)C(7)	105(1)	O(2)C(9)C(10)	107(2)
O(3)C(10)C(9)	111(2)	O(3)C(11)C(12)	106(1)
O(4)C(12)C(11)	101(2)	O(4)C(13)C(14)	109(2)
O(5)C(14)C(13)	108(1)	N(2)C(15)C(16)	132(1)
C(17)C(16)C(15)	114.3(9)	C(21)C(16)C(15)	125.7(9)
N(1)C(21)C(20)	118.1(9)	N(1)C(21)C(16)	121.8(9)
C(23)C(22)S	121(1)	C(27)C(22)S	119(1)
C(24)C(25)C(28)	119(1)	C(26)C(25)C(28)	121(1)

* Symmetry transformation: $^{\#1}$ $-x + 1, y, -z + 3/2$.

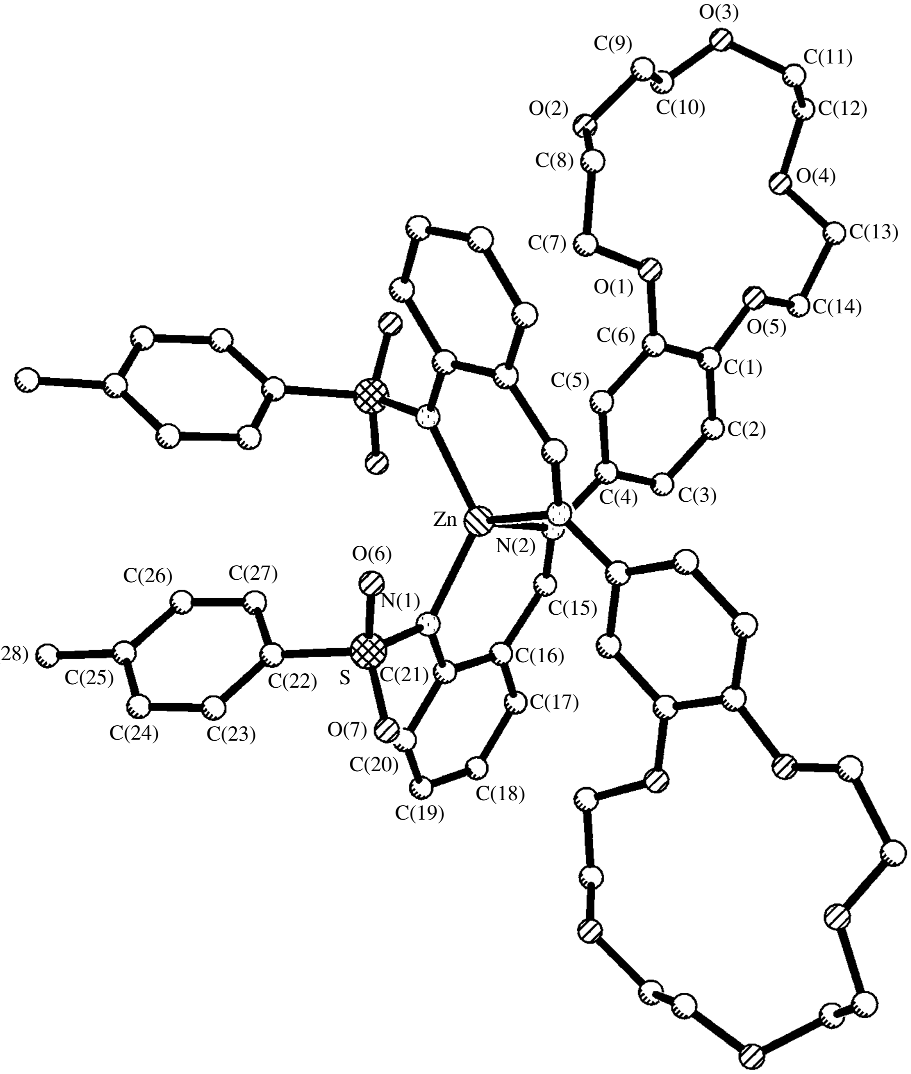

Fig. 1. The structure of molecule **I**.

The values of the torsion angles in a substituted B15C5 in structures **I** and **II** are given in Table 3. As noted in [1, 2], B15C5 exhibits variability of conformations in different compounds. Given below are the conformation formulas of a B15C5 macrocycle in compounds **I–IV**.

I: $T_-CT_-\ T_-G_+S_+\ S_+T_-S_-\ S_-S_-G_+\ T_-G_+T_-$,

II: $G_+CT_-\ T_-G_+T_-\ T_-G_+T_-\ TG_+S_-\ S_+T_-T$,

III: $TCT\ TG_+S\ G_+TT_-\ G_+GG_+\ TG_+T$,

IV: $TCT\ TG_+T\ G_+GT\ TG_+G_+\ TGT$.

The molecule of **I** has standard geometrical parameters. The averaged bond lengths and bond angles in a macrocycle of **I** are as follows (figures in parentheses refer to O–C(sp^3) 1.41(2) (1.42(1)), O–C(sp^2) 1.39(1) (1.376(8)), C(sp^3)–C(sp^3) 1.50(2) (1.49(1)) Å, OC(sp^2)C(sp^2) 116(1)° (117.3(7)°), C(sp^3)C(sp^3)O 106(2)° (109.1(8)°), C(sp^3)OC(sp^3) 116(2)° (112.7(8)°), C(sp^2)OC(sp^3) 119(1)° (114.2(4)°)). The C–C bond length in the phenyl rings is 1.38(1) Å.

The deprotonated L^- ligand in **I** retains benzoid tautomeric form common for solid azomethins. The length of the C=N bond (N(2)–C(15)) 1.29(1) Å (1.279(9) Å in **II**) and the C=N–C angle (C(4)N(2)C(15)) 120(1)° (119.7° in **II**) are close to the values found in **III** (1.278(5) Å and 121.8(4)°) and in 1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazole-3-one-4[(1*E*)-aminomethylidenyl)]-benzo-15-crown-5 (1.270(3) Å and 120.4(2)°) [6]. The value of the torsion angle C(4)N(2)C(15)C(16) 172.4° in **I** (175.9° in **II**) suggests the *trans*-configuration [anti (1*E*)] about the imine bond N(2)–C(15).

The molecules in crystal **I** are packed in chains along the *z* axis in a base-centered motif (Fig. 2). In chains, the complexes are bonded through the inversion centers. The benzene rings C(1)–C(6) of one molecule

Table 3. Torsion angles (τ) B15C5 in structures **I** and **II**

Angle	τ, deg	
	I	**II**
C(14)O(5)C(1)C(6)	–167.9	–83.1
O(5)C(1)C(6)O(1)	1.1	2.5
C(1)C(6)O(1)C(2)	166.3	162.7
C(6)O(1)C(7)C(8)	–175.9	–171.1
O(1)C(7)C(8)O(2)	69.8	67.0
C(7)C(8)O(2)C(9)	–131.9	–165.4
C(8)O(2)C(9)C(10)	140.8	170.5
O(2)C(9)C(10)O(3)	–171.5	–71.9
C(9)C(10)O(3)C(11)	93.6	175.1
C(10)O(3)C(11)C(12)	91.7	178.3
O(3)C(11)C(12)O(4)	–96.6	71.7
C(11)C(12)O(4)C(13)	86.5	–94.7
C(12)O(4)C(13)C(14)	172.5	131.0
O(4)C(13)C(14)O(5)	–69.8	170.9
C(13)C(14)O(5)C(1)	174.1	–177.6

lie above the C(16)–C(21) rings of a neighboring molecule at a distance of 3.5 Å; the angle between the plane of the rings being 11.5° (Fig. 3). The Zn···Zn distance in the chain (7.44 Å) is the shortest between the metal atoms in the structure.

The complexation between the HL molecule and the Zn atoms is confirmed by IR spectrum of compound **I**.

Since complex **I** contains the deprotonated ligand, its IR spectrum shows changes in the regions of vibrations of azomethin fragment.

First, unlike the spectrum of a free HL molecule, the spectrum of **I** does not exhibit the band corresponding to the vibration δ(NH) (1572 cm^{-1} in **II**).

Second, in complex **I**, the band due to the vibration ν(C=N) of coordinated azomethin group is slightly shifted toward low-frequency region (1609 cm^{-1}) as compared to its position in IR spectrum of **II** (1613 cm^{-1}). The coordination of L by a metal brings about a significant lowering in the frequency $\nu_{as}(SO_2)$ in **I** as compared to its value for a free ligand (1300 and 1338 cm^{-1}, respectively). A similar decrease in the value of $\nu_{as}(SO_2)$ was observed in the case of formation of chelates of transition metals with azomethins free of the crown ether fragment [7].

The band $\nu_s(SO_2)$ observed at 1156 cm^{-1} in **II** also undergoes a noticeable low-frequency shift and appears in IR spectrum of **I** at 1136 cm^{-1} as very intense band. The bands $\nu_s(SO_2)$ of the coordinated tosyl group are detected almost in the same region in the spectra of analogous compounds reported above [7].

In the spectrum of complex **I**, the band at 1155 cm^{-1} is retained (but its intensity is very low). This band is likely to be due to the deformation vibrations σ(CH) of benzene rings.

As follows from X-ray diffraction data, the complex formation results in the change in a macrocycle conformation in complex **I** as compared to that in a free ligand. Therefore, IR spectrum of **I** shows some changes in the region containing conformation-sensitive vibrations ν_{as}(COC) of a macrocycle (1140–1080 cm^{-1}). In this region of IR spectrum of complex **II**, five bands were observed, which were assigned to the corresponding conformations of ethylene glycol units of a macrocycle: 1134, 1128 (TG_+S), 1112 (S_+TT), 1110 and 1092 cm^{-1} (*TGT*) [1]. Two intense bands appear in the spectrum of **I** at 1136 (with the inflection point at 1111) and 1083 cm^{-1}.

X-ray diffraction data obtained for complex **I** made it possible to perform some corrections in assignment of IR bands, which was previously suggested for **II** and **III**. IR spectra of compounds **I–III** and of 4'-nitrobenzo-15-crown-5 (**NB15C5, V**) contain in a range of 1140–1130 cm^{-1} an intense band that is absent from the spectrum of a free compound **IV**. The conformation formulas of the B13C5 macrocycles in **I–IV** suggest that all azomethin derivatives of B15C5 contain in macrocycles the ethylene glycol units with the *trans*-conformation about the C–C bond (ST_-S, S_+T_-T, T_-TG, $S_+T_-G_+$), which is not typical of macrocycles in a free compound **IV** and in its complexes.

Therefore, we assumed that the band at 1134 cm^{-1} in the spectra of **I–IV** is due to vibrations of the units having *trans*-conformation about the C–C bond. Since X-ray diffraction analysis revealed that compound **I** contained such a unit ($S_+T_-S_-$), this region of IR spectrum (~1130 cm^{-1}) should exhibit the corresponding band. As mentioned above, IR spectrum of **I** has an intense band at 1136 cm^{-1}, which we assigned to $\nu_s(SO_2)$. This band is likely to overlap less intense band produced by the vibration ν_{as}(COC) of the $S_+T_-S_-$ unit.

According to correlations [8], the intense band at 1083 cm^{-1} in the spectrum of **I** is likely to be due to the *TGT* unit, while the inflection point at 1111 cm^{-1} is due to the *TGS* unit. In the spectrum of **II**, the band at 1112 cm^{-1} corresponds to vibrations of the *TGS* unit.

In addition to ν_{as}(COC), the conformation-sensitive vibrations also include composite stretching-deformation

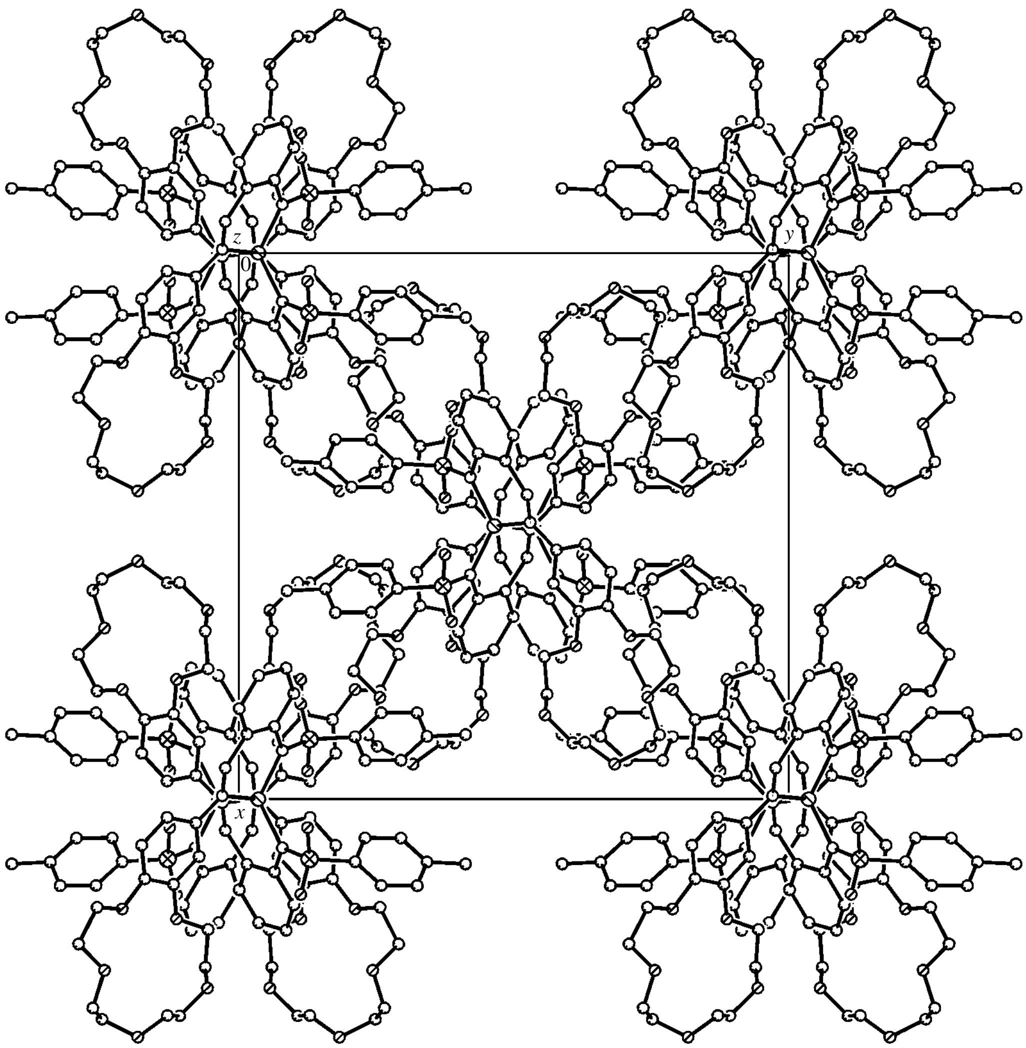

Fig. 2. The molecular packing in crystal **I** (projection along the *z* axis).

vibrations $\rho(CH_2) + \nu(CO) + \nu(CC)$ observed at 1000–800 cm^{-1} [9]. A moderate-intensity band appears in this region of IR spectrum of compound **I** at 947 cm^{-1}. This band is likely to be due to vibrations of the $T_-G_+TS_-G_+G_+$ fragment, since analogous band (at 943 cm^{-1}) appears in the spectrum of HL, whose macrocycle contains the TG_+T TG_+S_- fragment with close conformation [1]. The spectra of **III–V** also contain the moderte-intensity bands at 950–930 cm^{-1}. According to X-ray diffraction data [2, 5, 10], these compounds incorporate fragments with very close conformations.

Obviously, the low-intensity band at 912 cm^{-1} in IR spectrum of **I** corresponds to the *SGG* unit, since the spectrum of compound **V**, whose macrocycle also contains such a unit, exhibits analogous band as well (at 913 cm^{-1}).

The band at 901 cm^{-1} in IR spectrum of compound **I** can be assigned to the vibration of the *TGS* unit, since the spectra of **II, III**, and **V** contain the bands at 896, 891, and 888 cm^{-1}, respectively. X-ray diffraction analysis showed that macrocycles of these compounds contain ethylene glycol units with the *TGS* conformation. The characteristic band at 848 cm^{-1} in IR spectrum of complex **I** corresponds to the rocking vibrations of the *TGT* unit.

The band observed in IR spectrum of **I** at 877 cm^{-1} was assigned to the ST_-S unit vibrations, because the spectra of HL, **III**, and **V** contain the bands with close frequencies (878, 868, and 869 cm^{-1}, respectively), and the macrocycles of these three compounds contain the units with the *trans*-conformations about the C–C bonds (S_+T_-T, T_-TG_+, $S_+T_-G_+$, respectively).

The low-intensity band at 800 or 795 cm^{-1} in IR spectrum of **I** can be assigned to the pulsed vibration ν_{resp} of a macrocycle of alternate type [9].

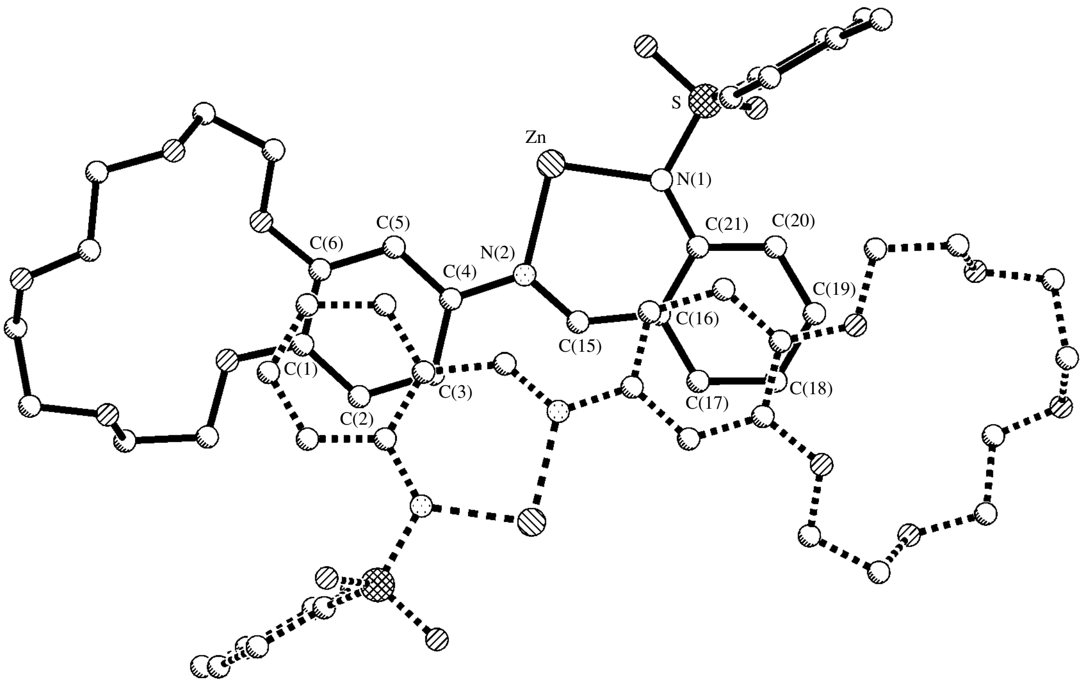

Fig. 3. Superposition of the phenyl rings in two molecules bonded through the inversion center (only an independent part of molecule **I** is shown for clarity).

The study of complexing and ion-selective properties of ZnL_2 will be the subject matter of our further investigations.

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