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Tetrahedral Complexes of Zinc(II) Chloride with N- and O-Containing Organic Ligands: Synthesis and Crystal Structures of $[ZnCl_2(C_{12}H_{12}N_2O)]$ and $[ZnCl_2(H_2O)_2](Me_4Pyz)_2$

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Abstract—New complex chlorides $[ZnCl_2(ODA)]$ (**I**) (ODA=oxydianiline, $C_{12}H_{12}N_2O$) and $[ZnCl_2(H_2O)_2](Me_4Pyz)_2$ (**II**) (Me_4Pyz = 2,3,5,6-tetramethylpyrazine) were synthesized and crystallographically characterized. Crystals of **I** are monoclinic, space group $C2/c$, $a = 22.682(2)$ Å, $b = 12.646(1)$ Å, $c = 9.951(1)$ Å, $\beta = 93.23(2)°$, $V = 2849.7(5)$ Å^3, $\rho_{calc} = 1.569$ g/cm^3, $Z = 8$. Structure **I** contains cyclic fragments consisting of two tetrahedral complexes ($ZnCl_2N_2$) and two coordinated bridging oxydianiline ligands. Crystals of **II** are monoclinic, space group $P2(1)/c$, $a = 8.972(2)$ Å, $b = 13.862(3)$ Å, $c = 17.528(4)$ Å, $\beta = 101.72(3)°$, $V = 2134.5(7)$ Å^3, $\rho_{calc} = 1.384$ g/cm^3, $Z = 4$. In structure **II**, supramolecular pseudo-metallocycles are formed due to formation of hydrogen bonds O(w)–H···N between coordinated water molecules and noncoordinated nitrogen atoms of tetramethylpyrazine molecules.

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The chemistry of coordination polymers and supramolecular compounds attract permanently increasing interest. Analysis and understanding of intra- and intermolecular interactions in crystals are important when for their use in synthesis of new compounds with potentially useful properties. Synthesis of such samples is often based on self-assembly of corresponding building units, resulting in coordination polymers and supramolecular assemblies due to covalent and hydrogen bonds other weak interactions. In coordination polymers, organic ligands are usually bound to metal ions by donor–acceptor bonds to form rigid frameworks, while weak intermolecular interactions give a possibility to obtain nonrigid assemblies.

To synthesize coordination and supramolecular compounds, salts of divalent cations (Zn, Cd, Ni, Co) and N- and O-containing organic ligands of various lengths, functionalities, and electronic structures are often used. In that way, different structural compositions in the form of chains, bands, rings, and 2D and 3D polymers were obtained. Formation of supramolecular pseudo-metallocycles is characteristic of compounds of divalent cations when water molecules enter the coordination sphere together with ditope nitrogen-containing ligands. When weak hydrogen bonds are involved in bonding, nonrigid supramolecular structures are formed, for example, in the compound $[Co(2\text{-}MePyz)_2(H_2O)_4](NO_3)_2$ [1].

Compounds with Zn contain predominantly tetrahedral complexes, whereas Cd compounds are characterized by higher coordination numbers [2, 3]. Synthesis and structure of tetrahedral and octahedral discrete coordination polymeric and supramolecular compounds of Zn(II) were considered, for example, for $ZnCl_2(4\text{-R-Py})_2$ (R = vinyl, acetyl, or cyano substitute), $ZnBr_2$(pyrimidine), and others [4–7].

The aim of this work is to obtain two complex compounds of zinc chloride with nitrogen-containing ligands, namely, $[ZnCl_2(ODA)]$ (**I**) (ODA = oxydianiline, $C_{12}H_{12}N_2O$) and $[ZnCl_2(H_2O)_2](Me_4Pyz)_2$ (**II**) (Me_4Pyz = 2,3,5,6-tetramethylpyrazine) and determine their structures.

EXPERIMENTAL

Syntheses of compounds I and II were performed by similar procedures with the use of Aldrich and Fluka reagents. To obtain **I,** zinc chloride of analytical grade and 2,2'-oxydianiline taken in a 1 : 2 ratio were separately dissolved in isopropanol; then, the solutions were mixed and allowed to evaporate at room temperature for several days. The resulting crystals were filtered off, rapidly washed with a small amount of isopropanol, and dried in air.

According to the chemical analysis data, the majority of crystals had the chemical composition

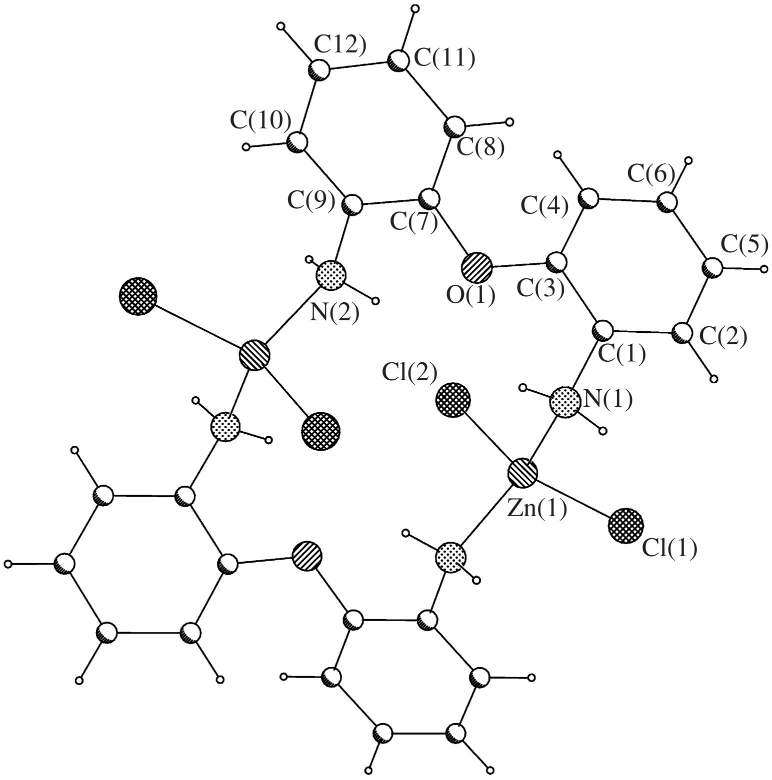

Fig. 1. Cyclic fragment in structure **I**.

$[ZnCl_2(ODA)_2]$, whereas single crystals suitable for X-ray crystallography formed in a small amount had the 1 : 1 composition.

For $C_{12}H_{12}Cl_2N_2OZn$ (**I**) anal. calc. (wt %): C, 42.79; N, 8.32; H, 3.56.

Found (wt %): C, 43.12; N, 8.08; H, 3.92.

Note that the synthesis in ethyl alcohol gave analogous results.

For the synthesis of crystals **II**, $ZnCl_2$ and Me_4Pyz taken in a 1 : 2 ratio were separately dissolved in ethanol, and then the solutions were mixed and allowed to at room temperature to slowly evaporate. The resulting crystals were filtered off, washed, and dried in air.

For $C_{16}H_{28}Cl_2N_4O_2Zn$ (**II**) anal. calc. (wt %): C, 43.17; N, 12.59; H, 6.29.

Found (wt %): C, 43.49; N, 12.36; H, 6.16.

IR spectra of the ligands and complexes were recorded as mineral oil mulls on KBr plates on a Specord 75 IR spectrophotometer in a range of 4000–400 cm^{-1}. The IR spectra of **I** and **II** show the bands of the corresponding coordinated ligands. The spectrum of **II** contains also a broad strong band due to the ν(O–H) vibrations of water shifted to 2900 cm^{-1}, which is indicative of the presence of hydrogen bonds.

X-ray crystallography. Experimental data for crystals **I** an **II** were obtained on an Enraf-Nonius automated CAD4 diffractometer. The structures were solved by the heavy-atom method and refined by full-matrix least-squares calculation in anisotropic approximation for all non-hydrogen atoms. Positions of hydrogen atoms were calculated from geometric considerations and refined as riding on their bonded atoms with fixed isotropic thermal parameters. The hydrogen atoms of water molecules were located from a difference Fourier synthesis.

Crystallographic data and basic experimental details for structures **I** and **II** are listed in Table 1; atomic coordinates and thermal parameters are presented in Table 2; selected bond lengths and bond angles are presented in Table 3; and geometric parameters of hydrogen bonds are shown in Table 4. All calculations were performed with the SHELXL-97 program package.

RESULTS AND DISCUSSION

The structure of **I** contains centrosymmetric cyclic fragments consisting of two tetrahedral complexes of zinc chloride with nitrogen atoms of two bridging oxydianiline ligands (Fig. 1). The Zn–Cl and Zn–N dis-

Table 1. Crystallographic data and experimental details for structures **I** and **II**

	I	**II**
FW	336.51	444.69
Crystal dimensions, mm	$0.10 \times 0.15 \times 0.23$	$0.15 \times 0.20 \times 0.24$
Crystal system	Monoclinic	
Space group	$C2/c$	$P2(1)/c$
Unit cell parameters:		
a, Å	22.682(2)	8.972(2)
b, Å	12.646(1)	13.862(3)
c, Å	9.951(1)	17.528(4)
β, deg	93.23(2)	101.72(3)
V, Å^3	2849.7(5)	2134.5(7)
Z	8	4
ρ_{calc}, g/cm^3	1.569	1.384
μ_{Cu}, mm^{-1}	5.757	4.034
$F(000)$	1360	928
Temperature, K	293	
Radiation (λ, Å)	CuK_α (1.54178), graphite monochromator	
Scan mode	ω	
θ range, deg	3.90–70.84	4.10–59.95
Index ranges	$-27 \le h \le 27$, $-15 \le k \le 15$, $-12 \le l \le 0$	$-10 \le h \le 9$, $0 \le k \le 15$, $0 \le l \le 19$
Number of measured reflections	5687	3068
Number of unique reflections	2755 [R(int) = 0.0171]	2959 [R(int) = 0.0242]
Number of observed reflections with $I \ge 2\sigma(I)$	2335	2324
Number of refined parameters	164	227
GOOF on F^2	0.816	0.985
R ($I \ge 2\sigma(I)$)	$R_1 = 0.0248$, $wR_2 = 0.0872$	$R_1 = 0.0329$, $wR_2 = 0.1056$
R for all reflections	$R_1 = 0.0333$, $wR_2 = 0.0949$	$R_1 = 0.0501$, $wR_2 = 0.1179$
Extinction coefficient	0.00075(8)	0.0061(5)
Residual electron density (max/min), e/Å^3	0.350/–0.250	0.368/–0.367

Table 2. Atomic coordinates and thermal parameters ($U_{eq} = 1/3\Sigma U_{ij}$) for structures **I** and **II**

Atom	*x*	*y*	*z*	U_{eq}, Å^2
		I		
Zn(1)	0.2902(1)	0.9335(1)	–0.0113(1)	0.042(1)
Cl(1)	0.3352(1)	1.0840(1)	–0.0567(1)	0.060(1)
Cl(2)	0.2649(1)	0.8344(1)	–0.1913(1)	0.052(1)
N(1)	0.2141(1)	0.9736(2)	–0.0859(2)	0.046(1)
N(2)	0.1619(1)	0.6549(1)	–0.1281(2)	0.043(1)
O(1)	0.1334(1)	0.8466(1)	–0.0314(2)	0.059(1)
C(1)	0.1706(1)	1.0172(2)	–0.0090(2)	0.047(1)
C(2)	0.1722(1)	1.1236(2)	–0.0438(3)	0.063(1)
C(3)	0.1295(1)	0.9506(2)	–0.0724(2)	0.050(1)
C(4)	0.0904(1)	0.9885(3)	–0.1725(3)	0.066(1)
C(5)	0.1324(1)	1.1615(2)	–0.1410(4)	0.078(1)
C(6)	0.0919(1)	1.0959(3)	–0.2050(3)	0.081(1)
C(7)	0.0869(1)	0.7771(2)	–0.0532(2)	0.050(1)
C(8)	0.0291(1)	0.8017(3)	–0.0258(3)	0.071(1)
C(9)	0.1017(1)	0.6774(2)	–0.0972(2)	0.045(1)
C(10)	0.0589(1)	0.5998(2)	–0.1084(3)	0.062(1)
C(11)	–0.0137(1)	0.7240(3)	–0.0415(3)	0.076(1)
C(12)	0.0014(1)	0.6237(3)	–0.0802(3)	0.073(1)
H(1A)	0.199	0.916	0.125	
H(1B)	0.223	1.021	0.151	
H(2A)	0.181	0.717	–0.138	
H(2B)	0.161	0.621	–0.208	
H(3A)	0.200	1.169	–0.002	
H(4A)	0.064	0.943	–0.217	
H(5A)	0.133	1.233	–0.164	
H(6A)	0.065	1.123	–0.271	
H(8A)	0.019	0.869	0.003	
H(10A)	0.069	0.532	–0.135	
H(11A)	–0.053	0.740	–0.026	
H(12A)	–0.027	0.571	–0.087	
		II		
Zn(1)	0.2323(1)	0.2657(1)	0.0913(1)	0.048(1)
Cl(2)	0.2920(1)	0.4187(1)	0.1108(1)	0.079(1)
Cl(3)	0.2759(1)	0.1787(1)	–0.0065(1)	0.081(1)
O(1w)	0.0083(2)	0.2520(2)	0.0882(1)	0.053(1)
O(2w)	0.3297(2)	0.2040(2)	0.1917(1)	0.067(1)
N(1)	0.6192(3)	0.2675(2)	0.2961(2)	0.053(1)
N(2)	–0.1293(2)	0.3836(2)	0.1726(1)	0.048(1)
N(3)	0.8178(3)	0.2593(2)	0.4375(2)	0.047(1)
N(4)	–0.2196(2)	0.5418(2)	0.2451(1)	0.049(1)
C(1)	–0.2409(3)	0.4397(2)	0.1353(2)	0.048(1)
C(2)	–0.0626(3)	0.4046(2)	0.2463(2)	0.044(1)
C(3)	0.7311(3)	0.2017(2)	0.3097(2)	0.054(1)
C(4)	0.6084(3)	0.3309(2)	0.3522(2)	0.050(1)
C(5)	0.7071(3)	0.3254(2)	0.4247(2)	0.049(1)
C(6)	0.6905(4)	0.3908(3)	0.4908(2)	0.068(1)
C(7)	0.8329(3)	0.1988(2)	0.3806(2)	0.050(1)
C(8)	0.9617(4)	0.1278(3)	0.3979(2)	0.070(1)
C(9)	–0.2830(3)	0.5230(2)	0.1708(2)	0.048(1)
C(10)	–0.1118(3)	0.4835(2)	0.2840(2)	0.047(1)
C(11)	0.0677(3)	0.3415(3)	0.2832(2)	0.061(1)
C(12)	–0.0462(4)	0.5073(3)	0.3674(2)	0.067(1)
C(13)	–0.3945(4)	0.5931(3)	0.1276(2)	0.066(1)
C(14)	0.4876(4)	0.4066(3)	0.3345(2)	0.071(1)
C(15)	–0.3181(4)	0.4097(3)	0.0550(2)	0.072(1)
C(16)	0.7407(4)	0.1336(3)	0.2441(2)	0.077(1)
H(6A)	0.769	0.376	0.535	
H(6B)	0.593	0.382	0.503	
H(6C)	0.701	0.457	0.476	
H(8A)	1.018	0.138	0.450	
H(8B)	1.028	0.136	0.362	
H(8C)	0.921	0.063	0.394	
H(11A)	0.083	0.292	0.247	
H(11B)	0.046	0.313	0.330	
H(11C)	0.159	0.380	0.296	
H(12A)	–0.096	0.563	0.382	
H(12B)	0.061	0.518	0.374	
H(12C)	–0.064	0.453	0.400	
H(13A)	–0.409	0.645	0.162	
H(13B)	–0.491	0.561	0.109	
H(13C)	–0.358	0.618	0.084	
H(14A)	0.432	0.398	0.282	
H(14B)	0.533	0.469	0.340	
H(14C)	0.419	0.400	0.370	
H(15A)	–0.273	0.351	0.042	
H(15B)	–0.305	0.459	0.019	
H(15C)	–0.424	0.400	0.053	
H(16A)	0.660	0.147	0.200	
H(16B)	0.730	0.068	0.261	
H(16C)	0.837	0.140	0.229	
H(1w)	–0.045	0.248	0.047	
H(2w)	–0.046	0.030	0.106	
H(3w)	0.300	0.155	0.215	
H(4w)	0.417	0.222	0.220	

Table 3. Bond lengths (*d*) and bond angles (ω) in structures **I** and **II**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
I				**II**			
Zn(1)–N(2)$^{\#1}$	2.045(2)	C(4)–C(6)	1.396(5)	Zn(1)–O(2)	1.991(2)	C(1)–C(9)	1.400(4)
Zn(1)–N(1)	2.088(2)	C(5)–C(6)	1.368(5)	Zn(1)–O(1)	2.008(2)	C(1)–C(15)	1.495(4)
Zn(1)–Cl(1)	2.2189(6)	C(7)–C(9)	1.383(3)	Zn(1)–Cl(3)	2.196(1)	C(2)–C(10)	1.395(4)
Zn(1)–Cl(2)	2.2341(6)	C(7)–C(8)	1.390(3)	Zn(1)–Cl(2)	2.197(1)	C(2)–C(11)	1.498(4)
N(1)–C(1)	1.436(3)	C(8)–C(11)	1.383(4)	N(1)–C(3)	1.342(4)	C(3)–C(7)	1.385(4)
O(1)–C(3)	1.379(3)	C(9)–C(10)	1.380(3)	N(1)–C(4)	1.338(4)	C(3)–C(16)	1.504(5)
O(1)–C(7)	1.381(2)	C(9)–N(2)	1.446(2)	N(2)–C(1)	1.330(4)	C(4)–C(5)	1.395(4)
C(1)–C(3)	1.383(3)	C(10)–C(12)	1.382(4)	N(2)–C(2)	1.340(4)	C(4)–C(14)	1.496(4)
C(1)–C(2)	1.391(3)	C(11)–C(12)	1.374(4)	N(3)–C(7)	1.331(4)	C(5)–C(6)	1.502(5)
C(2)–C(5)	1.371(4)			N(3)–C(5)	1.337(4)	C(7)–C(8)	1.500(4)
C(3)–C(4)	1.382(3)			N(4)–C(10)	1.336(4)	C(9)–C(13)	1.487(4)
Angle	ω, deg	Angle	ω, deg	N(4)–C(9)	1.337(4)	C(10)–C(12)	1.497(4)
N(2)$^{\#1}$Zn(1)N(1)	103.87(7)	C(3)C(4)C(6)	118.7(3)	Angle	ω, deg	Angle	ω, deg
N(2)$^{\#1}$Zn(1)Cl(1)	111.97(5)	C(6)C(5)C(2)	120.9(3)	O(2)Zn(1)O(1)	103.93(10)	N(1)C(3)C(16)	116.6(3)
N(1)Zn(1)Cl(1)	106.72(5)	C(5)C(6)C(4)	120.5(3)	O(2)Zn(1)Cl(3)	109.76(9)	C(7)C(3)C(16)	122.6(3)
N(2)$^{\#1}$Zn(1)Cl(2)	109.84(5)	O(1)C(7)C(9)	115.6(2)	O(1)Zn(1)Cl(3)	105.24(7)	N(1)C(4)C(5)	120.5(3)
N(1)Zn(1)Cl(2)	109.05(5)	O(1)C(7)C(8)	123.2(2)	O(2)Zn(1)Cl(2)	103.16(8)	N(1)C(4)C(14)	117.7(3)
Cl(1)Zn(1)Cl(2)	114.74(3)	C(9)C(7)C(8)	121.0(2)	O(1)Zn(1)Cl(2)	107.87(6)	C(5)C(4)C(14)	121.8(3)
C(1)N(1)Zn(1)	110.1(1)	C(11)C(8)C(7)	118.8(3)	Cl(3)Zn(1)Cl(2)	125.11(5)	N(3)C(5)C(4)	120.2(3)
C(3)O(1)C(7)	121.6(2)	C(10)C(9)C(7)	119.4(2)	C(3)N(1)C(4)	118.6(3)	N(3)C(5)C(6)	118.1(3)
C(3)C(1)C(2)	120.1(2)	C(10)C(9)N(2)	120.8(2)	C(1)N(2)C(2)	119.5(3)	C(4)C(5)C(6)	121.7(3)
C(3)C(1)N(1)	119.2(2)	C(7)C(9)N(2)	119.8(2)	C(7)N(3)C(5)	119.4(2)	N(3)C(7)C(3)	120.3(3)
C(2)C(1)N(1)	120.6(2)	C(9)N(2)Zn(1)$^{\#1}$	115.8(1)	C(10)N(4)C(9)	120.0(3)	N(3)C(7)C(8)	117.0(3)
C(5)C(2)C(1)	119.3(3)	C(9)C(10)C(12)	119.7(3)	N(2)C(1)C(9)	120.5(3)	C(3)C(7)C(8)	122.7(3)
O(1)C(3)C(4)	124.9(2)	C(12)C(11)C(8)	120.3(2)	N(2)C(1)C(15)	117.4(3)	N(4)C(9)C(1)	119.6(3)
O(1)C(3)C(1)	114.5(2)	C(11)C(12)C(10)	120.7(2)	C(9)C(1)C(15)	122.2(3)	N(4)C(9)C(13)	118.5(3)
C(4)C(3)C(1)	120.5(2)			N(2)C(2)C(10)	120.3(2)	C(1)C(9)C(13)	121.8(3)
				N(2)C(2)C(11)	116.5(3)	N(4)C(10)C(2)	119.9(3)
				C(10)C(2)C(11)	123.2(3)	N(4)C(10)C(12)	117.7(3)
				N(1)C(3)C(7)	120.8(3)	C(2)C(10)C(12)	122.4(3)

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

Table 4. Geometric parameters of hydrogen bonds in structures **I** and **II**

Bond A–H···B	Position of B atom	A···B	A–H	H···B	Angle AHB, deg
		Å			
		I			
N(1)–H(1A)···O(1)	*x*, *y*, *z*	2.656(2)	0.90	2.27	106
N(1)–H(1B)···Cl(2)	*x*, 2 – *y*, 1/2 + *z*	3.443(2)	0.90	2.56	169
N(2)–H(2A)···O(1)	*x*, *y*, *z*	2.700(2)	0.90	2.26	110
N(2)–H(2B)···Cl(1)	1/2 – *x*, –1/2 + *y*, –1/2 – *z*	3.266(2)	0.90	2.40	162
		II			
O(1)–H(1)···N(3)	–1 – *x*, 1/2 – *y*, –1/2 – *z*	2.844(3)	0.79	2.06	179
O(1)–H(2)···N(3)	*x*, *y*, *z*	2.791(3)	0.87	1.94	165
O(2)–H(3)···N(4)	–*x*, 1/2 + *y*, 1/2 – *z*	2.775(3)	0.86	1.92	173
O(2)–H(4)···N(1)	*x*, *y*, *z*	2.990(3)	0.87	2.12	174

tances in the distorted $ZnCl_2N_2$ tetrahedron have average values 2.227 Å and 2.067 Å, respectively. The angles at zinc atoms vary in a range of 103.87(7)°–114.74(3)° (Table 3). The ring containing two zinc atoms and two oxydianiline molecules has dimensions 4.995(1) Å (the Zn(1)···Zn(1A) distance) × 5.827(2) Å (the O(1)···O(1A) distance between oxygen atoms of two ligands). The rings are linked by hydrogen bonds N–H···Cl to form a supramolecular framework. The NH_2 groups of oxydianiline also form hydrogen bonds N–H···O within the rings (Table 4).

In structure **II**, two Cl atoms ($Zn–Cl_{av}$, 2.197 Å) and two oxygen atoms of the water molecules ($Zn–O_{av}$, 2.00 Å) form the tetrahedral coordination sphere of the zinc atom. The angles at the Zn atom deviate considerably from the ideal tetrahedral angle (109.5°) and lie within the range of 103.9(1)°–125.11(5)° (Table 3). Noncoordinated tetramethylpyrazine molecules act as bridges between adjacent complexes of Zn^{2+} and produce centrosymmetric pseudo-metallocycles due to formation of hydrogen bonds O(w)–H···N between the water molecules and the nitrogen atoms of the Me_4Pyz molecules (Fig. 2). The conjugated rings form the supramolecular framework containing channels with dimensions 14.83 × 15.57 Å (distances between the Zn atoms) with methyl groups directed inward the channels (Fig. 3).

In compound **I**, the ring is formed by donor–acceptor bonds. The long enough distance between the nitrogen atoms in oxydianiline (N(1)···N(2) 4.680(3) Å) does not allow the ligand to act as a chelate; therefore, this ligand forms the bridge between the symmetrically bound zinc atoms.

In compound **II**, the supramolecular pseudo-metallocycles are formed due to the hydrogen bonds between the O atoms of the coordinated water molecules of the complex $[ZnCl_2(H_2O)_2]$ and the N atoms of tetramethylpyrazine. These rings, in contrast to the rings in **I**, are nonrigid.

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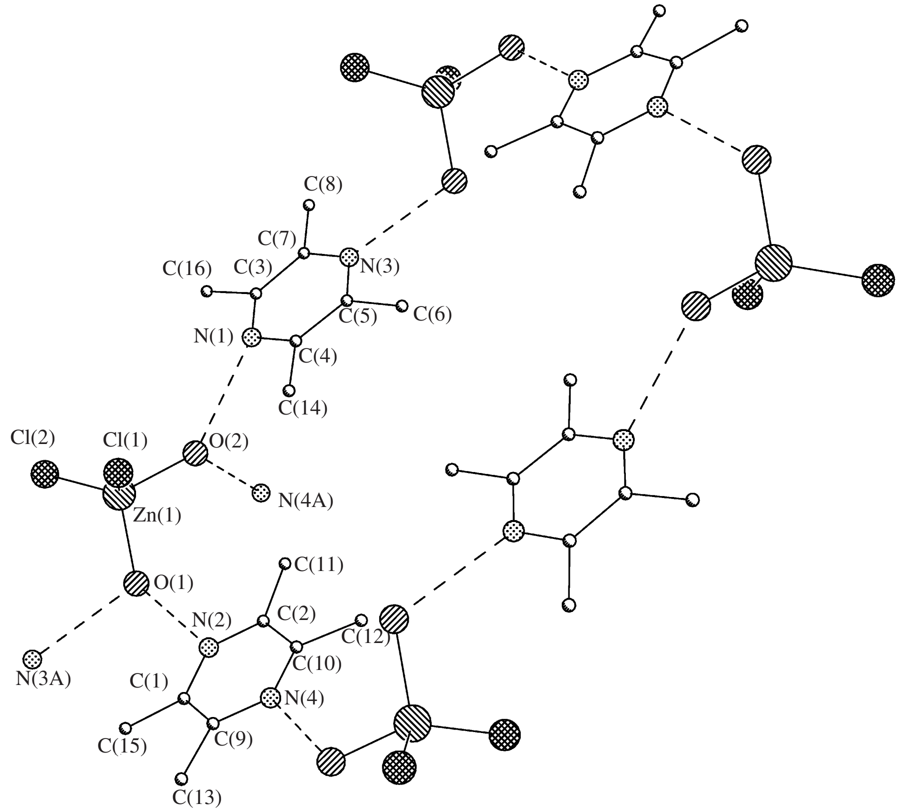

Fig. 2. Supramolecular pseudo-metallocycle in structure **II** (symmetry codes: –1 + *x*, 1/2 – *y*, –1/2 + *z* for N(3A) and –*x*, –1/2 + *y*, 1/2 – *z* for N(4A)).

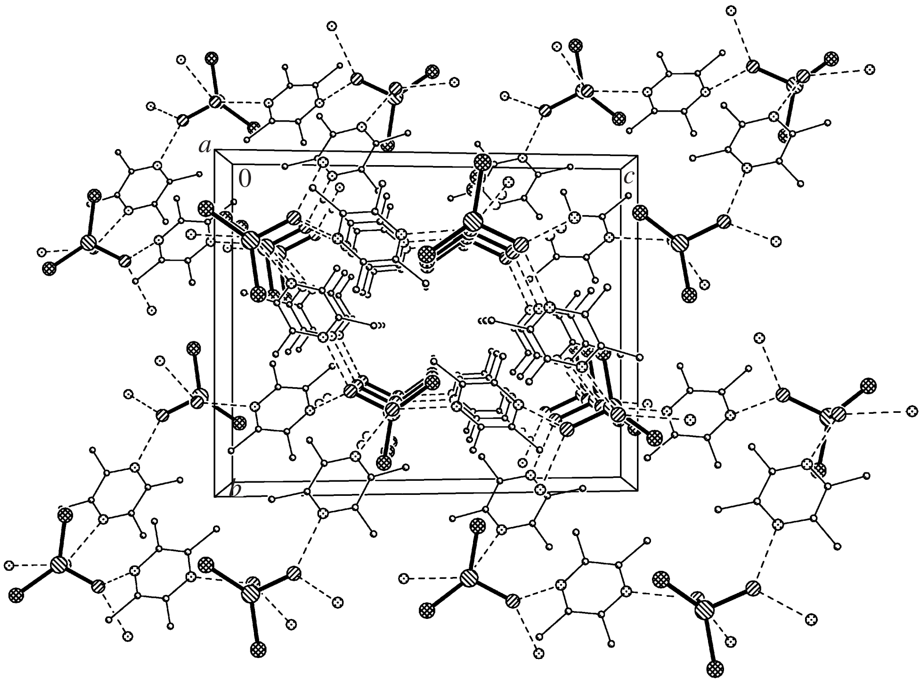

Fig. 3. General view of structure **II** along the [100] direction.

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