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Dianilineglyoxime Salt and Its Binuclear Zn(II) and Mn(II) Complexes with 1,3-Benzenedicarboxylic Acid: Synthesis and Structures

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Abstract—Single crystals of dianilineglyoxime ($DAnH_2$) in the form of hydrate salts $[DAnH_3](ClO_4) \cdot H_2O$ (**I**) and $[DAnH_3](ClO_4) \cdot 1.25H_2O$ (**II**) are obtained. Three binuclear zinc(II) and manganese(II) compounds with dianilineglyoxime and 1,3-benzenedicarboxylic acid (1,3-H_2Bdc) are synthesized: $[Zn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF \cdot MeOH \cdot 1.5H_2O$ (**III**), $[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF$ (**IV**), and $\{[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_2(CH_3OH)_2][Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2CH_3OH\}$ (**V**). The compositions and structures of compounds **I**–**V** are confirmed by IR spectroscopy and X-ray diffraction analysis (CIF files CCDC nos. 1906360 (**I**), 1906362 (**II**), 1906361 (**III**), 1906363 (**IV**), and 1906364 (**V**)). The X-ray diffraction data show that $DAnH_2$ coordinates to the metal ions via the bidentate chelate mode, whereas $(1,3\text{-}Bdc)^{2-}$ coordinates to the metal ions via the bidentate bridging mode to form binuclear structures. As the most part of α-dioximes, one neutral $DAnH_2$ coordinates to each metal atom by two nitrogen atoms, whereas two $(1,3\text{-}Bdc)^{2-}$ anions coordinate to the metal atom by one oxygen atom each. The coordination polyhedra of the metals in compounds **III**–**V** are built up by the oxygen atoms of DMF and CH_3OH.

Keywords: coordination compounds of manganese and zinc, dianilineglyoxime, 1,3-benzenedicarboxylic acid, IR spectra, X-ray diffraction analysis

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INTRODUCTION

α-Dioximes have been widely used as chelating agents in coordination chemistry since the early 1900s [1–3]. When synthesizing the coordination compounds, three geometric isomers should be taken into account due to two oxime groups present in these ligands: *anti*/*cis* (*E,E*), *amphi* (*E,Z*), and *syn*/*trans* (*Z,Z*) [4, 5]. The structural results available at the Cambridge Crystallographic Data Centre (CCDC) [6] confirm that the *N,N*-chelating mode that stabilizes the (*E,E*) *anti*-form is used most frequently in the dioximate compounds for the coordination of these ligands.

The structures of transition metal dioximates are determined by many factors: the nature and oxidation state of the complexing agent, the properties of the dioxime itself, the pH of the medium, etc. The nickel(II), palladium(II), and platinum(II) dioximates with the square coordination mode [7], binuclear copper(II) dioximates with the square pyramidal structure [8], and those of iron(II) [9], iron(III) [10], cobalt(II) [5], cobalt(III) [11, 12], and nickel(III) [13] with the octahedral structures are known. In all mentioned complexes, two oxime ligands are coordinated as monoanions localized in the approximately one plane to form fairly strong intramolecular hydrogen bonds O–H···O. The iron(II) [14], nickel(II), cobalt(II), copper(II) [15–17], and copper(I) complexes [18] synthesized in a highly acidic medium, where dioxime molecules are not deprotonated and are coordinated without formation of similar hydrogen bonds with different orientations of the OH groups, are also described. The mononuclear Zn(II) and Cd(II) dioximates [19], binuclear Co(III), Cu(II), Zn(II), and Cd(II) complexes using additional bidentate pyridine ligands [20–22], and coordination polymers [23] are described. Transition metal complexes of the bi- and polynuclear type of Zn(II), Cd(II), Mn(II), Co(III), and Cu(II) with mixed ligands including dioximes and di- or tricarboxylic acids [24, 25] are known. However, coordination compounds of manganese(II) and zinc(II) of the binuclear type with dioximes, including dianilineglyoxime, are poorly studied. These complexes are of interest from the viewpoint of their biological activity, for example, antibacterial and fungicidal properties [26–28].

This work is devoted to the synthesis and studies of the crystal structures of the binuclear coordination compounds of manganese(II) and zinc(II) based on dianilineglyoxime ($DAnH_2$) and 1,3-benzenedicarboxylic acid (1,3-H_2Bdc).

EXPERIMENTAL

Commercial reagents and solvents (reagent grade) were used as received.

Dianilineglyoxime was synthesized by the condensation of dichloroglyoxime with aniline using a known procedure [26].

IR (ν, cm^{-1}): 3370 m, 3057 s, 3039 sh, 2977 vw, 2903 m, 2833 sh, 2780 s, 2087 w.br, 1939 w, 1871 w.br, 1794 w, 1744 w.br, 1637 vs, 1596 vs, 1494 vs, 1475 sh, 1432 vs, 1354 s, 1292 m, 1272 s, 1218 m, 1183 w, 1110 vw, 1074 m, 1026 w, 1004 w, 971 vs, 909 m, 884 m, 804 w.br, 752 s, 733 m, 711 w, 689 vs, 619 vw, 605 m, 588 m, 508 sh, 503 m, 487 m, 441 m, 425 sh, 407 m.

We failed to obtain single crystals of dianilineglyoxime with the parameters suitable for X-ray diffraction analysis (XRD). However, an attempt to synthesize the cadmium complexes with dianilineglyoxime gave colorless crystals of two types: $[DAnH_3]ClO_4 \cdot H_2O$ (**I**) and $[DAnH_3]ClO_4 \cdot 1.25H_2O$ (**II**), which differed only by the ratio cation : anion : number of water molecules.

Synthesis of $[Zn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF \cdot MeOH \cdot 1.5H_2O$ (III). A mixture of $DAnH_2$ (0.054 g, 0.2 mmol) and $Zn(CH_3COO)_2 \cdot 2H_2O$ (0.022 g, 0.1 mmol) was dissolved in a dimethylformamide (DMF)–methanol mixture (12 mL) in a ratio of 1 : 1 (vol/vol). A solution of 1,3-H_2Bdc (0.034 g, 0.2 mmol) in methanol (3 mL) was added to the obtained solution with permanent stirring. A light yellow reaction mixture was additionally stirred for 10–15 min, after which the solution was filtered and the filtrate was left to stay at room temperature for crystallization. Block colorless crystals were formed in 30 days. The product was soluble in DMSO and insoluble in DMF, methanol, water, and diethyl ether.

For $C_{63}H_{85}N_{14}O_{20.5}Zn_2$

Anal. calcd., %	C, 50.53	H, 5.72	N, 13.11	Zn, 8.74
Found, %	C, 51.04	H, 5.77	N, 13.34	Zn, 8.89

IR (ν, cm^{-1}): 3317 m, 3068 w, 3045 vw, 2930 m, 2807 vw, 2310 m.sh, 1869 m.sh, 1672 sh, 1649 vs, 1599 s, 1540 m, 1497 s, 1470 m, 1458 m, 1435 m, 1374 vs, 1315 w, 1297 m, 1253 m, 1179 w, 1155 w, 1104 m, 1087 m, 1080 sh, 1062 w, 994 s, 949 m, 911 m, 892 m, 868 w, 837 w, 805 m, 749 s, 707 s, 699 s, 677 m, 658 m, 633 m, 602 m, 553 vw, 518 m, 493 m, 451 m, 413 w.

Synthesis of $[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF$ (IV). Compounds $DAnH_2$ (0.054 g, 0.2 mmol) and $Mn(CH_3COO)_2 \cdot 4H_2O$ (0.024 g, 0.1 mmol) were dissolved in a mixture of solvents DMF—ethanol (12 mL) in a ratio of 1 : 1 (vol/vol). The formed weakly yellow solution was added at room temperature by 1,3-H_2Bdc (0.034 g, 0.2 mmol) dissolved in ethanol (3 mL), after which the solution turned light brown. The solution was stirred at room temperature for 20 min. The formed yellow precipitate was filtered off, and the filtrate was left to stay for crystallization. Prismatic yellow crystals suitable for XRD were formed in the filtrate in three weeks. The product was soluble in DMSO and insoluble in DMF, methanol, water, and diethyl ether.

For $C_{62}H_{78}N_{14}O_{18}Mn_2$

Anal. calcd., %	C, 52.54	H, 5.55	N, 13.84	Mn, 7.75
Found, %	C, 52.77	H, 5.38	N, 13.88	Mn, 7.81

IR (ν, cm^{-1}): 3622 w, 3296 m, 3078 vw, 2932 w, 2540 m.sh, 2331 m.sh, 1877 w.br, 1649 s, 1628 sh, 1599 s, 1539 m, 1497 m, 1472 m, 1453 w, 1436 m, 1419 w, 1376 vs, 1309 w, 1295 w, 1178 w, 1157 w, 1112 m, 1096 w, 1078 m, 994 s, 1112 sh, 949 m, 909 m, 893 w, 835 w, 807 w, 802 w, 752 s, 708 s, 696 s, 679 m, 633 w, 603 w, 546 w, 513 m, 490 m, 443 m.

Synthesis of $\{[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_2(CH_3OH)_2][Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2CH_3OH\}$ (V). Compounds $DAnH_2$ (0.054 g, 0.2 mmol) and $Mn(CH_3COO)_2 \cdot 4H_2O$ (0.024 g, 0.1 mmol) were dissolved in a DMF—methanol mixture (12 mL) in a ratio of 1 : 1 (vol/vol). A solution of 1,3-H_2Bdc (0.034 g, 0.2 mmol) in methanol (3 mL) was added to the obtained yellow solution at room temperature with permanent stirring. A weakly yellow precipitate was formed on stirring of the reaction mixture for 10–15 min. The precipitate was filtered off, and the filtrate was left to stay at room temperature for crystallization. Prismatic crystals of the same color suitable for XRD were formed in the filtrate in 26 days. The product was soluble in DMSO and insoluble in DMF, methanol, water, and diethyl ether.

For $C_{110}H_{130}N_{22}O_{34}Mn_4$

Anal. calcd., %	C, 52.34	H, 5.19	N, 12.21	Mn, 8.71
Found, %	C, 52.63	H, 5.17	N, 12.54	Mn, 8.73

IR (ν, cm^{-1}): 3617 w, 3400 w.br, 3302 m, 3063 vw, 2937 w.br, 2479 w.br, 1840 m.sh, 1649 vs, 1628 sh, 1598 m, 1539 m, 1497 m, 1472 m, 1436 m, 1419 w, 1376 vs, 1295 m, 1281 w, 1254 w, 1177 w, 1156 w, 1114 m, 1095 w, 1078 m, 1027 sh, 1007 sh, 999 s, 949 m, 920 w, 906 m, 892 m, 832 w.br, 812 w, 805 w, 751 vs, 735 sh, 708 vs, 698 s, 680 m, 666 m, 659 sh, 634 m, 603 m, 552 w, 514 m, 490 m, 477 m, 442 m, 432 sh.

IR spectra were recorded on a Perkin-Elmer 100 FT-IR spectrometer in a range of 4000–400 cm^{-1} (suspensions in Nujol) and in a range of 4000–650 cm^{-1} for the attenuated total reflection mode (ATR).

X-ray diffraction analysis. Experimental data for compounds **I**–**V** were obtained at room temperature (293(2) K) on an Xcalibur E diffractometer (MoK_α radiation, graphite monochromator). The unit cell parameters refined over the whole set and other experimental data were obtained using the CrysAlis Oxford Diffraction program package [29]. The structures of the compounds were solved by direct methods and refined by least squares in the anisotropic full-matrix variant for non-hydrogen atoms (SHELX-97) [30]. The positions of the hydrogen atoms of the solvate water molecules were determined from the difference Fourier synthesis, and positions of other hydrogen atoms were calculated geometrically. All hydrogen atoms were refined isotropically in the rigid body model with $U_{eff} = 1.2\ U_{eq}$ or $1.5\ U_{eq}$ of the corresponding O, N, and C atoms. The experimental characteristics and structure refinement data for compounds **I**–**V** are presented in Table 1. Selected interatomic distances and bond angles are given in Tables 2 and 3. The geometric parameters of hydrogen bonds are presented in Table 4.

The positional and thermal parameters of atoms in compounds **I**–**V** were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1906360 (**I**), 1906362 (**II**), 1906361 (**III**), 1906363 (**IV**), and 1906364 (**V**)); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The IR spectra of the studied compounds are complicated but bear valuable information about the compositions of the complexes, coordination modes of the ligands to the metal ion, interaction of the ligands, hydrogen bond formation, etc. The spectrum of $DAnH_2$ exhibits rather narrow medium-intensity absorption band at 3370 cm^{-1}, which can be assigned to the ν(NH) vibrations [31]. The intense absorption bands at 3057 and 2780 cm^{-1} can probably be attributed to ν(OH) of the oxime groups involved in the formation of various hydrogen bonds in $DAnH_2$ in the solid state. Four absorption bands of weak intensity are observed in a range of 2000–1667 cm^{-1} at 1941, 1865, 1791, and 1737 cm^{-1} (an overtone and the resulting component bands of the major frequencies of the out-of-plane CH vibrations of the aromatic ring lying in the range 1000–700 cm^{-1}). The intense absorption bands at 752 and 689 cm^{-1} caused by the $\delta(CH_{out\text{-}of\text{-}plane})$ vibrations are characteristic of the monosubstituted benzene ring [31, 32]. It is difficult to identify the δ(CH) planar bending vibrations of the aromatic ring because of other absorption bands manifested in this range. The intense absorption bands at 1637 and 971 cm^{-1} can be attributed to the ν(C=N) and ν(N–O) vibrations of the oxime groups, respectively [32]. The planar vibrations of the C=C skeleton appear as absorption bands at 1595, 1493, and 1432 cm^{-1} [33].

The contours of the spectra of complexes **III**–**V** have many common features. All the spectra contain the ν(NH) absorption bands in a range of 3317–3296 cm^{-1} and have no ν(OH) absorption bands of oxime groups in the spectral range usual for oximes. Probably, two broad and fairly intense absorption bands in the ranges 2700–2200 and 2000–1700 cm^{-1} are assigned to ν(OH) of the oxime groups involved in the formation of rather strong hydrogen bonds differed in length [34, 35].

The asymmetric part of the unit cell of compound **I** contains the $[DAnH_3]^+$ organic cation, ClO_4^- anion, and one water molecule. The molecular structure of compound **I** is shown in Fig. 1a. The $[DAnH_3]^+$ cation has a shape of a butterfly in which two central parts ONCN form a dihedral angle of 54.4° and the oxime groups are in the *anti* (*E,E*) conformation. The O–N and N–C interatomic distances in the oxime fragments differ insubstantially from similar distances in neutral modified dioximes **VI** and **VII** containing the amino group [36, 37]. For example, the O–N interatomic distances in compound **I** are 1.383(4) and 1.408(4) Å (Table 3), those in compound **VI** are 1.434 Å, and those in compound **VII** are equal to 1.422 Å.

In the crystal of compound **I**, the $[DAnH_3]^+$ cations are joined by intermolecular hydrogen bonds O–H···N into the centrosymmetric dimer (Fig. 1b, Table 4), and the cations with the anions are linked by intermolecular hydrogen bonds O–H···O or involve the water molecule by intermolecular hydrogen bonds N–H···O and O–H···O (Fig. 1a, Table 4). The ClO_4^- anions and water molecules are linked into chains (Fig. 1c, Table 4) on which $[DAnH_3]^+$ cations are stringed (Fig. 1d, Table 4).

The asymmetric part of the unit cell of the crystal of compound **II** contains four independent organic cations $[DAnH_3]^+$ (A–D), four ClO_4^- anions (A–D), and five water molecules, two of which are disordered over two positions. The molecular structure of cation A in compound **II** is shown in Fig. 2a, and the structures of all components are shown in Fig. 2b. The $[DAnH_3]^+$ cations in compound **II** are similar to those found in compound **I**. The oxime groups have the *anti* (*E,E*) conformation, and the dihedral angles between the central fragments ONCN in four independent cations take values from 47.3° to 58.4°. The O–N and N–C interatomic distances in the oxime fragments differ insignificantly from similar distances found in compound **I** (Table 3).

Table 1. Crystallographic data and experimental characteristics for the structures of compounds **I–V**

Parameters	Value				
	I	**II**	**III**	**IV**	**V**
Empirical formula	$C_{14}H_{17}N_4O_7Cl$	$C_{56}H_{70}N_{16}O_{28.4}Cl_4$	$C_{63}H_{85}N_{14}O_{20.5}Zn_2$	$C_{62}H_{78}N_{14}O_{18}Mn_2$	$C_{110}H_{130}N_{22}O_{34}Mn_4$
FW	388.77	1563.48	1497.19	1417.26	2524.12
Crystal system	Monoclinic	Triclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$	$P2/n$	$P2_1/n$	$P\bar{1}$
Unit cell parameters					
a, Å	12.2803(7)	10.7746(7)	14.1178(5)	9.4953(5)	13.9891(9)
b, Å	10.6201(7)	12.8312(8)	9.1592(4)	13.7464(5)	14.6400(8)
c, Å	14.2762(6)	26.7126(16)	28.4951(9)	26.9683(13)	16.2122(9)
α, deg	90	89.810(5)	90	90	93.752(4)
β, deg	92.367(5)	84.983(5)	90.826(3)	93.122(5)	113.819(6)
γ, deg	90	85.435(5)	90	90	94.536(5)
V, Å^3	1860.27(18)	3667.2(4)	3684.3(2)	3514.8(3)	3010.6(3)
Z	4	2	2	2	1
ρ_{calc}, g/cm^3	1.388	1.416	1.350	1.339	1.392
μ, mm^{-1}	0.248	0.253	0.729	0.436	0.497
F(000)	808	1626	1570	1484	1316
Crystal sizes, mm	0.35 × 0.18 × 0.01	0.35 × 0.15 × 0.08	0.42 × 0.36 × 0.22	0.35 × 0.2 × 0.08	0.2 × 0.18 × 0.06
Range of θ, deg	2.94–25.50	2.92–25.50	3.00–25.05	2.96–25.05	2.97–25.05
Ranges of reflection indices	$-14 \le h \le 14$, $-12 \le k \le 12$, $-17 \le l \le 11$	$-11 \le h \le 13$, $-15 \le k \le 15$, $-20 \le l \le 32$	$-16 \le h \le 16$, $-10 \le k \le 9$, $-33 \le l \le 25$	$-11 \le h \le 6$, $-16 \le k \le 16$, $-32 \le l \le 28$	$-15 \le h \le 16$, $-17 \le k \le 16$, $-19 \le l \le 14$
Number of measured/ independent reflections (R_{int})	6375/ 3446 (0.0370)	20 663/ 13 525 (0.0442)	11 720/ 6486 (0.0378)	13 487/ 6204 (0.0583)	16 374/ 10 611 (0.0413)
Number of reflections with $I > 2\sigma(I)$	1907	6545	4493	3230	6024
Filling, %	99.4 (θ =25.50°)	99.1 (θ = 25.50°)	99.5 (θ = 25.05°)	99.7 (θ = 25.05°)	99.5 (θ = 25.05°)
Number of refined parameters	235	955	470	440	757
GOOF	1.005	1.000	1.003	1.005	1.000
R factors ($I > 2\sigma(I)$)	$R_1 = 0.0619$, $wR_2 = 0.1542$	$R_1 = 0.0818$, $wR_2 = 0.1921$	$R_1 = 0.0648$, $wR_2 = 0.1670$	$R_1 = 0.0713$, $wR_2 = 0.1330$	$R_1 = 0.0722$, $wR_2 = 0.1413$
R factors (for all data)	$R_1 = 0.1306$, $wR_2 = 0.1817$	$R_1 = 0.1656$, $wR_2 = 0.2386$	$R_1 = 0.0991$, $wR_2 = 0.1911$	$R_1 = 0.1440$, $wR_2 = 0.1669$	$R_1 = 0.1367$, $wR_2 = 0.1714$
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.260/–0.236	0.529/–0.406	0.589/–0.447	0.452/–0.393	0.697*–0.506

Table 2. Interatomic distances (*d*) and bond angles (ω) in the coordination polyhedra of the structures of compounds **III**–**V**

Bond	*d*, Å			
	III (M = Zn(1))	**IV** (M = Mn(1))	**V** (M = Mn(1)/Mn(2))	
			A	*B*
M–N(1)	2.168(3)	2.310(3)	2.320(3)	2.306(3)
M–N(2)	2.302(2)	2.371(3)	2.322(4)	2.347(4)
M–O(3)	2.122(4)	2.172(4)	2.131(4)	2.173(4)
M–O(4)	2.207(5)	2.189(4)	2.251(4)	2.184(4)
M–O(5)	2.014(3)	2.086(3)	2.152(4)	2.110(3)
M–O(7)$^{\#1}$	2.035(3)	2.102(3)	2.106(3)	2.126(3)
Angle	ω, deg			
	III (M = Zn(1))	**IV** (M = Mn(1))	**V** (M = Mn(1)/Mn(2))	
			A	*B*
N(1)MN(2)	71.6(1)	69.0(1)	69.9(1)	68.7(1)
N(1)MO(3)	91.7(1)	85.3(2)	90.4(1)	91.4(1)
N(1)MO(4)	85.0(2)	85.4(2)	89.4(1)	89.6(2)
N(1)MO(5)	169.0(1)	166.3(1)	96.8(1)	161.8(1)
N(1)MO(7)$^{\#1/\#2}$	100.9(1)	99.3(1)	168.7(2)	97.3(1)
N(2)MO(3)	90.9(2)	91.3(2)	96.4(2)	87.9(1)
N(2)MO(4)	86.3(2)	86.2(1)	84.6(1)	99.4(1)
N(2)MO(5)	97.7(1)	97.3(1)	163.8(1)	93.5(1)
N(2)MO(7)$^{\#1/\#2}$	172.5(1)	168.0(1)	99.1(1)	165.1(1)
O(3)MO(4)	176.0(2)	170.7(2)	178.9(2)	172.5(2)
O(3)MO(5)	91.0(2)	96.8(2)	92.7(1)	91.4(1)
O(3)MO(7)$^{\#1/\#2}$	90.2(2)	90.5(2)	93.3(1)	87.5(1)
O(4)MO(5)	91.9(2)	92.4(2)	86.2(2)	90.0(2)
O(4)MO(7)$^{\#1/\#2}$	92.3(2)	90.1(2)	87.0(1)	84.9(1)
O(5)MO(7)$^{\#1/\#2}$	89.7(1)	94.2(1)	93.7(1)	100.8(1)

* Symmetry codes: $^{\#1}$ $-x + 2, -y + 1, -z + 1$ (**III**); $^{\#1}$ $-x + 1, -y + 2, -z + 1$ (**IV**); $^{\#1}$ $-x + 2, -y + 2, -z + 1$; $^{\#2}$ $-x, -y + 1, -z$ (**V**).

Table 3. Interatomic distances (*d*) and bond angles (ω) in the fragments of the organic cations of compounds **I** and **II** and coordinated ligands **III**–**V**

Bond	*d*, Å								
	I	**II**				**III**	**IV**	**V**	
		A	*B*	*C*	*D*			*A*	*B*
N(1)–C(1)	1.299(4)	1.293(5)	1.287(5)	1.289(6)	1.281(6)	1.288(5)	1.293(5)	1.286(6)	1.284(5)
N(1)–O(1)	1.383(4)	1.413(4)	1.402(4)	1.380(4)	1.414(4)	1.397(4)	1.396(4)	1.398(4)	1.394(4)
N(2)–C(2)	1.290(5)	1.302(5)	1.305(5)	1.292(5)	1.316(5)	1.289(5)	1.294(5)	1.292(5)	1.289(5)
N(2)–O(2)	1.408(4)	1.392(4)	1.381(4)	1.416(4)	1.380(4)	1.396(4)	1.399(4)	1.401(4)	1.400(4)
C(1)–C(2)	1.492(5)	1.509(6)	1.487(6)	1.502(6)	1.473(6)	1.502(5)	1.489(5)	1.488(6)	1.481(6)
Angle	ω, deg								
	I	**II**				**III**	**IV**	**V**	
		A	*B*	*C*	*D*			*A*	*B*
C(1)N(1)O(1)	117.4(3)	107.8(3)	108.4(3)	117.2(4)	109.8(4)	112.0(3)	111.4(3)	110.6(3)	111.3(3)
C(2)N(2)O(2)	109.5(3)	117.1(3)	117.9(4)	109.5(3)	116.9(4)	111.1(3)	110.5(3)	111.2(4)	110.3(4)
N(1)C(1)C(2)	114.1(3)	113.4(4)	113.6(4)	114.2(5)	113.9(5)	113.1(3)	112.9(4)	113.9(4)	113.7(4)
N(2)C(2)C(1)	113.4(3)	113.7(4)	115.3(4)	113.2(4)	115.1(4)	111.8(4)	113.0(4)	112.9(4)	113.3(4)

Table 4. Geometric parameters of intra- and intermolecular hydrogen bonds in the structures of compounds **I–V**

Contact D–H···A	Distance, Å			Angle DHA, deg	Coordinates of atoms A
	D–H	H···A	D···A		
			I		
N(1)–H···O(1*w*)	0.89	1.79	2.677(4)	174	*x*, *y*, *z*
N(3)–H···O(5)	0.86	2.26	3.082(4)	161	–*x* + 1, –*y* + 2, –*z* + 1
N(4)–H···O(5)	0.86	2.22	3.027(4)	157	–*x* + 1, *y* – 1/2, –*z* + 3/2
O(1)–H···N(2)	0.82	1.87	2.684(4)	175	–*x* + 1, –*y* + 2, –*z* + 1
O(2)–H···O(3)	0.82	2.09	2.895(5)	168	*x*, *y*, *z*
O(1*w*)–H(1)···O(4)	0.82	2.03	2.812(4)	159	*x*, *y*, *z*
O(1*w*)–H(2)···O(6)	0.92	2.17	3.016(4)	153	*x*, –*y* + 3/2, *z* – 1/2
O(1*w*)–H(2)···O(3)	0.92	2.40	3.144(4)	138	*x*, –*y* + 3/2, *z* – 1/2
			II		
N(2*A*)–H···O(1*w*)	1.06	1.70	2.716(5)	159	*x*, *y*, *z*
N(3*A*)–H···O(5*B*)	0.86	2.34	3.105(6)	148	*x*, *y*, *z*
N(4*A*)–H···O(6*B*)	0.86	2.10	2.860(5)	146	*x* + 1, *y*, *z*
N(2*B*)–H···O(3*w*)	0.74	2.04	2.743(5)	158	*x*, *y*, *z*
N(3*B*)–H···O(5*A*)	0.86	2.39	3.089(5)	139	*x* – 1, *y*, *z*
N(3*B*)–H···O(6*w*)	0.86	2.37	3.07(2)	138	*x* – 1, *y*, *z*
N(4*B*)–H···O(6*A*)	0.86	2.40	3.102(5)	139	*x*, *y*, *z*
N(4*B*)–H···O(4*A*)	0.86	2.41	3.189(6)	151	*x*, *y*, *z*
N(1*C*)–H···O(4*w*)	0.91	1.74	2.648(6)	175	*x*, *y*, *z*
N(2*D*)–H···O(2*w*)	0.84	1.83	2.662(5)	168	–*x* + 1, –*y* + 1, –*z*
N(4*D*)–H···O(6*C*)	0.86	2.47	3.101(7)	131	*x* – 1, *y*, *z*
N(4*D*)–H···O(4*C*)	0.86	2.37	3.157(6)	153	*x* – 1, *y*, *z*
O(1*A*)–H···N(1*D*)	0.82	1.97	2.791(5)	175	–*x* + 1, –*y* + 1, –*z*
O(2*A*)–H···N(1*A*)	0.82	1.86	2.677(4)	180	–*x* + 2, –*y* + 2, –*z*
O(1*B*)–H···O(5*w*)	0.82	1.83	2.653(5)	177	*x* – 1, *y*, *z*
O(2*B*)–H···O(4*A*)	0.82	1.91	2.722(6)	170	–*x* + 1, –*y* + 2, –*z* + 1
O(1*C*)–H···N(2*C*)	0.82	1.83	2.651(5)	175	–*x* + 1, –*y* + 1, –*z* + 1
O(2*C*)–H···N(1*B*)	0.81	1.93	2.744(5)	177	–*x* + 1, –*y* + 1, –*z* + 1
O(1*D*)–H···O(1*A*)	0.82	2.36	3.089(4)	149	*x*, *y* – 1, *z*
O(2*D*)–H···O(4*C*)	0.82	1.86	2.675(7)	174	–*x* + 1, –*y* + 1, –*z*
O(1*w*)–H(1)···O(5*C*)	0.85	2.32	3.078(9)	149	*x*, *y* + 1, *z*
O(1*w*)–H(2)···O(2*B*)	0.85	2.26	3.108(7)	179	–*x* + 1, –*y* + 2, –*z*
O(2*w*)–H(2)···O(3*B*)	0.82	2.07	2.870(6)	167	*x*, *y*, *z*
O(3*w*)–H(1)···O(5*A*)	0.89	2.16	3.047(7)	175	–*x* + 1, –*y* + 2, –*z* + 1
O(3*w*)–H(2)···O(2*C*)	0.85	2.19	3.040(6)	179	–*x* + 1, –*y* + 1, –*z* + 1
O(4*w*)–H(1)···O(5*w*)	0.88	1.91	2.764(5)	164	*x*, *y*, *z*
O(4*w*)–H(2)···O(3*A*)	0.85	2.26	3.115(7)	180	*x*, *y*, *z*
O(5*w*)–H(1)···O(5*D*)	1.06	2.01	3.06(1)	170	–*x* + 1, –*y* + 1, –*z* + 1
O(5*w*)–H(2)···N(2*C*)	0.88	2.41	3.286(6)	171	*x*, *y*, *z*
O(5*w*)–H(2)···O(2*C*)	0.88	2.20	2.952(6)	142	*x*, *y*, *z*
O(6*w*)–H(1)···O(4*D*)	0.91	2.01	2.92(2)	176	*x* + 1, *y*, *z*
O(6*w*)–H(2)···O(5*A*)	0.77	2.03	2.80(2)	178	*x*, *y*, *z*
			III		
N(3)–H···O(1)	0.86	2.33	3.159(5)	162	–*x* + 5/2, *y*, –*z* + 1/2

Table 4. (Contd.)

Contact D–H···A	Distance, Å			Angle DHA, deg	Coordinates of atoms A
	D–H	H···A	D···A		
N(4)–H···O(2)	0.86	2.25	3.087(5)	164	−*x* + 3/2, *y*, −*z* + 1/2
O(1)–H···O(8)	0.82	1.71	2.508(4)	163	−*x* + 2, −*y* + 1, −*z* + 1
O(2)–H···O(6)	0.82	1.74	2.523(4)	158	*x*, *y*, *z*
O(1*M*)–H···O(1)	0.85	2.39	3.19(3)	178	*x* – 1, *y*, *z*
O(1*w*)–H(1)···O(1*w*)	0.96	2.56	3.39(6)	144	−*x* + 1/2, *y*, −*z* + 1/2
O(3*w*)–H(1)···O(3*w*)	0.98	1.82	2.75(8)	158	−*x* + 1/2, *y*, −*z* + 1/2
			IV		
O(2)–H···O(6)	0.82	1.73	2.539(4)	171	*x*, *y*, *z*
O(1)–H···O(8)	0.82	1.72	2.522(4)	166	−*x* + 1, −*y* + 2, −*z* + 1
N(4)–H···O(1)	0.86	2.14	2.960(4)	159	−*x* + 1/2, *y*–1/2, −*z* + 3/2
N(3)–H···O(2)	0.86	2.34	3.179(4)	167	−*x* + 1/2, *y* + 1/2, −*z* + 3/2
			V		
N(3*A*)–H···O(8*B*)	0.86	2.24	3.007(5)	148	−*x*, −*y* + 1, −*z*
N(4*A*)–H···O(2*B*)	0.86	2.22	3.044(5)	162	*x*, *y* + 1, *z*
N(3*B*)–H···O(6*A*)	0.86	2.41	3.258(5)	171	*x*, *y*, *z*
N(4*B*)–H···O(2*A*)	0.86	2.27	3.085(5)	158	*x*, *y* – 1, *z*
O(1*A*)–H···O(6*A*)	0.82	1.81	2.626(5)	172	*x*, *y*, *z*
O(2*A*)–H···O(8*A*)	0.82	1.71	2.524(4)	170	−*x* + 2, −*y* + 2, −*z* + 1
O(4*A*)–H···O(1*M*)	0.93	1.78	2.689(6)	164	*x*, *y*, *z*
O(1*B*)–H···O(8*B*)	0.82	1.73	2.549(4)	173	−*x*, −*y* + 1, −*z*
O(2*B*)–H···O(6*B*)	0.82	1.67	2.481(5)	171	*x*, *y*, *z*
O(1M)–H···O(5*A*)	0.79	2.04	2.823(6)	176	*x*, *y*, *z*

In the crystal of compound **II**, the ClO_4^- anions and water molecules are linked to form various synthones (Figs. 2c and 2d, Table 4) and the $[DAnH_3]^+$ cations are linked to each other and with the anions by a branched system of intermolecular hydrogen bonds O–H···O (Fig. 3, Table 4).

Attempts to obtain complexes of various metals with mixed ligands, namely, 1,3-H_2Bdc and synthesized $DAnH_2$, gave one Zn(II) complex (**III**) and two Mn(II) complexes (**IV** and **V**).

The nonsymmetric part of the unit cell of compound **III** contains one crystallographically independent Zn^{2+} cation that coordinates one neutral $DAnH_2$, bideprotonated carboxylate $(1,3\text{-Bdc})^{2-}$, two DMF molecules, and DMF, MeOH, and H_2O molecules of crystallization in a ratio of 2 : 1 : 1.5, respectively. As a result, the crystal contains the centrosymmetric binuclear molecular complexes, two metal atoms of which are linked by the bridging bidentate carboxylate ligands (Fig. 4). The metal atom coordinates the $DAnH_2$ ligand via the chelate mode by two nitrogen atoms and two $(1,3\text{-Bdc})^{2-}$ ligands via the monodentate mode by one oxygen atom. Its environment is built up by two oxygen atoms of two monodentate DMF ligands. The coordination polyhedron of the Zn(II) atom is the N_2O_4 octahedron. The Zn–N(oxime) interatomic distances in the equatorial plane of the coordination polyhedron of the metal are 2.167(3) and 2.300(4) Å, the Zn–O(Bdc) distances are 2.013(3) and 2.037(3) Å, and the Zn–O(DMF) axial distances are equal to 2.123(4) and 2.205(5) Å (Table 2), which is consistent with the data on the Zn(II) complex with dioxime [38]. In the complex of compound **III**, each neutral $DAnH_2$ ligand forms two intramolecular hydrogen bonds O–H···O with two $(1,3\text{-Bdc})^{2-}$ (O···O 2.508(4) and 2.521(4) Å, Table 4, Fig. 4). An analysis of the CCDC data [6] revealed the binuclear Zn(II) complex with dioxime containing bipyridine as the bridging ligand [22]. Both mononuclear [38] and polymer compounds [39] were obtained for the Zn(II) compounds with mixed ligands, dioxime, and carboxylates.

The binuclear complexes in the crystal of compound **III** are linked into layers parallel to the *ac* plane by intermolecular hydrogen bonds N–H···O, where the functional NH groups act as proton donors and the oxygen atoms of the oxime ligands are acceptors.

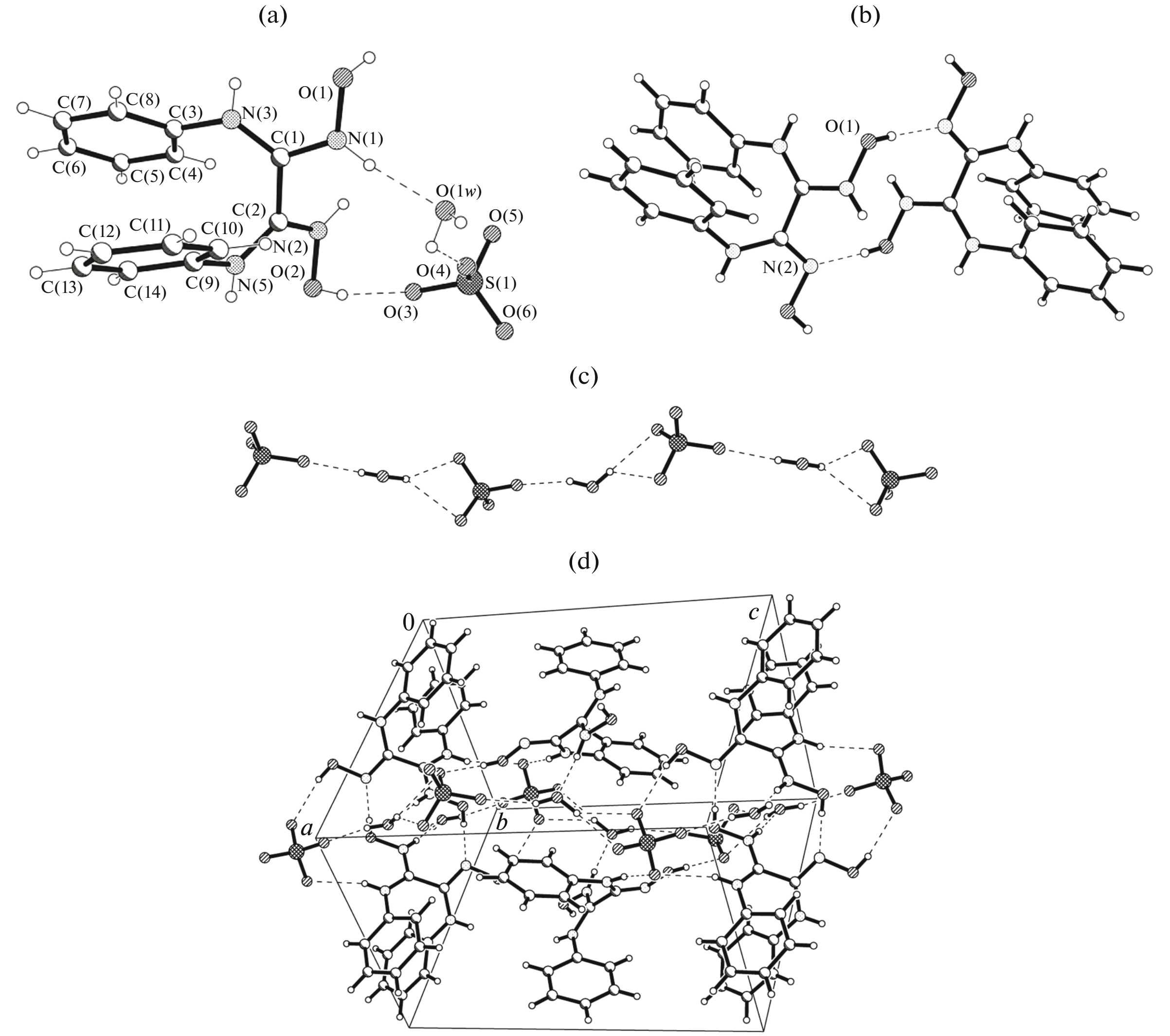

Fig. 1. (a) Structure of compound **I**, (b) centrosymmetric dimers of the $[DAnH_3]^+$ cations linked to each other by intermolecular hydrogen bonds O–H···N, (c) chains of the ClO_4^- anions and water molecules, and (d) packing fragment of the components in compound **I**.

The layers are additionally stabilized by weaker hydrogen bonds C–H···O: C(4)–H···O(8) ($x + 1/2$, $-y + 1$, $z - 1/2$), H···O 2.47, C···O 3.380 Å, angle CHO 166°; C(14)–H···O(6) ($-x + 3/2$, y, $-z + 1/2$) – H···O 2.57, C···O 3.489 Å, angle CHO 170° (Fig. 5). The molecules of crystallization are linked to the complexes by weak hydrogen bonds O–H···O and C–H···O (C(5)–H···O(9) ($x + 1$, y, $z - 1$), H···O 2.71, C···O 3.415 Å, angle CHO 133°).

The nonsymmetric part of the unit cell of the Mn(II) compound with the same ligands (**IV**) contains one crystallographically independent metal cation (coordinated to the neutral $DAnH_2$ ligand, bideprotonated carboxylate $(1,3\text{-Bdc})^{2-}$, and two DMF molecules) and one outer-sphere DMF molecule. As a result, the centrosymmetric binuclear molecular Mn(II) complexes (Fig. 6) similar to those found in compound **III** are formed in the crystal of compound **IV**. The coordination polyhedra of the metal atoms are octahedra formed by a set of donor atoms N_2O_4 belonging to $DAnH_2$ coordinating via the chelate mode, two $(1,3\text{-Bdc})^{2-}$ ligands, and two coordinated DMF molecules. The Mn–N(oxime) interatomic distances (2.310(3) and 2.371(4) Å, Table 2) are consistent with the data for the Mn(II) complexes with dioxime [40–42], and the Mn–O(Bdc) distances equal to 2.086(3) and 2.102(3) Å correspond to the data for the Mn(II) complex with $(1,3\text{-Bdc})^{2-}$ characterized by a similar coordination mode in the Mn(II) polymers [43, 44]. The Mn–O(DMF) bond lengths are 2.172(4) and 2.189(4) Å. Three trinuclear Mn(II) complexes with dioximes presented in the CCDC [6] are coordinated via the chelate mode (N, O) to one

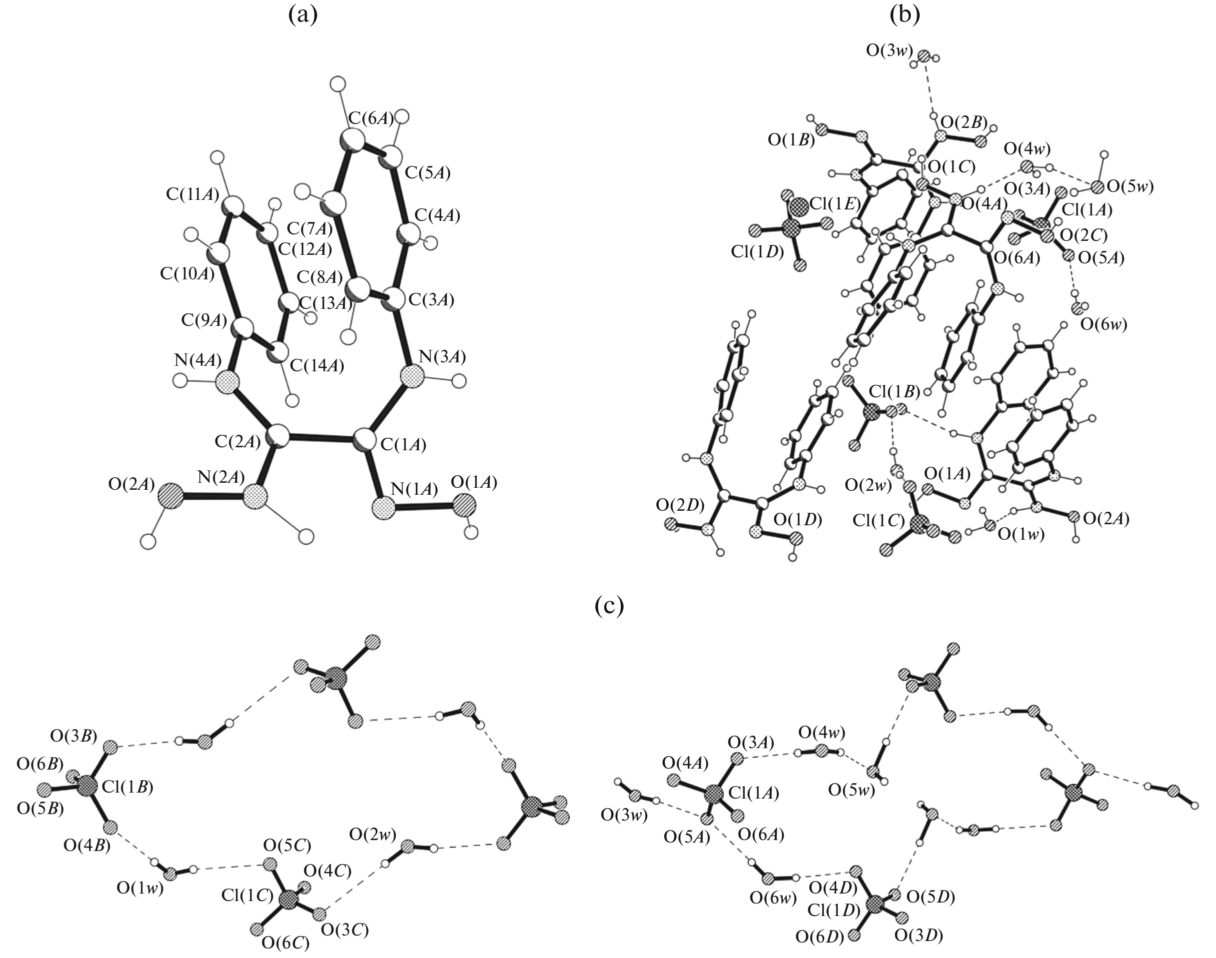

Fig. 2. (a) Structure of cation **A** in compound **II**, (b) packing of the components, and (c) formation of various synthones from the anions and water molecules by hydrogen bonds.

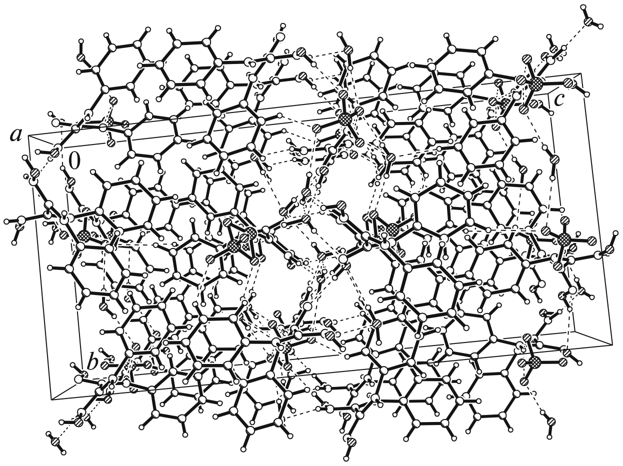

Fig. 3. Fragment of the crystal structure of compound **II**.

metal atom and to another metal atom by another oxygen atom [45, 46]. Each neutral $DAnH_2$ ligand in complex **IV** forms two intramolecular hydrogen bonds O–H···O with two $(1,3\text{-Bdc})^{2-}$ (O···O 2.539(4) and 2.522(4) Å, Table 4, Fig. 6). In the crystal of compound **IV** (Fig. 7), the binuclear complexes are linked to each other by intermolecular hydrogen bonds N–H···O and C–H···O into layers (similar to those in compound **III**) parallel to the *bc* plane. The outer-sphere DMF molecules are bound to the complexes by weak hydrogen bonds (C(13)–H···O(9) ($x - 1/2, -y + 3/2, z + 1/2$): H···O (2.63 Å) and C···O (3.412 Å), angle CHO 142°.

In the Mn(II) compound with the same ligands (**V**), the nonsymmetric part of the unit cell contains two crystallographically independent Mn^{2+} cations, two neutral ligands $DAnH_2$, two bideprotonated carboxylates $(1,3\text{-Bdc})^{2-}$, three DMF molecules, and two methanol molecules coordinated to the metal atoms, as well as the methanol molecule of crystallization. As

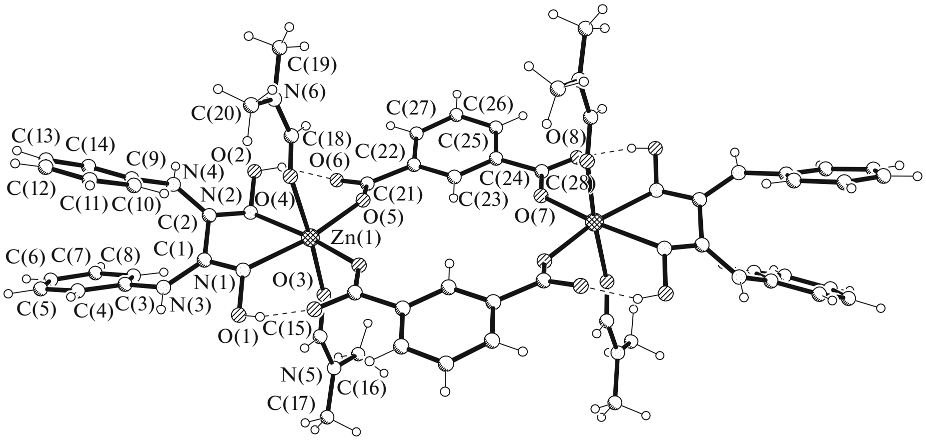

Fig. 4. Structure of the centrosymmetric binuclear Zn(II) complex in compound **III**.

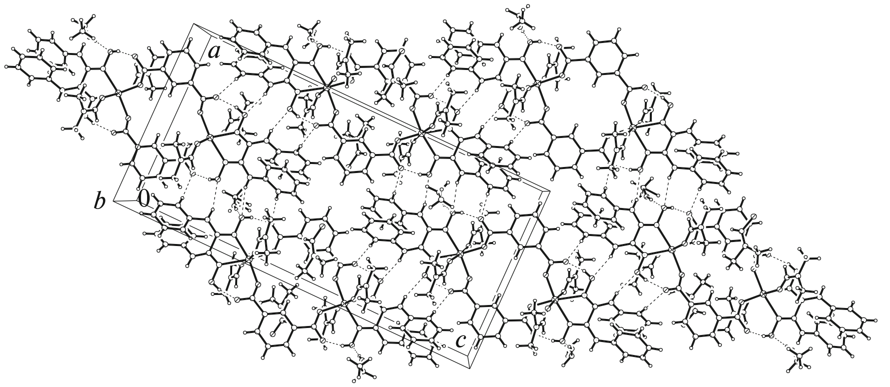

Fig. 5. Formation of the layer of the binuclear Zn(II) complexes in compound **III**.

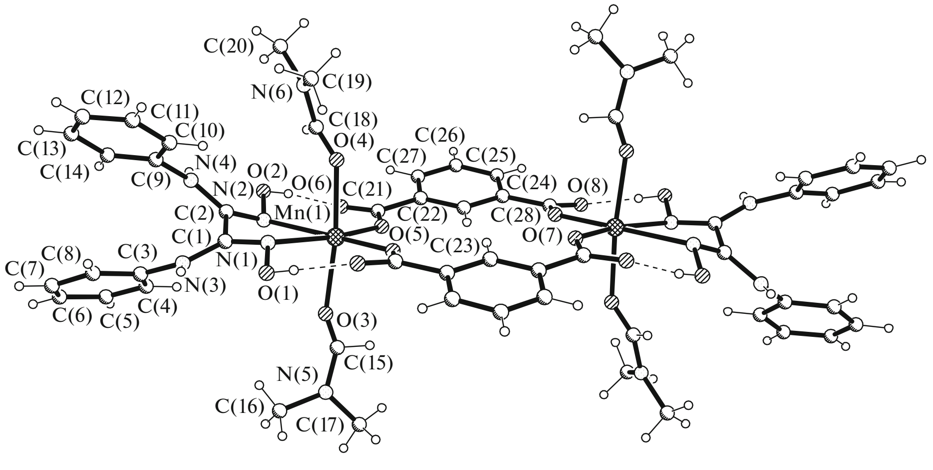

Fig. 6. Structure of the centrosymmetric binuclear Mn(II) complex in compound **IV**.

a result, the crystal of compound **V** contains two centrosymmetric binuclear molecular complexes (A and B) differed in axial ligands. In the same complex, neutral methanol and DMF molecules coordinate in the *trans* positions via the monodentate mode to the Mn(1) atom, and two DMF molecules coordinate to the Mn(2) atom (Fig. 8). The octahedral coordination polyhedra of the Mn(1) and Mn(2) atoms are formed by the same set of donor atoms N_2O_4 as in compound **IV**. Four atoms N_2O_2 are arranged in the equatorial planes of the coordination polyhedra of the metal atoms: two nitrogen atoms of one ligand $DAnH_2$ and two oxygen

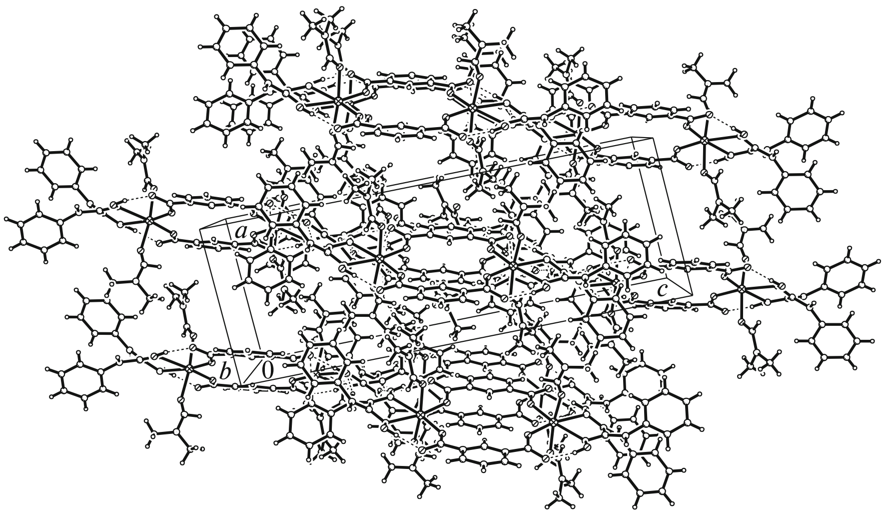

Fig. 7. Fragment of the crystal structure of compound **IV** and the packing of the layers of the binuclear Mn(II) complexes.

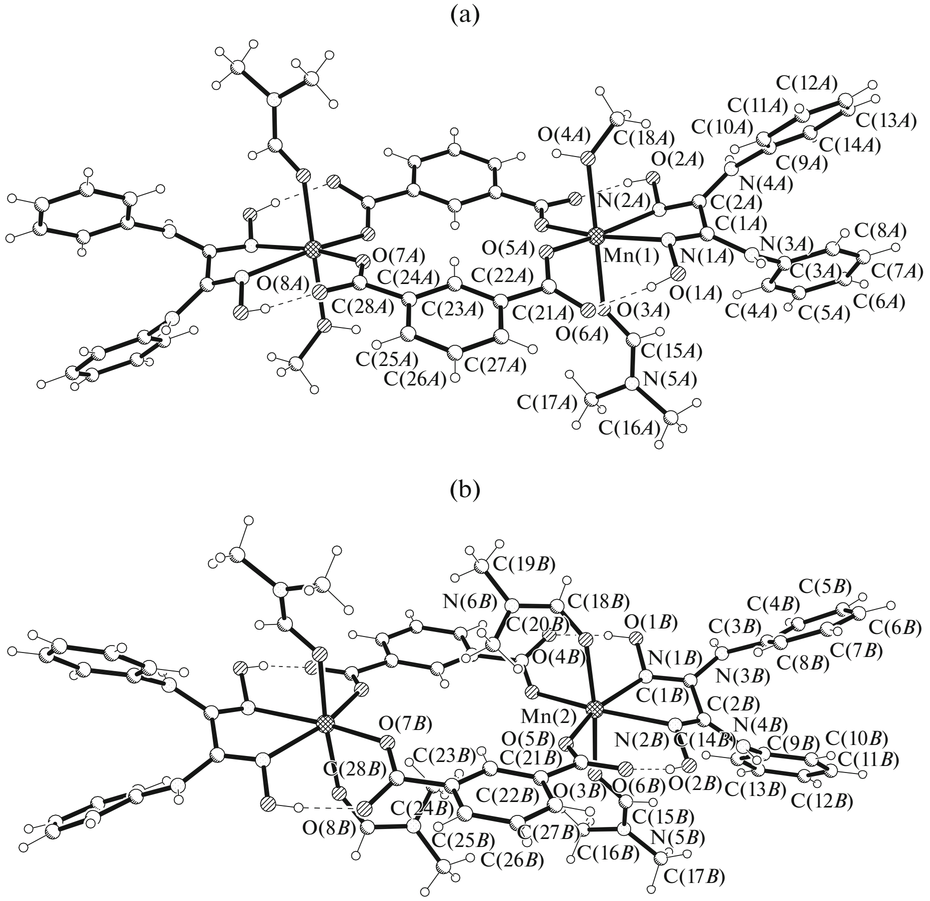

Fig. 8. Structure of the centrosymmetric binuclear complexes $[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_2(CH_3OH)_2]$ and $[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4]$ in compound **V**.

atoms of two ligands $(1,3\text{-Bdc})^{2-}$. Each neutral ligand $DAnH_2$ coordinates to one metal atom of the dimer via the chelate mode to form five-membered metallocycles. Each doubly deprotonated bridging ligand $(1,3\text{-Bdc})^{2-}$ in compound **V**, as in compounds **III** and **IV**, is coordinated by two oxygen atoms of two carboxyl groups to the central atoms. The interatomic distances (Mn–N(oxime) 2.306(3)–2.347(4), Mn–O(Bdc)

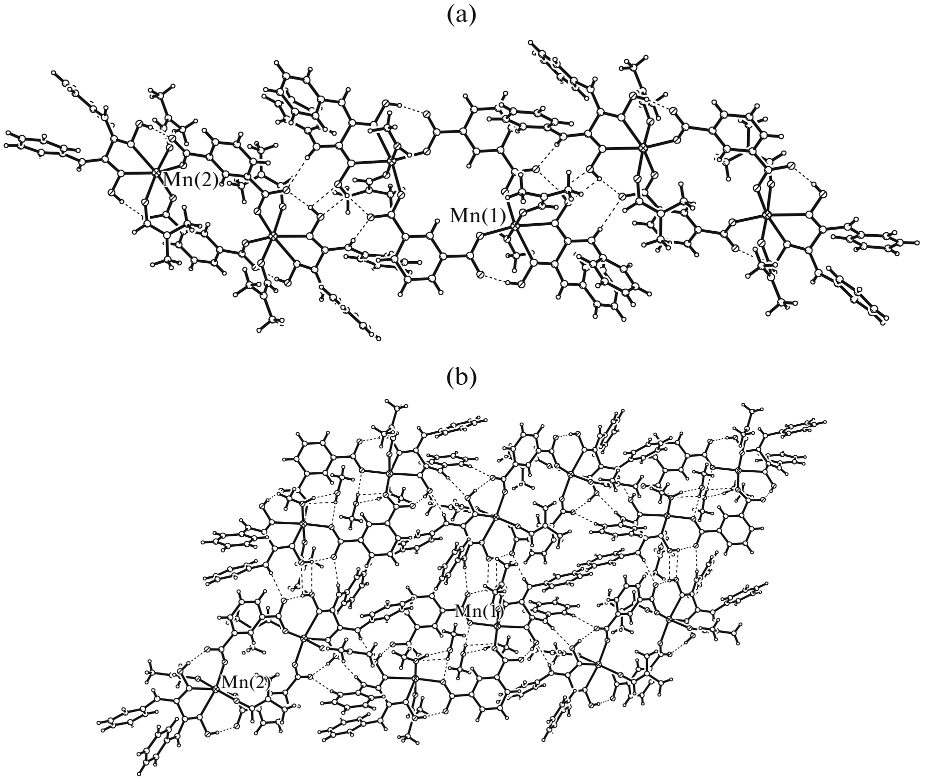

Fig. 9. (a) Fragment of the chain formed by the alternation of complexes A (Mn(1)) and B (Mn(2)) in compound **V** and (b) joining of the chains into layers.

2.106(3)–2.152(4), and Mn–O(DMF) 2.131(4)–2.184(4) Å) (Table 2) are consistent with the data on complex **IV**, and the Mn–O(methanol) distance in compound **V** is 2.251(4) Å. The binuclear complexes in compound **V** are stabilized in the same way as those in compounds **III** and **IV** by two intramolecular hydrogen bonds O–H···O (O···O 2.481(5)–2.625(5) Å, Table 4, Fig. 8). The outer-sphere methanol molecules also stabilize complexes A to bind the latter by intermolecular hydrogen bonds O–H···O in which they are involved as both the proton donor and acceptor (O(1*M*)–H···O(5*A*), O(4*A*)–H···O(1*M*), Table 4).

In the crystal of compound **V** (Fig. 9), binuclear alternating complexes A and B are bound to each other by intermolecular hydrogen bonds N–H···O and C–H···O into chains (Fig. 9a), which are joined by hydrogen bonds into layers (Fig. 9b) similar to those formed in compounds **III** and **IV**.

Thus, the reactions of the zinc(II) and manganese(II) salts with dianilineglyoxime and 1,3-benzenedicarboxylic acid afforded three new binuclear compounds: $[Zn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF \cdot MeOH \cdot 1.5H_2O$, $[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2DMF$, and $\{[Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_2(CH_3OH)_2][Mn_2(DAnH_2)_2(1,3\text{-}Bdc)_2(DMF)_4] \cdot 2CH_3OH\}$.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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