

NOVEL Zn(II) BINUCLEAR AND Ni(II) 1D POLYMERIC COORDINATION COMPOUNDS BASED ON DIANILINEGLYOXIME AND DICARBOXYLIC ACIDS: SYNTHESIS AND STRUCTURE

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Abstract. Two coordination compounds $[Zn_2(DAnH_2)_2(1,3\text{-}bdc)_2(\text{DMSO})_4]$ (**1**) and $[Ni(DAnH_2)(1,4\text{-}bdc)(\text{DMF})_2]_n$ (**2**) were synthesized starting from dianilineglyoxime ($DAnH_2$), 1,3-benzenedicarboxylic acid ($1,3\text{-}bdcH_2$) and 1,4-benzenedicarboxylic acid ($1,4\text{-}bdcH_2$), where DMSO is dimethyl sulphoxide and DMF is dimethylformamide. The molecular and crystal structures of the compounds were studied by infrared spectroscopy and single crystal X-ray diffraction; and for the Zn(II) compound, additionally, the ^1H and ^{13}C NMR spectroscopy was used. The results show that **1** is a binuclear molecular complex while **2** is a unidimensional coordination polymer. In both compounds, the neutral $DAnH_2$ ligand coordinates in a bidentate-chelate mode, while dianions 1,3-bdc and 1,4-bdc coordinate as bidentate bridges. The ligands are interconnected by intramolecular O-H···O hydrogen bonds, involving the oximic groups as proton donors and the carboxylate anions as acceptors. The metal atoms in both compounds have an octahedral geometry.

Keywords: dianilineglyoxime, zinc complex, nickel unidimensional coordination polymer, benzenedicarboxylic acid, X-ray crystallography.

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Introduction

The synthesis of dioxime-based complex compounds began in 1905 when L. Chugaev showed that α -dioximes easily coordinated to some transition metals [1]. Nowadays, a large number of coordination compounds based on dioxime ligands are known, and the evolution from mononuclear to bi- and polynuclear complexes is taking place. Those compounds are characterized by high stability, characteristic colours, and specific behaviour towards solvents at high temperatures.

It is well known that in complexes with advanced nuclearity, the function of bridging ligands is realized by additional organic molecules, such as bipyridine [2-5], polycarboxylic [6-9] or inorganic [10,11] anions. A large number of binuclear compounds, including the bridging ligands containing oximic groups [12-16] or a combination of these functions [17], are known. Moreover, compounds that contain modified bridging ligands have already been successfully created [18,19]. A significant number of publications deal with a series of square planar nickel(II), palladium(II) and platinum(II) dioximates with the coordination

number (CN) of the central atom equal to four [20-23], copper(II) binuclear dioximates with tetragonal-pyramidal structure with CN 5 [24-26], and iron(II) [27-29], iron(III) [30], cobalt(II) [31-33], and cobalt(III) [33-37] compounds with metals octahedral geometry with CN 6. In *bis*-dioxime complexes, those ligands usually coordinate as monoanions and participate in the formation of strong intramolecular hydrogen bonds. In addition, iron(II) [38], nickel(II), cobalt(II), copper(II) [39-41], and copper(I) [42] complexes are known where oximes coordinate as neutral ligands, a part of them being *tris*-dioximic compounds. In some complexes, dianilineglyoxime can coordinate with metal atoms in its neutral [43,44], monodeprotonated [43], or bideprotonated [44] forms. Moreover, it has been established that the zinc(II) complex with dianilineglyoxime ($DAnH_2$) and 1,3-benzenedicarboxylic acid ($1,3\text{-}H_2bdc$) [7] in dimethyl sulphoxide (DMSO) undergoes modifications over time into a known 3D coordination polymer [45], where DMSO replaces both dimethylformamide (DMF) and $DAnH_2$ ligands. Recently, new coordination compounds of zinc with fluorine-containing ligands, which

exhibit luminescent properties, have been reported. Based on them, the aggregation-induced emission effect (AIE) was investigated [46,47].

The purpose of the present study was to synthesize new coordination compounds via the interaction of Zn(II) and Ni(II) salts with DAnH₂, 1,3-bdcH₂, and 1,4-benzenedicarboxylic acid (1,4-bdcH₂), and the resulted Zn(II) binuclear complex [Zn₂(DAnH₂)₂(1,3-bdc)₂(DMSO)₄] (**1**) and Ni(II) one-dimensional coordination polymer [Ni(DAnH₂)(1,4-bdc)(DMF)₂]_n (**2**).

Experimental Materials

All reagents were used as purchased from commercial sources. The starting materials were: nickel(II) acetate tetrahydrate, zinc(II) acetate dihydrate, methanol, DMF, ethanol, DMSO, 1,3- and 1,4-bdcH₂, all being purchased from Sigma Aldrich. DAnH₂ was synthetized by condensation of dichloroglyoxime with aniline, according to the method in [48].

General procedure of syntheses

Compounds **1** and **2** have been synthesized according to Scheme 1.

Synthesis of [Zn₂(DAnH₂)₂(1,3-bdc)₂(DMSO)₄] (**1**)

The dianilinoglyoxime (0.054 g, 0.2 mmol) and zinc acetate dihydrate (0.022 g, 0.1 mmol) were dissolved in a DMSO-EtOH mixture (6 mL) in a ratio of 1:1 (v/v). A solution of 1,3-benzenedicarboxylic acid (0.034 g, 0.2 mmol) in EtOH (3 mL) was further added to the obtained mixture, with permanent stirring. The resulted light-yellow reaction mixture was additionally stirred for 10–15 min and then filtered, and the filtrate was left at room temperature for crystallization. After 16 days, block colorless crystals were formed. The compound is soluble in DMSO and insoluble in DMF, methanol, water, and diethyl ether. Yield: 0.09 g, 35 %. Calcd., %: C, 49.41; H, 4.78; N, 8.86; Zn, 10.35; S, 10.15 for C₅₂H₆₀N₈O₁₃S₄Zn₂. Found, %: C, 50.04; H, 4.98; N, 8.99; Zn, 10.37; S, 10.11. The IR spectra (ν , cm⁻¹): 3663 w, 3304 w, 2989 w, 2972 w, 2909 w, 2554 w, 1841 w, 1670 w, 1624 m, 1600 s, 1540 m, 1497 s, 1472 w, 1452 w, 1437 m,

1410 vw, 1375 vs, 1314 w, 1293 w, 1282 vw, 1265 vw, 1178 w, 1158 w, 1104 vw, 1090 vw, 1078 m, 1065 w, 1009 vs, 949 m, 934 vw, 913 w, 895 w, 836 w, 807 w, 745 s, 708 s, 694 s, 658 w, 633 w, 607 w, 589 w, 545 w, 516 w, 487 m, 451 w, 428 w.

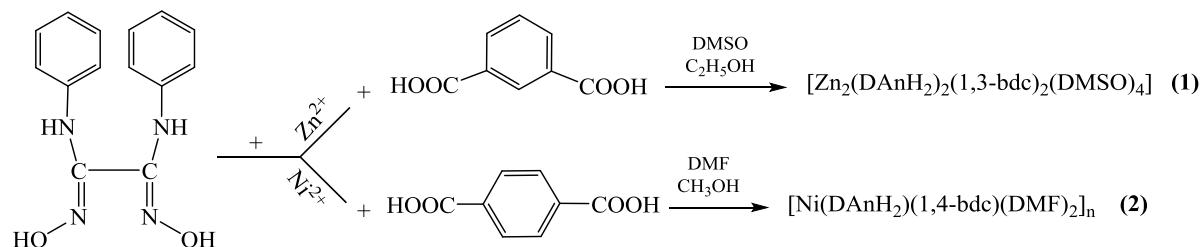
Synthesis of [Ni(DAnH₂)(1,4-bdc)(DMF)₂]_n (**2**)

Both dianilinoglyoxime (0.054 g, 0.2 mmol) and nickel acetate tetrahydrate (0.025 g, 0.1 mmol) were dissolved in a mixture of DMF-MeOH (6 mL) in a ratio of 1:1 (v/v), then a solution of 1,4-H₂bdc (0.034 g, 0.2 mmol) in MeOH (3 mL) was added to the obtained red-black mixture. Gradually, the color of the reaction mixture turned into red-green, after which the mixture was stirred for another 10–15 minutes, filtered, and the filtrate was left to crystallize at room temperature. After three weeks, the green hexagonal prism-shaped crystals soluble in DMSO, less soluble in methanol, and insoluble in water, ethanol, and diethyl ether were formed. Yield: 0.027 g, 42%. Calc., %: C, 52.61; H, 5.05; N, 13.15; Ni, 9.18 for C₂₈H₃₂N₆NiO₈. Found, %: C, 52.48; H, 5.12; N, 13.20; Ni, 9.17. IR spectrum (ν , cm⁻¹): 3316 m, 3045 vw, 2939 vw, 2331 w, 1831 w, 1646 s, 1598 m, 1546 m, 1499 m, 1470 w, 1436 w, 1419 w, 1376 vs, 1315 vw, 1295 m, 1253 w, 1164 vw, 1153 w, 1137 vw, 1105 m, 1077 w, 1063 vw, 1034 vw, 1028 w, 1013 w, 1001 w, 982 vw, 975 vw, 930 vw, 916 m, 886 m, 870 vw, 850 vw, 838 vw, 831 vw, 813 m, 806 m, 753 s, 716 m, 691 s, 678 vw, 660 vw, 628 m, 606 w, 541 s, 508 vw, 481 w.

Physical measurements

Elemental analysis of the synthesized complexes for C, H, and N was performed on a Vario EL (III) Elemental Analyzer, and for metals - on an AAS-1N atomic absorption spectrometer (Carl Zeiss).

Infrared spectra (IR) were recorded on a FT-IR Spectrum-100 Perkin Elmer spectrometer in Nujol (4000–400 cm⁻¹) using the attenuated total reflection (ATR) technique (4000–650 cm⁻¹). Intensities are given as: vs-very strong, s-strong, m-medium, w-weak, and vw-very weak.



Scheme 1. Synthetic pathways to complexes **1 and **2**.**

¹H, and ¹³C NMR spectra were recorded in DMSO-d₆ (99.95 %) on a Bruker Advance DRX 400 (400.13, 100.61 and 40.54 MHz). Chemical shifts (δ) are reported in ppm and are referenced to the residual nondeuterated solvent peak (2.50 ppm for ¹H and 39.50 ppm for ¹³C).

Crystallographic studies

Diffraction data for **1** and **2** were collected on an Xcalibur E diffractometer with a charge-coupled device (CCD) using the graphite-monochromatized MoK α radiation at 160 and 293 K, respectively. The determination of the unit cell parameters and processing of the experimental data were performed using a CrysAlis Oxford Diffraction Ltd. as in [49]. The structures were solved by direct methods and refined by the full-matrix least-squares on weighted F^2 values for all reflections using a SHELXL 2014 software [50]. All non-H atoms in the compounds were refined with anisotropic displacement parameters. The positions of hydrogen atoms in the structures were located on difference Fourier maps or calculated geometrically and refined isotropically in the “rigid body” model with $U=1.2$ Ueq or 1.5 Ueq of the corresponding O, N, and C atoms. The disordering problems for DMSO ligands were resolved for compound **1**: of the eight DMSO ligands belonging to two crystallographically independent complexes, six were located in two positions. The details of the structure solution and refinement for compounds **1** and **2** are given in Table 1. Selected bond distances and bond angles are given in Table 2. Geometric parameters of hydrogen bonds are listed in Table 3. Both CCDC 2144144 and 2144143 contain the supplementary crystallographic data for this paper. Those data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

Results and discussion

The IR spectrum of compound **1** shows the absorption maxima at 3662 and 3304 cm⁻¹, which may be attributed to the $\nu(\text{OH})$ and $\nu(\text{NH})$ groups [51]. The very large absorption bands of a low intensity at 2600–1800 cm⁻¹ are characteristic to the $\nu(\text{OH})$ vibration of the oxime groups participating in the intramolecular hydrogen bond formation with carboxylic anions. The absorption maxima from 1667 and 1623 cm⁻¹ may be attributed to the $\nu(\text{C=O})$ vibration of dicarboxylic ligands and to the $\nu(\text{C=N})$ oxime fragment,

respectively [52,53]. The peaks at 1600 and 1497 cm⁻¹ of $\nu(\text{C=C})$ belong to aromatic rings, as well as the peaks of the non-planar deformation of $\delta(\text{CH})$ in a range of 807–694 cm⁻¹. In the IR spectra, also, a band of a high intensity at 1009 cm⁻¹ is vivid, which belongs to the $\nu(\text{S=O})$ vibrations of DMSO molecules [52,54].

The IR spectrum of compound **2** has many similarities with that of compound **1**. The absorption bands of $\nu(\text{OH})$ and $\nu(\text{NH})$ groups are localized at 3316 and 3045 cm⁻¹, respectively [51]. Also, there is a large signal with a low intensity at 1831 cm⁻¹, which probably belongs to $\nu(\text{OH})$ vibration of the oxime groups, and, similarly to the case of compound **1**; it participates in the formation of intramolecular hydrogen bonds with carboxylic anions. The maximum of absorption of a high intensity at 1646 cm⁻¹ belongs to the $\nu(\text{C=N})$ and $\nu(\text{C=O})$ vibrations of coordinated DMF molecules [51–53]. For benzene rings, the $\nu(\text{C=C})$ vibrations at 1546 and 1499 cm⁻¹, respectively, have been identified as well as those of non-planar deformation $\delta(\text{CH})$ in a range of 753–691 cm⁻¹.

The ¹H NMR spectrum of compound **1** confirms its structure by signals in a weak field at 6.76–7.09 ppm, which reveals the presence of the aromatic protons from the molecule of dianilineglyoxime. The presence of carboxylic dianions is proved by the signal in a range of 7.45–8.24 ppm and the protons from the iminic and the oxime groups are visible at 8.62 ppm and 10.32 ppm, respectively. In the ¹³C NMR spectrum, there are signals of both aromatic rings. Those of unsubstituted carbon atoms from dianilineglyoxime are well seen in a range of 118.18–121.99 ppm, that of tetrasubstituted - at 139.99 ppm, and of carboxylates - from 128.11 ppm to 135.07 ppm. The signal at 171.78 ppm is attributed to carbon atoms from carboxylic groups and that at 143.43 ppm - to the tetrasubstituted carbon from the oxime group.

The binuclear complex (**1**) crystallizes in the triclinic *P*-1 space group (Table 1). The asymmetric part of the unit cell contains four crystallographically independent Zn(II) ions, four neutral DANH₂ ligands, four bideprotonated carboxylate anions (1,3-bdc)²⁻, and eight DMSO ligands. The crystal contains two crystallographically independent binuclear molecular complexes (denoted A and B); in each of them, two metal atoms are linked together by two bidentate carboxylate bridging ligands (Figure 1(a)). At each of the metal atoms, a bidentate-chelate DANH₂ ligand coordinates, implying two nitrogen atoms, and the

coordination polyhedron of a zinc atom is completed by two monodentate DMSO ligands that are coordinated by oxygen atoms. Thus, the Zn(II) ions have octahedral coordination geometries provided by N₂O₄ sets of donor atoms. The interatomic Zn-N_(oxime) distances in the equatorial plane of the coordination polyhedron of the metal are in a range of 2.130(4)–2.366(4) Å, Zn-O_(bdc) - 2.025(3)–2.093(4) Å, and axial Zn-O_(DMSO) distances - 2.065(4)–2.187(4) Å (Table 2). These are in agreement with the values for Zn(II) complex with dioximes [7]. Each binuclear complex in compound **1** is stabilized by four intramolecular hydrogen bonds between two DAnH₂ ligands and (1,3-bdc)²⁻ residues, the distances donor-acceptor O···O vary in a range of 2.475(5)–2.568(5) Å

(Table 3). The survey of the Cambridge Structural Database [55] identified a binuclear Zn(II)-dioxime complex with the 4,4'-bipyridine as a bridging ligand [56], and the mixed-ligand Zn(II) compounds with dioximes and carboxylates ligands, with mononuclear [57] and polymeric [58] structures.

The binuclear complexes in the crystal of compound **1** are linked in the layers parallel to the *ac* crystallographic plane by intermolecular N-H···O hydrogen bonds, where =NH functional groups act as proton donors and oxygen atoms of oxime ligands as acceptors. The layers are additionally stabilized by weaker hydrogen bonds of C-H···O type, with donor-acceptor distances being in the range of 3.14(2)–3.60(5) Å (Figure 1(*b*)).

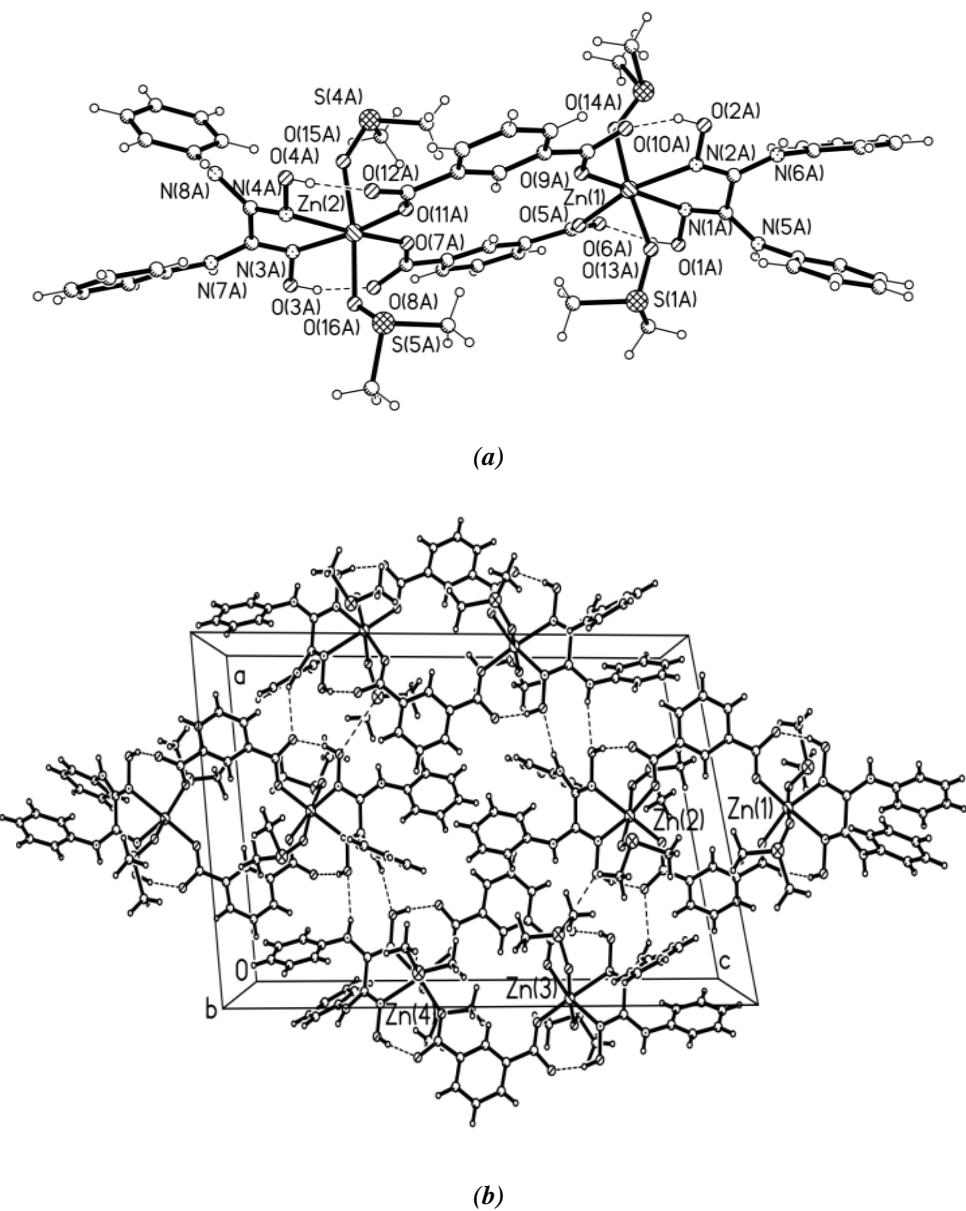


Figure 1. Molecular structure of: binuclear Zn(II) complex **1 (*a*) and fragment of the H-bonded layer (*b*).**

The coordination compound **2** crystallizes in the monoclinic $P2_1/n$ space group (Table 1), and it is a unidimensional (1D) coordination polymer. The asymmetric part of the unit cell contains Ni(II) metal ion, a DAnH₂ neutral ligand, a (1,4-bdc)²⁻ bideprotonated ligand, and two DMF neutral ligands (Figure 2(a)). Each Ni(II) ion in this polymeric compound (Figure 2(b)) is hexacoordinated and has a distorted octahedral geometry with the N₂O₄ set of donor atoms, two nitrogen atoms belonging to the bidentate-chelate coordinated DAnH₂ neutral ligand, two oxygen atoms from two (1,4-bdc)²⁻ bideprotonated ligands, and two oxygen atoms from two DMF ligands that occupy axial coordinates. The carboxylate ligand is a bridging ligand which coordinates *exo*-bidentate *via* two oxygen atoms from terminal carboxyl groups to two metal atoms. Because of this, one of the positions from the

coordination polyhedron of the metal is occupied by the oxygen atom from the carboxyl group of the neighboring fragment [Ni(DAnH₂)(1,4-bdc)(DMF)₂]. The interatomic Ni–N_(oxime) distances are equal to 2.102(2) and 2.082(2) Å, Ni–O_(bdc) – 2.013(2) and 2.040(2) Å, Ni–O_(DMF) – 2.081(2) and 2.083(2) Å (Table 2).

The 1D coordination polymer **2** in the crystal is stabilized in a zig-zag form with Ni-Ni separation across the (1,4-bdc)²⁻ bridging ligand equal to 11.086 Å, the polymeric step being of 14.011 Å.

In the crystal of compound **2**, a system of intra- and intermolecular hydrogen bonds was registered. The intramolecular hydrogen bonds O_(oxime)–H···O_(carb.) with donor-acceptor distances 2.538(3) and 2.536(3) Å (Table 3) form two seven-membered pseudo-chelate rings which stabilize an extended Ni(II) equatorial platform in the polymeric complex.

Table 1

Crystal data and details of data collection for (**1**) and (**2**).

Parameter	Value	
	1	2
Empirical formula	C ₅₂ H ₆₀ N ₈ O ₁₃ S ₄ Zn ₂	C ₂₈ H ₃₂ NiN ₆ O ₈
<i>M</i>	1312.06	639.30
Temperature, K	160(2)	293(2)
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> -1	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> , Å	16.7185(3)	14.6306(5)
<i>b</i> , Å	16.7218(4)	14.0111(5)
<i>c</i> , Å	24.8930(4)	15.9393(6)
α , °	94.7468(15)	90
β , °	96.1010(14)	109.175(4)
γ , °	119.152(2)	90
<i>V</i> , Å ³	5970.3(2)	3086.1(2)
<i>Z</i>	4	4
$\rho_{\text{calcd.}}$, g/cm ³	1.450	1.376
μ , mm ⁻¹	1.016	0.686
<i>F</i> (000)	2720	1336
Crystal size, mm	0.28×0.18×0.08	0.42×0.32×0.07
2θ range, °	1.52–25.50	2.95–25.05
Index ranges	$-20 \leq h \leq 18$, $-19 \leq k \leq 20$, $-30 \leq l \leq 30$	$-17 \leq h \leq 17$, $-10 \leq k \leq 16$, $-18 \leq l \leq 18$
Reflections collected/independent reflections (<i>R</i> _{int})	55923/21896(0.0513)	11361/5439(0.0397)
Reflections with <i>I</i> > 2σ(<i>I</i>)	13893	3927
Completeness, %	98.6 ($\theta = 25.24^\circ$)	99.5 ($\theta = 25.05^\circ$)
Parameters	1675	394
GOOF	1.006	1.003
Final <i>R</i> indices (<i>I</i> > 2σ(<i>I</i>))	<i>R</i> ₁ = 0.0632 <i>wR</i> ₂ = 0.1350	<i>R</i> ₁ = 0.0489 <i>wR</i> ₂ = 0.1050
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.1120 <i>wR</i> ₂ = 0.1536	<i>R</i> ₁ = 0.0757 <i>wR</i> ₂ = 0.1202
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$, e Å ⁻³	1.821–1.298	0.354–0.290

Table 2

Selected bond lengths (Å) and bond angles (°) in 1 and 2.

Structure of 1			
Bond	Bond length, Å	Bond	Bond length, Å
Zn(1)-N(1A)	2.309(4)	Zn(3)-N(1B)	2.366(4)
Zn(1)-N(2A)	2.128(4)	Zn(3)-N(2B)	2.139(4)
Zn(1)-O(5A)	2.023(3)	Zn(3)-O(5B)	2.050(3)
Zn(1)-O(9A)	2.053(3)	Zn(3)-O(9B)	2.030(3)
Zn(1)-O(13A)	2.188(4)	Zn(3)-O(13B)	2.145(3)
Zn(1)-O(14A)	2.074(4)	Zn(3)-O(14B)	2.039(4)
Zn(2)-N(3A)	2.167(4)	Zn(4)-N(3B)	2.166(4)
Zn(2)-N(4A)	2.290(4)	Zn(4)-N(4B)	2.299(4)
Zn(2)-O(7A)	2.047(3)	Zn(4)-O(7B)	2.041(3)
Zn(2)-O(11A)	2.035(3)	Zn(4)-O(11B)	2.091(3)
Zn(2)-O(15A)	2.131(4)	Zn(4)-O(15B)	2.132(8)
Zn(2)-O(16A)	2.068(4)	Zn(4)-O(16B)	2.061(3)
Bond angle	Bond angle value, °	Bond angle	Bond angle value, °
N(1A)-Zn(1)-N(2A)	71.4(1)	N(1B)-Zn(3)-N(2B)	71.4(1)
N(1A)-Zn(1)-O(5A)	93.3(3)	N(1B)-Zn(3)-O(5B)	96.3(1)
N(1A)-Zn(1)-O(9A)	174.2(1)	N(1B)-Zn(3)-O(9B)	170.8(1)
N(1A)-Zn(1)-O(13A)	90.4(1)	N(1B)-Zn(3)-O(13B)	82.7(2)
N(1A)-Zn(1)-O(14A)	91.4(2)	N(1B)-Zn(3)-O(14B)	94.4(2)
N(2A)-Zn(1)-O(5A)	163.6(1)	N(2B)-Zn(3)-O(5B)	167.4(1)
N(2A)-Zn(1)-O(9A)	104.3(1)	N(2B)-Zn(3)-O(9B)	102.0(1)
N(2A)-Zn(1)-O(13A)	79.1(1)	N(2B)-Zn(3)-O(13B)	87.0(1)
N(2A)-Zn(1)-O(14A)	95.0(2)	N(2B)-Zn(3)-O(14B)	88.9(2)
O(5A)-Zn(1)-O(9A)	90.6(1)	O(5B)-Zn(3)-O(9B)	90.0(1)
O(5A)-Zn(1)-O(13A)	95.7(1)	O(5B)-Zn(3)-O(13B)	88.8(1)
O(5A)-Zn(1)-O(14A)	91.1(2)	O(5B)-Zn(3)-O(14B)	94.9(2)
O(9A)-Zn(1)-O(13A)	84.9(1)	O(9B)-Zn(3)-O(13B)	90.7(1)
O(9A)-Zn(1)-O(14A)	92.9(2)	O(9B)-Zn(3)-O(14B)	91.8(2)
O(13A)-Zn(1)-O(14A)	172.9(2)	O(13B)-Zn(3)-O(14B)	175.6(2)
N(3A)-Zn(2)-N(4A)	72.7(1)	N(3B)-Zn(4)-N(4B)	71.6(1)
N(3A)-Zn(2)-O(7A)	101.6(1)	N(3B)-Zn(4)-O(7B)	102.5(1)
N(3A)-Zn(2)-O(11A)	170.7(1)	N(3B)-Zn(4)-O(11B)	169.2(1)
N(3A)-Zn(2)-O(15A)	83.3(2)	N(3B)-Zn(4)-O(15B)	80.1(2)
N(3A)-Zn(2)-O(16A)	87.5(1)	N(3B)-Zn(4)-O(16B)	90.1(1)
N(4A)-Zn(2)-O(7A)	171.6(2)	N(4B)-Zn(4)-O(7B)	173.7(1)
N(4A)-Zn(2)-O(11A)	98.2(1)	N(4B)-Zn(4)-O(11B)	97.6(1)
N(4A)-Zn(2)-O(15A)	82.0(2)	N(4B)-Zn(4)-O(15B)	93.6(3)
N(4A)-Zn(2)-O(16A)	91.9(1)	N(4B)-Zn(4)-O(16B)	90.4(2)
O(7A)-Zn(2)-O(11A)	87.3(1)	O(7B)-Zn(4)-O(11B)	88.3(1)
O(7A)-Zn(2)-O(15A)	91.6(2)	O(7B)-Zn(4)-O(15B)	83.1(3)
O(7A)-Zn(2)-O(16A)	94.3(2)	O(7B)-Zn(4)-O(16B)	91.8(2)
O(11A)-Zn(2)-O(15A)	90.8(2)	O(11B)-Zn(4)-O(15B)	100.9(2)
O(11A)-Zn(2)-O(16A)	94.6(1)	O(11B)-Zn(4)-O(16B)	90.1(1)
O(15A)-Zn(2)-O(16A)	172.3(2)	O(15B)-Zn(4)-O(16B)	167.7(2)
Structure of 2			
Bond	Bond length, Å	Bond	Bond length, Å
Ni-N(1)	2.102(2)	Ni-O(6) ^{#1}	2.040(2)
Ni-N(2)	2.082(2)	Ni-O(8)	2.081(2)
Ni-O(3)	2.013(2)	Ni-O(7)	2.083(2)
Bond angle	Bond angle value, °	Bond angle	Bond angle value, °
N(1)-Ni-N(2)	76.58(10)	N(2)-Ni-O(8)	91.75(10)
N(1)-Ni-O(3)	176.26(10)	O(3)-Ni-O(6) ^{#1}	84.49(9)
N(1)-Ni-O(6) ^{#1}	98.68(10)	O(3)-Ni-O(7)	89.27(10)
N(1)-Ni-O(7)	88.75(10)	O(3)-Ni-O(8)	90.82(10)
N(1)-Ni-O(8)	91.34(10)	O(6) ^{#1} -Ni-O(7)	89.75(9)
N(2)-Ni-O(3)	100.29(10)	O(6) ^{#1} -Ni-O(8)	87.14(10)
N(2)-Ni-O(6) ^{#1}	175.11(9)	O(7)-Ni-O(8)	176.86(10)
N(2)-Ni-O(7)	91.32(10)		

^{#1} -x+I/2, y+I/2, -z+I/2 (2).

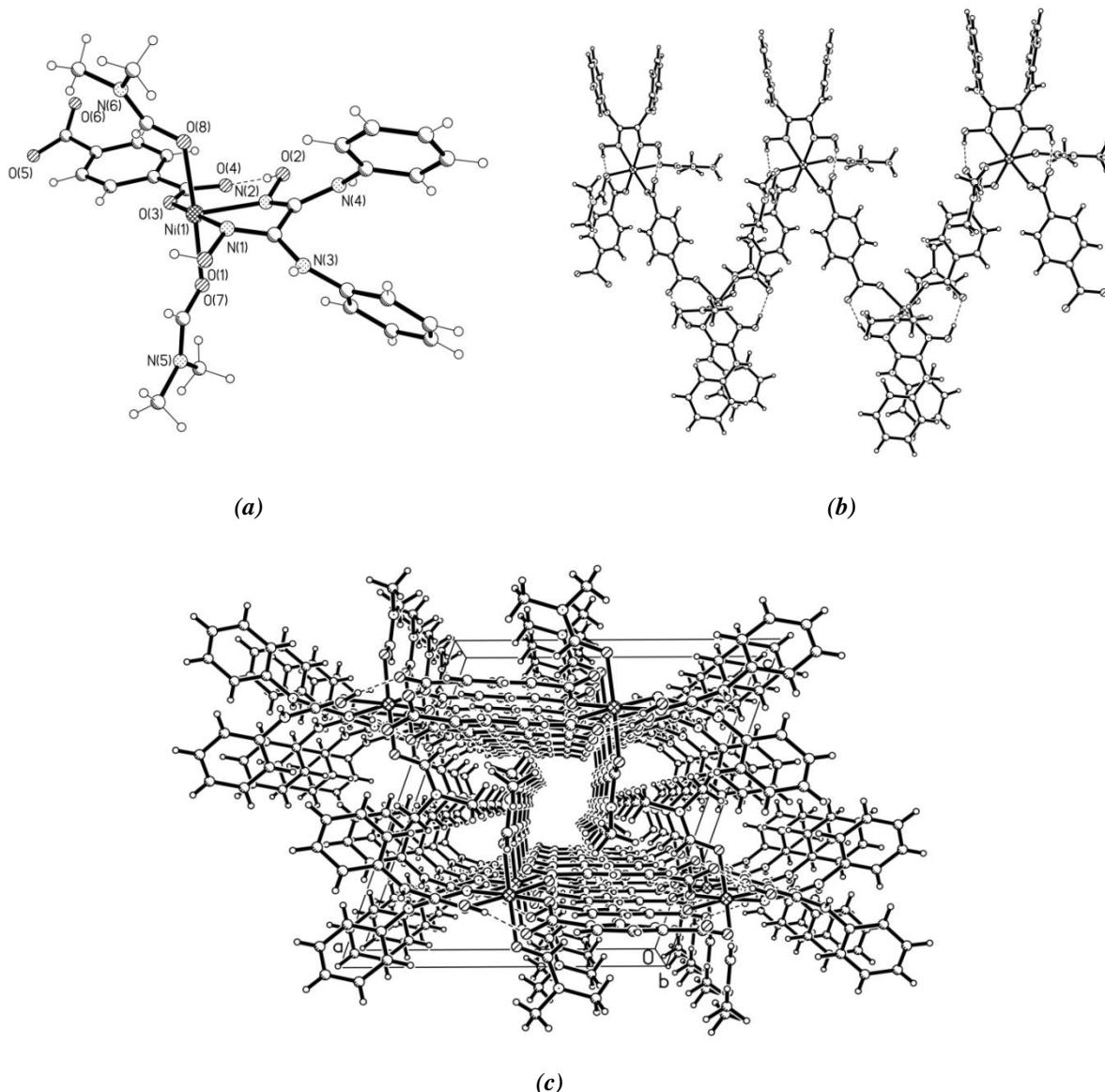


Figure 2. The asymmetric part of coordination polymer 2 with partial numbering of atoms (a); fragment of 1D coordination polymer (b); a perspective view of crystal packing (c).

Table 3

Geometric parameters of intra- and intermolecular hydrogen bonds in the structures of 1 and 2.

$D-H\cdots A$	$D-H, \text{\AA}$	$H\cdots A, \text{\AA}$	$D\cdots A, \text{\AA}$	DHA, °
<i>Structure of 1</i>				
N(5A)-H(5A)…O(2B) ^{#1}	0.88	2.10	2.965(5)	167
N(8A)-H(8A)…O(3B) ^{#3}	0.88	2.01	2.846(5)	158
N(5B)-H(5B)…O(8A) ^{#4}	0.88	2.23	2.901(5)	132
N(6B)-H(6B)…O(1A) ^{#1}	0.88	2.32	3.057(5)	142
N(7B)-H(7B)…O(4A) ^{#3}	0.88	2.18	3.007(5)	158
N(8B)-H(8B)…O(10A) ^{#5}	0.88	2.30	3.021(5)	139
O(1A)-H(1A)…O(6A)	0.84	1.72	2.525(4)	161
O(2A)-H(2A)…O(10A)	0.84	1.77	2.564(4)	157
O(3A)-H(3A)…O(8A)	0.84	1.71	2.531(4)	165
O(4A)-H(4A)…O(12A)	0.84	1.73	2.536(4)	160
O(1B)-H(1B)…O(6B)	0.84	1.76	2.550(5)	156
O(2B)-H(2B)…O(10B)	0.84	1.69	2.500(4)	162
O(3B)-H(3B)…O(8B)	0.84	1.64	2.470(5)	171
O(4B)-H(4B)…O(12B)	0.84	1.74	2.538(5)	158

Continuation of Table 3

D-H···A	D-H, Å	H···A, Å	D···A, Å	DHA, °
Structure of 2				
O(1)-H(1O)···O(5) ^{#1}	0.82	1.74	2.538(3)	164
O(2)-H(2O)···O(4)	0.82	1.73	2.536(3)	166
N(3)-H(1N)···O(2) ^{#3}	0.86	2.20	3.048(4)	167
N(4)-H(2N)···O(1) ^{#4}	0.86	2.19	3.011(4)	160

^{#1} -x+1, -y+1, -z+1; ^{#2} x, y+1, z; ^{#3} -x+2, -y+1, -z; ^{#4} x+1, y, z+1; ^{#5} x, y-1, z (1);
^{#1} -x+1/2, y+1/2, -z+1/2; ^{#2} -x+1/2, y-1/2, -+1/2; ^{#3} -x+3/2, y+1/2, -z+1/2; ^{#4} -x+3/2, y-1/2, -z+1/2 (2).

Those coordination chains are joined together by intermolecular hydrogen bonds of the N-H···O type, the donor-acceptor distances being equal to 3.048(4) and 3.011(4) Å (Figure 2(c), Table 3), where amino =NH groups are proton donors and oxygen atoms from the oxime groups are acceptors. A detailed analysis of the fine hydrogen bonding system of the C-H···O type shows that DMF ligands do not participate in the formation of intermolecular hydrogen bonds. They generate only intramolecular bonds, stabilizing the polymeric chain; instead, such types of bonds were detected between chains where =CH groups from the aromatic rings of DAnH₂ ligand play the role of proton donors.

Conclusions

The synthesis of the studied here coordination compounds in the simultaneous presence of dianilineglyoxime, 1,3- and 1,4-benzendicarboxylic acids, and DMSO/DMF has led to zinc(II) binuclear and nickel(II) polynuclear coordination compounds.

It was proved that, in the reported compounds, dianilineglyoxime coordinates as a neutral ligand in a bidentate-chelate mode, generating a five-membered metallocycle, and 1,3- and 1,4-benzendicarboxylates act as *exo*-bidentate bridging ligands that bind both metal atoms in di- and polynuclear structures, while molecules of DMSO/DMF act as monodentate terminal ligands.

The structure of the nickel(II) coordination compound is a zig-zag chain, which indicates a 1D coordination polymer.

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“Implementation of crystal engineering approach and X-ray crystallography for design and creation of hybrid organic/inorganic materials with advanced physical and biologically active functions”.

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