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Crystal structure of an oxalate-bridged tetranuclear 8-hydroxyquinoline Zn(II) cluster: $[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$

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Abstract: The chain structure of a tetranuclear zinc(II) cluster $[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$ ($[\text{Zn}_4(\text{C}_9\text{H}_6\text{NO})_6(\text{C}_2\text{O}_4)]_{0.5n}$) (**1**) (Q = 8-hydroxyquinoline anion, Ox = oxalate dianion) was determined by X-ray crystallography and characterized by elemental analysis, IR spectroscopy and thermal analysis. It crystallizes in the monoclinic system, space group $\text{P}2_1/n$ (No. 14), with the lattice parameters $a = 13.2222(15)$ Å, $b = 11.0566(12)$ Å, $c = 16.2224(18)$ Å, $\beta = 92.1770(10)^\circ$, $V = 2369.9(5)$ Å³, $Z = 4$, $M_r = 607.23$ g mol⁻¹, $D_c = 1.702$ g cm⁻³. The tetranuclear zinc(II) clusters form 1D polymeric chains parallel to the b -axis. The π - π stacking interactions involving aryl rings support the formation of the 1D polymeric structure. The neighboring polymeric chains are connected by C–H \cdots π interactions.

Keywords: 8-hydroxyquinoline; zinc(II) cluster; single crystal structure.

INTRODUCTION

The rapid development of supramolecular chemistry and metal–organic coordination polymer research *via* crystal engineering have produced many new polymers with unique, well-defined structures,^{1,2} which are of great interest for potential applications in catalysis,³ adsorption,⁴ ion-exchange,⁵ sensor technology,⁶ optoelectronics,⁷ and nanowires.⁸ These metal–organic network structures depend on the judicious selection of metal centers and organic ligands that may afford the desirable covalent bonds, hydrogen-bonding linkages, or π - π interactions for the construction of new supramolecules or polymers. 8-Hydroxy-

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quinoline is a well-known molecule which due to its metal-complexation ability is frequently used for analysis or metal precipitation.⁹ To date, in the research field of organic luminescent materials, many molecules, such as metal based quinoline derivatives, have shown promising results as strong contenders for future display technology.¹⁰ Extensive work has been performed on tris-(8-hydroxyquinoline) aluminum (AlQ₃) and ZnQ₂ due to their properties, such as excellent flexibility, high photoconductivity and high life time of devices.¹¹ Zinc(II), as a d¹⁰ ion, has been used as the metal center in building coordination polymers in the search for non-linear optical materials.¹² Zinc(II) complexes also show luminescence, which is typically red shifted compared to the corresponding Al(III) compounds.¹³ In supramolecular chemistry, 8-hydroxyquinoline derivatives can be used for the formation of hydrogen bonded networks^{14,15} as well as of supramolecular aggregates.¹⁶ In coordination chemistry, simple 8-hydroxyquinolines coordinated with zinc(II) ions are very versatile and they can form 2:1 and 3:1 complexes.^{16,17} To further research zinc(II) complexes, in the presented study, an oxalate bridged, ternary tetranuclear 8-hydroxyquinoline zinc(II) coordination polymer **1** was obtained.

EXPERIMENTAL

Synthesis

All reagents employed were of analytical grade. Zn(OAc)₂·H₂O (0.235 g, 1 mmol), 8-hydroxyquinoline (0.146 g, 1 mmol) and oxalic acid (0.050 g, 0.5 mmol) in water (17 ml) were mixed with stirring. The pH was adjusted to 8.0, then the mixture was sealed in a 25 ml stainless steel reactor equipped with a Teflon liner and heated at 170 °C for 96 h whereby dark yellow crystals of the title polymer were obtained (yield 27 %, based on Zn). Upon cooling, the C H N contents were determined by elemental analysis: Calcd. for C₂₈H₁₈N₃O₅Zn₂: C, 55.34; H, 2.96, N, 6.92 %. Found: C, 54.91; H, 2.84; N, 6.70 %; IR (KBr, cm⁻¹): 3052w, 1674s, 1642sh, 1640s, 1619s, 1512s, 1455m, 1352w, 1304m, 1103m, 1033m, 807m, 755m.

Crystal structure determination and physical measurements

A yellow block crystal with dimensions of 0.25 mm×0.20 mm×0.17 mm was chosen for X-ray diffraction analysis. Crystal structure measurement for polymer **1** was performed on a Bruker Smart Apex II CCD diffractometer using the ω scan technique with MoK α radiation ($\lambda = 0.71073$ Å) at room temperature (293(2) K). Absorption corrections were applied using the multi-scan technique¹⁸ (the crystal data and refinement relative parameters are listed in Table I). A total of 19891 reflections were obtained in the range of $2.20 \leq \theta \leq 26.00^\circ$, of which 4654 were independent ($R_{\text{int}} = 0.0831$) and 2777 observed reflections with $I > 2\sigma(I)$ were employed for the structure determination and refinement. The structure was solved by the direct method and refined with full-matrix least-squares techniques using the SHELXTL program¹⁹ within WINGX.²⁰ All non-hydrogen atoms were refined anisotropically with the final $R_1 = 0.0943$, $\omega R_2 = 0.0939$ ($\omega = 1/[\sigma^2(F_o^2) + (0.0325P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$, $(\Delta/\sigma)_{\text{max}} = 0.011$, $F(000) = 1228$, $\mu = 2.072$ mm⁻¹, $S = 0.989$, $(\Delta\rho)_{\text{max}} = 0.691$ e Å⁻³ and $(\Delta\rho)_{\text{min}} = -0.423$ e Å⁻³. The hydrogen atoms on the carbon atoms were generated geometrically.

The FT-IR spectrum was recorded from KBr pellets in range 4000–400 cm^{-1} on a Perkin-Elmer 240C spectrometer. Thermogravimetric analysis was performed using a Perkin-Elmer TG-7 analyzer in nitrogen.

TABLE I. Crystal data and refinement relative parameters

Crystal data	
Formula: $\text{C}_{28}\text{H}_{18}\text{N}_3\text{O}_5\text{Zn}_2$	$V = 2369.9 (5) \text{ \AA}^3$
$M_r = 607.19$	$Z = 4$
$a = 13.2222 (15) \text{ \AA}$	$F(000) = 1228$
$b = 11.0566 (12) \text{ \AA}$	$D_x = 1.702 \text{ Mg m}^{-3}$
$c = 16.2224 (18) \text{ \AA}$	Mo K_α radiation, $\lambda = 0.71073 \text{ \AA}$
$\alpha = 90^\circ$	$\mu = 2.07 \text{ mm}^{-1}$
$\beta = 92.177 (1)^\circ$	$T = 298 \text{ K}$
$\gamma = 90^\circ$	0.25 mm×0.20 mm×0.17 mm
Data collection	
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.083$
Radiation monochromator: graphite	$\theta_{\text{max}} = 26.0^\circ, \theta_{\text{min}} = 2.2^\circ$
Measured reflections: 19891	$h = -16 \rightarrow 16$
Independent reflections: 4654	$k = -13 \rightarrow 13$
Reflections: 2777 (with $I > 2\sigma(I)$)	$l = -19 \rightarrow 20$
Refinement	
Refinement on: F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: Full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighboring sites
$wR(F^2) = 0.094$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.99$	$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$
Reflections: 4654	$(\Delta/\sigma)_{\text{max}} < 0.001$
Parameters: 343	$\Delta\rho_{\text{max}} = 0.69 \text{ e \AA}^{-3}$
Restraints: 0	$\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$

RESULTS AND DISCUSSION

IR spectra

In the IR spectrum, polymer **1** exhibited absorption at 1103 cm^{-1} (*m*), corresponding to the presence of quinoline C–O bonds. The C–H stretching mode for the phenyl ring was relatively weak and was observed at about 3052 cm^{-1} . The peaks at 1640 cm^{-1} (*s*) are attributed to $\nu_{\text{C}=\text{N}}$ stretching,²¹ and the peak at 1455 cm^{-1} is assigned to $\nu_{\text{s}}(-\text{C}=\text{N}-\text{C}=\text{C}-)$.²² The peaks at 1619 (*s*) and 1512 cm^{-1} (*s*) are assigned to C=C stretching vibrations of the aryl rings and the peak at 755 cm^{-1} is attributed to the $\nu_{\text{C}-\text{H}}$ of aryl rings. The strong carboxylic acid peak at 1726 cm^{-1} (*s*) diminished and two peaks developed at 1674 (*s*) and 1642 cm^{-1} (*sh*)

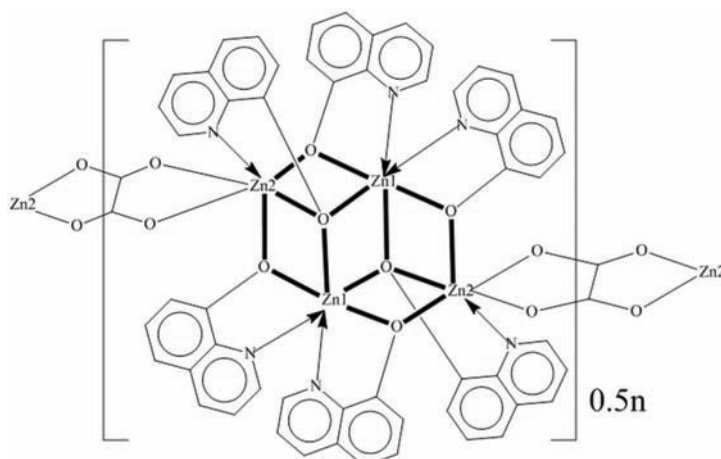
assigned to the asymmetric stretching of carboxyl (CO_2) and another two peaks at 1352 cm^{-1} (*w*) and 1304 cm^{-1} (*m*) can be regarded to the symmetric stretching of carboxyl (CO_2), while the peak at 1033 cm^{-1} (*m*) can be considered as the stretching of carboxyl (CO). The peak at 807 cm^{-1} (*m*) can be assigned to the stretching of the δ band of carboxyl CO_2 .

Thermal analysis

The TG curve of the title polymer $[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$ showed two weight loss steps peaking at 375 and 446 °C. Based on the weight changes, the first weight loss process may be related to the loss of one Ox (found 6.4 %; calcd. 7.2 %); the second weight loss event corresponds to the loss of six Qs (found 69.3 %; calcd. 71.1 %). After 560 °C, basically no loss weight occurred while the residual weight of 24.3 % suggests that the residue may be ZnO (calcd. 26.8 %).

Crystal structure of $[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$ (**1**)

The structure of the title polymer (**1**) ($[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$) is illustrated in Scheme 1 and Fig. 1.



Scheme 1. The structure of the title tetranuclear coordination polymer **1** ($[\text{Zn}_4\text{Q}_6(\text{Ox})]_{0.5n}$).

The crystal structure consists of tetranuclear dimeric clusters. The centre of inversion is located at the middle of the tetranuclear cluster. There are two crystallographically independent zinc center sites (Zn1 and Zn2) within half of one cluster. The two zinc centers have the same coordination geometry (each zinc center is coordinated by six atoms and is located at the centre of an octahedron). Four 8-hydroxyquinoline anions (Qs) are involved in coordination with Zn1. The coordination is achieved by four O and two N atoms. The N2, O1, O2 and O3 atoms are located in an equatorial plane, while $\text{O}3^i$ (*i* stands for the sym-

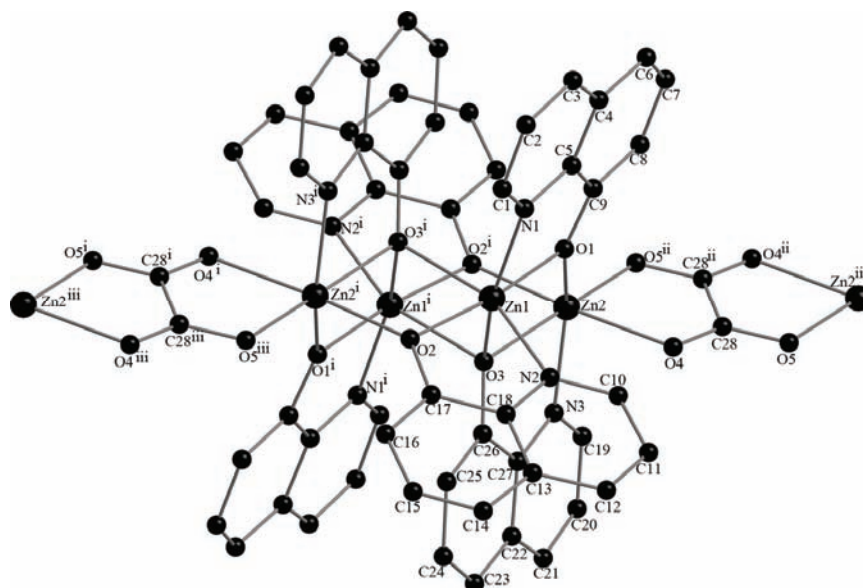


Fig. 1. Coordination geometry of the tetranuclear coordination polymer **1**, hydrogen atoms are omitted for clarity. Symmetry codes for i: $2-x, -y, -z$, ii: $2-x, 1-y, -z$ and iii: $x, -1+y, z$.

metry operation $2-x, -y, -z$) and N1 occupy the axial sites. Three Qs and one Ox are involved in coordination with Zn2. Zn2 is coordinated with five O atoms (two from oxalate and three from 8-hydroxyquinoline anions) and one N atom. The $O2^i$, O3, O4 and $O5^{ii}$ (*ii* stands for the symmetry operation $2-x, 1-y, -z$) are located at the equatorial positions, while O1 and N3 occupy the axial sites. The phenolic oxygen atoms O1 and O2 are two-center μ_2 -type oxygen atoms; O1 connects Zn1 and Zn2 and O2 connects Zn1 and Zn2^{*i*}. The phenolic oxygen O3 and its symmetry equivalent $O3^i$ are two three-center μ_3 -type oxygen atoms; O3 connects Zn1, Zn2 and Zn1^{*i*} and $O3^i$ connects the Zn1^{*i*}, Zn2^{*i*} and Zn1 atoms. The Zn–N distances range between 2.086 and 2.139 Å, which is consistent with reported Zn–N distances, from 2.003²³ to 2.144.²⁴ The Zn–O_{phenolic} distances range from 2.054 to 2.219 Å (Table II), and are similar to those reported in the literature (from 2.063–2.220 Å).^{24,25} The $O4_{Ox}$ and $O5^{ii}_{Ox}$ are two doubly bridging O atoms coordinated to the Zn2 center; the Zn2–O_{Ox} distances are 2.211(3) ($O4_{Ox}$ –Zn2), and 2.057(3) Å ($O5^{ii}$ –Zn2). The average Zn–O distance is 2.120(5) Å, which is slightly longer than the normal Zn–O distance 2.070 Å.²⁵ The dihedral angles between the Q rings are 18.88° (ring containing N1 and ring containing N3), 64.62° (ring containing N2 and ring containing N3) and 81.49° (ring containing N1 and ring containing N2). The Zn–Zn distances are 3.324 (Zn1 \cdots Zn2), 3.278 (Zn1^{*i*} \cdots Zn2), and 3.264 Å (Zn1^{*i*} \cdots Zn1). These three distances are consistent with the Zn \cdots Zn distances of the related multinuclear 8-hyd-

roxyquinoline Zn(II) clusters (from 3.232 to 3.303 Å (tetranuclear^{26a,26b}); 3.397 Å (trinuclear^{26c}); 3.240 and 3.496 Å (binuclear^{26d,26e})). These distances are longer than the sum of covalent radii of two Zn (2.44 Å, 1.22 Å×2),²⁷ but clearly shorter than the sum of the van der Waals radii of two Zn (4.20 Å, 2.10 Å×2).²⁸ These data reveal that there are the metal–metal interactions in the tetranuclear Zn centers in each cluster of the polymer **1**. The oxalate dianion exhibits the bis-bidentate coordination mode bridging two Zn2 atoms, *i.e.*, it chelates two symmetry-related Zn2 atoms. The Ox ligand acts as a spacer between adjacent two tetranuclear clusters, thus linking the clusters into a one-dimensional chain (Fig. 2). In the crystal lattice, the centroid-to-centroid distances to about 4 Å between the aryl-rings and their equivalent symmetry molecules at $2-x$, $-y$, $-z$ are listed in Table II. The data given in Table II suggest the existence of intra-chain offset π -stacking interactions and inter-chains point-to-face (T-shaped) C–H $\cdots\pi$ interactions. Moreover, in polymer **1** there are intra-chain, non-classic hydrogen bonds^{29,30} C(16)–H(16) \cdots O(5)^c (symmetry code *c*: x , $-1+y$, z) (Table II). It is obvious that in solid state, all these molecular interactions contribute to the stabilization of the supramolecular structure **1**.^{31–34}

TABLE II. Selected bond lengths, Å, and bond angles, ° (symmetry transformation: #1: $-x+2v, -y+1, v-z$; #2: $-x+2, -y, -z$)

Bond	Length	Bond	Length
N(1)–Zn(1)	2.125(3)	O(3)–Zn(1)	2.120(3)
N(2)–Zn(1)	2.086(3)	O(3)–Zn(1)#2	2.216(2)
N(3)–Zn(2)	2.139(3)	O(3)–Zn(2)	2.219(3)
O(1)–Zn(1)	2.059(3)	Zn(2)–O(2)#2	2.086(3)
O(1)–Zn(2)	2.060(3)	O(4)–Zn(2)	2.211(3)
O(2)–Zn(1)	2.054(3)	O(5)–Zn(2)#1	2.057(3)
Bonds	Angle	Bonds	Angle
O(2)–Zn(1)–O(1)	172.80(11)	O(3)–Zn(1)–O(3)#2	82.36(10)
O(2)–Zn(1)–N(2)	80.54(13)	N(1)–Zn(1)–O(3)#2	94.40(11)
O(1)–Zn(1)–N(2)	106.65(13)	O(5)#1–Zn(2)–O(1)	111.89(11)
O(2)–Zn(1)–O(3)	102.51(10)	O(1)–Zn(2)–O(3)	75.05(10)
O(1)–Zn(1)–O(3)	77.26(10)	O(2)#2–Zn(2)–O(3)	79.51(10)
N(2)–Zn(1)–O(3)	93.60(12)	O(5)#1–Zn(2)–O(2)#2	96.04(10)
O(2)–Zn(1)–N(1)	100.44(13)	O(1)–Zn(2)–O(2)#2	96.40(11)
O(1)–Zn(1)–N(1)	79.05(12)	O(5)#1–Zn(2)–N(3)	99.25(12)
N(3)–Zn(2)–O(4)	85.42(12)	O(1)–Zn(2)–N(3)	144.98(12)
O(5)#1–Zn(2)–O(3)	172.30(11)	O(2)#2–Zn(2)–N(3)	95.83(11)
N(2)–Zn(1)–N(1)	97.35(13)	O(5)#1–Zn(2)–O(4)	78.19(11)
O(3)–Zn(1)–N(1)	155.90(12)	O(1)–Zn(2)–O(4)	85.66(11)
O(2)–Zn(1)–O(3)#2	80.28(10)	O(2)#2–Zn(2)–O(4)	174.23(10)
O(1)–Zn(1)–O(3)#2	92.59(10)	N(3)–Zn(2)–O(3)	75.13(11)
N(2)–Zn(1)–O(3)#2	159.02(12)	O(4)–Zn(2)–O(3)	106.25(10)

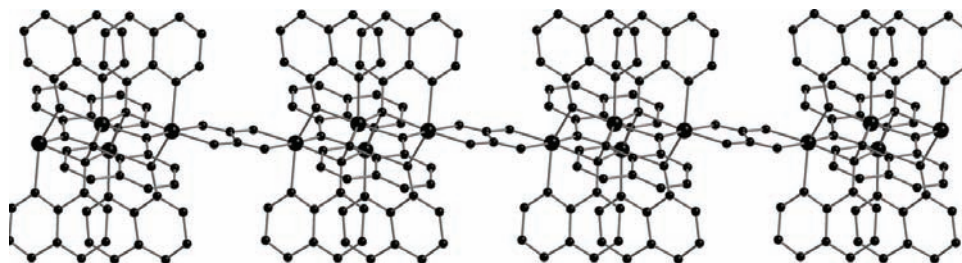


Fig. 2. The 1D infinite chains structure and π - π stacking interactions of polymer **1**, the hydrogen atoms are omitted for clarity.

TABLE III. The relative parameters of the intermolecular π - π interactions (with distinct centroids ≈ 4.0 Å), X-H \cdots Cg (π -ring) interactions ($\text{H}\cdots\text{Cg} < 3.0$ Å, $\gamma < 30.0^\circ$) and non-classic hydrogen bonds in complex **1** (defined ring and symbol explanations: Cg(1): N(1) \rightarrow C(1) \rightarrow C(2) \rightarrow C(3) \rightarrow C(4) \rightarrow C(5) \rightarrow ; Cg(2): N(3) \rightarrow C(19) \rightarrow C(20) \rightarrow C(21) \rightarrow C(22) \rightarrow C(27) \rightarrow ; Cg(3): C(4) \rightarrow C(5) \rightarrow C(9) \rightarrow C(8) \rightarrow C(7) \rightarrow C(6) \rightarrow ; Cg(4): C(22) \rightarrow C(23) \rightarrow C(24) \rightarrow C(25) \rightarrow C(26) \rightarrow C(27) \rightarrow ; Cg(5): C(13) \rightarrow C(14) \rightarrow C(15) \rightarrow C(16) \rightarrow C(17) \rightarrow C(18) \rightarrow ; Cg(I) = plane number I (= ring number in () above); dihedral angle = dihedral angle between planes I and J ($^\circ$); distinct centroids = distance between ring centroids (Å); CgI_Perp = perpendicular distance of Cg(I) on ring J (Å); γ = angle Cg(I) \rightarrow Cg(J) vector and normal to plane J ($^\circ$); symmetry codes: a: $2-x, -y, -z$; b: $3/2-x, -1/2+y, 1/2-z$; c: $x, -1+y, z$)

Cg(I)	Cg(J)	Distinct centroids, Å	Dihedral angle, $^\circ$	CgI_Perp, Å	$\gamma / ^\circ$
Cg(1) \rightarrow Cg(2)		3.999(3)	17.83	3.563	26.99
Cg(1) \rightarrow Cg(4)		3.559(3)	19.93	3.523	8.14
Cg(2) \rightarrow Cg(1)		3.998(3)	17.83	3.331	33.59
Cg(3) \rightarrow Cg(4)		4.032(3)	20.15	3.523	29.11
Cg(4) \rightarrow Cg(1)		3.559(3)	19.93	3.400	17.16
Cg(4) \rightarrow Cg(3)		4.032(3)	20.15	3.422	31.92
X-H(I)	Cg(J)	H \cdots Cg distance, Å	H-Perp, Å	X-H \cdots Cg angle, $^\circ$	$\gamma / ^\circ$
C(6)-H(6) \rightarrow Cg(5)		2.93	2.815	157	16.34
C(15)-H(15) \rightarrow Cg(4)		2.83	2.791	136	9.78
D-H \cdots A		D-H distance, Å	H \cdots A distance, Å	D \cdots A distance, Å	D-H \cdots A angle, $^\circ$
C(16)-H(16) \cdots O(5)		0.93	2.48	3.254(5)	141

CONCLUSIONS

A catenarian oxalate-bridged ternary tetranuclear 8-hydroxyquinoline zinc(II) coordination polymer of slightly distorted octahedral geometric configuration was obtained in the reaction of $\text{Zn}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ with 8-hydroxyquinoline and oxalic acid. The coordination geometry and the intermolecular interactions (π -stacking interactions, C-H \cdots π interactions and non-classic hydrogen bonds) were investigated. Elemental analysis, TG analysis and FT-IR spectroscopy were also employed to characterize the title coordination polymer in detail.

Supplementary data. Supplementary material is deposited in the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, as supplementary material No. CDCC 784785 and can be obtained by contacting the CCDC.

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ИЗВОД

КРИСТАЛНА СТРУКТУРА ТЕТРАНУКЛЕАРНОГ 8-ХИДРОКСИХИНОЛИН $Zn(II)$ КЛАСТЕРНОГ КОМПЛЕКСА СА ОКСАЛАТНИМ АНИЈОНОМ У МОСТУ: $[Zn_4Q_6(Ox)]_{0.5n}$ JIAJUN WANG¹, QIANG WANG², YANJUN SUN³, YUEMEI WANG⁴, GUOSHENG ZHAO¹ и YUNCHENG CUI¹¹Chemistry College of Jilin Normal University; Key Laboratory of Preparation and Application of Environmental Friendly Materials (Jilin Normal University), Ministry of Education, Siping, 136000,²Department of Biological Medicine and Chemical Engineering, Liaoning Institute of Science and Technology, Benxi, 117004, ³Boda College of Jilin Normal University, Siping, 136000, China и⁴Xi'an Modern Chemistry Research Institute, Xi'an, 710065, China

Применом рендгенске структурне анализе одређена је структура тетрануклеарног цинк(II) кластерног комплекса $[Zn_4Q_6(Ox)]_{0.5n}$ ($[Zn_4(C_9H_6NO)_6(C_2O_4)]_{0.5n}$) **1** (Q = 8-хидроксихинолински анијон, Ox = оксалат). За карактеризацију овог комплекса употребљени су елементарна микроанализа, IR спектроскопија и термална анализа. Комплекс је кристалисао у моноклиничном систему, просторна група $P2_1/n$ (No. 14), са параметрима јединичне ћелије: $a = 13.2222(15)$ Å, $b = 11.0566(12)$ Å, $c = 16.2224(18)$ Å, $\beta = 92.1770(10)^\circ$, $V = 2369.9(5)$ Å³, $Z = 4$, $M_r = 607.23$, $D_c = 1.702$ g cm⁻³. Тетрануклеарни цинк(II) кластерни комплекс гради 1D полимерне ланце паралелне са b -осом. Интеракције слагања (π - π) које укључују арилне прстенове доприносе грађењу 1D полимерне структуре. Суседни полимерни ланци су повезани преко $CH \cdots \pi$ интеракција.

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