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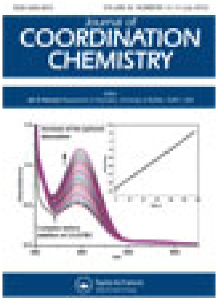

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A tetradentate bisoxime and its supramolecular nickel(II) cluster: synthesis, crystal structure, and spectral properties

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A tetradentate bisoxime and its supramolecular nickel(II) cluster: synthesis, crystal structure, and spectral properties

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Salen-type bisoxime 5,5′-dimethoxy-2,2′-[(ethylenedioxy)bis(nitrilomethylidyne)]diphenol (H_2L) and its trinuclear Ni(II) cluster $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\}\cdot n\text{-}BuOH$ have been synthesized and structurally characterized. The structure of H_2L adopts an L-shape conformation where the two salicylaldoxime moieties are well separated. In the trinuclear Ni(II) cluster, two acetates coordinate to three Ni(II)'s through Ni–O–C–O–Ni bridges, four μ-phenoxos from two [NiL(*n*-BuOH)] units also coordinate to Ni(II), and two *n*-butanols coordinate to two terminal Ni(II)'s forming a distorted octahedral geometry. The Ni–O–C–O–Ni and μ-phenoxo bridges play important roles in assembling Ni(II) and the ligands. H_2L forms a rectangle-like large cave structure through $O\text{–}H\cdots N$, $C\text{–}H\cdots O$, and $C\text{–}H\cdots\pi$ hydrogen-bond interactions, whereas its trinuclear Ni(II) cluster exhibits a 3-D supramolecular network structure through intermolecular $O\text{–}H\cdots O$, $C\text{–}H\cdots O$, and $C\text{–}H\cdots\pi$ hydrogen-bond interactions.

Keywords: Bisoxime ligand; Trinuclear Ni(II) cluster; Synthesis; Crystal structure; Spectral property

1. Introduction

Salen-type compounds and their transition metal complexes have ubiquitous use in a variety of catalytic chemical transformations [1–4], offering both high reactivity and selectivity for epoxidation of olefins, asymmetric ring-opening of epoxides, olefin aziridination, olefin cyclopropanation, and formation of cyclic and linear polycarbonates [5]. These complexes can be used to obtain non-linear optical materials [6–8], models of biological systems [9, 10], interesting magnetic properties [11–13], and building blocks for cyclic supramolecular structures [14]. Thus, new materials can be produced by using these compounds, which are suitable candidates for further chemical modifications [15].

Salen-type bisoxime ligands have been reported by using an *O*-alkyloxime ($\text{–CH=N–O–(CH}_2)_n\text{–O–N=CH–}$) instead of the double Schiff base ($\text{–CH=N–(CH}_2)_n\text{–N=CH–}$)

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group and the oxygen atoms strongly affect the electronic properties of N_2O_2 coordination sphere, leading to different properties and structures of the resulting complexes [16–20]. Although Salen–Ni(II) complexes are known [21–23], there could be new specific applications for such compounds. In this article, we report the synthesis and structural characterization of a new salen-type bisoxime ligand H_2L (H_2L = 5,5′-dimethoxy-2,2′-[(ethylene)dioxybis(nitrilomethylidyne)]diphenol) with an *O*-alkyloxime unit (–CH=N–O–$(CH_2)_n$–O–N=CH–) and its trinuclear Ni(II) cluster, $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\}\cdot n\text{-}BuOH$.

2. Experimental

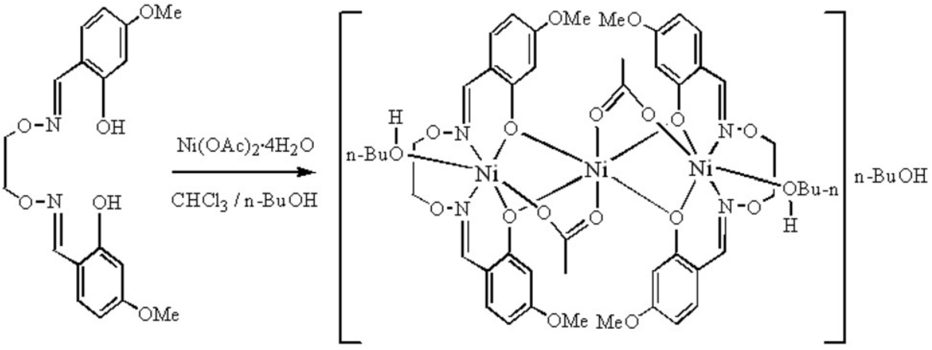
2.1. *Materials and physical measurements*

4-Methoxy-2-hydroxybenzaldehyde (≥98%) was purchased from Alfa Aesar and used without purification. The other reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory. Infrared (IR) spectra were recorded on a VERTEX-70 FT-IR spectrophotometer, with samples prepared as KBr (500–4000 cm^{-1}) or CsI (100–500 cm^{-1}) pellets. ^1H NMR spectra were recorded on a Bruker DRX 400 spectrometer in $CDCl_3$ solution with TMS as an internal standard. X-ray single-crystal structures were determined on a Bruker Smart 1000 CCD area detector. Molar conductance measurements were carried out on a model DDS-11D type conductivity bridge using $1.0\times10^{-3}\,mol\,dm^{-3}$ solution in DMF at 25°C.

2.2. *Preparation of H_2L and its cluster*

2.2.1. Preparation of H_2L. H_2L was synthesized with a slightly modified method reported earlier [24–26]. To an ethanol solution (5 mL) of 4-methoxy-2-hydroxybenzaldehyde (394.6 mg, 2.56 mmol), an ethanol solution (5 mL) of 1,2-bis(aminooxy)ethane (118 mg, 1.28 mmol) was added. The solution was stirred at 55°C for 4 h, cooled to room temperature, filtered, and washed successively with ethanol : hexane (1 : 4) and hexane, respectively. The product was dried under vacuum, giving colorless crystalline solid of 320.69 mg. Yield: 76.3%. m.p. 97–98°C. ^1H NMR (400 MHz, $CDCl_3$): 3.79 (s, 6H), 4.41 (s, 4H), 6.46 (d, $J=2.8$ Hz, 2H), 6.47 (dd, $J=8.4$, 2.6 Hz, 2H), 7.04 (d, $J=8.8$ Hz, 2H), 8.17 (s, 2H), 9.94 (s, 2H). Anal. Calcd for $C_{18}H_{20}N_2O_6$ (%): C, 59.99; H, 5.59; N, 7.77. Found (%): C, 59.95; H, 5.86; N, 7.63. Single-crystals of H_2L suitable for X-ray crystal analysis were grown from a solution of acetone and ethanol by slow evaporation at room temperature.

2.2.2. Preparation of the Ni(II) cluster. A solution of Ni(II) acetate tetrahydrate (4.2 mg, 0.015 mmol) in *n*-butanol (3 mL) was added dropwise to a solution of H_2L (6.20 mg, 0.016 mmol) in chloroform (3 mL) at room temperature. Vapor phase diffusion of *n*-hexane into the solution afforded dark green crystals of the cluster (scheme 1). Anal. Calcd for $C_{52}H_{72}N_4Ni_3O_{19}$ (%): C, 50.64; H, 5.88; N, 4.54; Ni, 14.28. Found (%): C, 50.69; H, 5.95; N, 4.49; Cu, 14.22.



Scheme 1. Synthesis of the cluster.

2.3. *X-ray structure determination of* $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\} \cdot n\text{-}BuOH$

The crystal data and structure refinement for H_2L and $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\} \cdot n\text{-}BuOH$ are given in table 1. Single crystals of H_2L and $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\} \cdot n\text{-}BuOH$ with approximate dimensions of $0.45 \times 0.25 \times 0.16\,mm^3$ and $0.22 \times 0.19 \times 0.18\,mm^3$ were placed on a Bruker Smart 1000 CCD area detector. The diffraction data were collected using graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at 298(2) K. The structures were solved using SHELXL-97 and Fourier difference techniques, and refined by full-matrix least-squares on F^2. All hydrogen atoms were added in the calculated positions.

3. Results and discussion

3.1. *Molar conductance of* $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\} \cdot n\text{-}BuOH$

The cluster is soluble in DMF and DMSO, but not soluble in ethanol, methanol, acetonitrile, acetone, THF, ethyl acetate, and hexane. Molar conductance of the Ni(II) cluster at 25°C of $10^{-3}\,mol\,dm^{-3}$ DMF solution is $10.3\,\Omega^{-1}\,cm^2\,mol^{-1}$, indicating that the Ni(II) cluster is a non-electrolyte. Thus, acetates in the Ni(II) cluster are coordinated in solution.

3.2. *IR spectra of* H_2L *and* $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\} \cdot n\text{-}BuOH$

IR spectra of H_2L and its cluster are given in table 2. H_2L exhibits Ar–O and C=N stretching bands at 1282 and $1633\,cm^{-1}$, respectively, which shift to lower frequencies for the Ni(II) cluster upon complexation. This lowering of energy results from the Ni–O and Ni–N interactions upon complexation, similar to that reported for Ni(II) complexes [27]. IR spectrum of the cluster shows the expected absorption due to the stretching of *n*-BuOH at *ca* $3437\,cm^{-1}$, which is the evidence for *n*-BuOH.

The far-IR spectrum of the cluster was also obtained ($500\text{–}100\,cm^{-1}$) to identify frequencies due to Ni–O and Ni–N bonds. The $\nu_{(Ni\text{–}O)}$ and $\nu_{(Ni\text{–}N)}$ frequencies are at

Table 1. Crystal data and structure refinement for H_2L and $\{[(NiL)(n\text{-BuOH})]_2(\mu\text{-OAc})_2Ni\} \cdot n\text{-BuOH}$.

Empirical formula	$C_{18}H_{20}N_2O_6$	$C_{52}H_{72}N_4Ni_3O_{19}$
Formula weight (gm^{-1})	360.36	1233.27
Temperature (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	*P*-1	*P*-1
Unit cell dimensions (Å, °)		
a	4.7261(6)	11.4203(12)
b	13.7759(15)	11.5630(14)
c	13.9490(16)	13.0626(16)
α	95.690(2)	115.787(2)
β	97.160(4)	99.1980(10)
γ	96.851(4)	106.7850(10)
Volume (Å^3), Z	888.64(18), 2	1403.1(3), 1
Calculated density ($Mg\,m^{-3}$)	1.347	1.460
Absorption coefficient (mm^{-1})	0.102	1.073
$F(000)$	380	648
Crystal size (mm^3)	0.28 × 0.24 × 0.22	0.22 × 0.19 × 0.18
θ range for data collection (°)	1.48–25.00	1.83–25.02
Limiting indices	$-5 \le h \le 5$; $-10 \le k \le 16$; $-16 \le l \le 16$	$-13 \le h \le 13$; $-11 \le k \le 13$; $-15 \le l \le 10$
Reflections collected	4646	7118
Independent reflection	3088 [$R(\text{int}) = 0.0189$]	4862 [$R(\text{int}) = 0.0198$]
Completeness to $\theta = 25.02$ (%)	98.3	98.5
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.9779 and 0.9720	0.8304 and 0.7983
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	3088/0/311	4862/6/404
Goodness-of-fit on F^2	1.018	1.034
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0439$, $wR_2 = 0.0961$	$R_1 = 0.0483$, $wR_2 = 0.1181$
R indices (all data)	$R_1 = 0.1051$, $wR_2 = 0.1277$	$R_1 = 0.0740$, $wR_2 = 0.1370$
Largest difference peak and hole ($e\,Å^{-3}$)	0.129 and −0.139	0.991 and −0.629

Table 2. Main IR bands for H_2L and its cluster (cm^{-1}).

Compound	$\nu_{(C=N)}$	$\nu_{(Ar–O)}$	$\nu_{(Ni–N)}$	$\nu_{(Ni–O)}$	$\nu_{(O–H)}$	$\nu_{(C=C)}$ benzene ring skeleton
H_2L	1633	1282	–	–	3433	1571, 1510, 1448
Cluster	1611	1170	475	407	3437	1538, 1494, 1446

407 and 475 cm^{-1}, respectively, consistent with the literature [28, 29]. As pointed out by Percy and Thornton [30], the metal–oxygen and metal–nitrogen frequency assignments are at times difficult.

3.3. *Crystal structure of H_2L*

The crystal structure of H_2L is shown in figure 1. Selected bond lengths and angles of H_2L are given in the "Supplementary material". The structure of the ligand H_2L adopts an *L*-shape conformation where the two salicylaldoxime moieties are well separated from each other. The dihedral angle of the corresponding two benzene rings

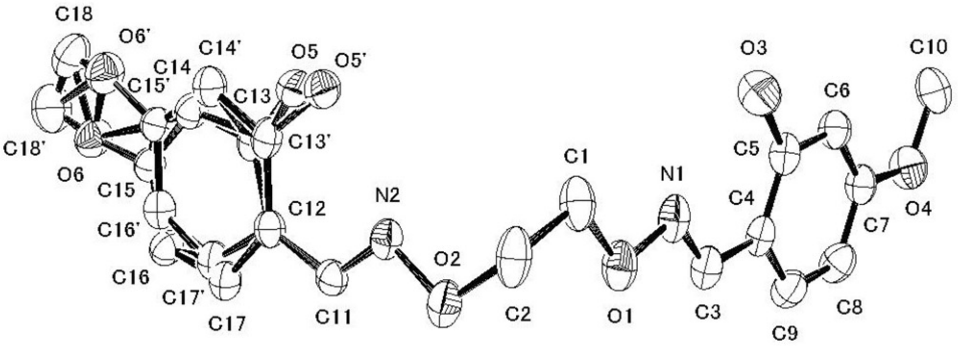


Figure 1. Molecule structure and atom numberings of H_2L with hydrogen atoms omitted for clarity.

in H_2L is 85.10(3)°. The oxime groups and phenolic alcohols have the anti-conformation.

In the crystal structure, there are two strong intramolecular $O3{-}H3\cdots N1$, $O5{-}H5\cdots N2$ hydrogen bonds between the phenolic hydroxyl and the oxime nitrogen, which generate six-membered S(6) ring motifs (Supplementary material) [31–33]. In addition, intermolecular $C16{-}H16\cdots O1$ hydrogen bonds link a pair of molecules to produce a 20-membered ring with a graph motif of $R_2^2(20)$ (Supplementary material) [34, 35]. Neighboring molecules are further interlinked by the intermolecular $C10{-}H10A\cdots\pi_{\mathrm{centroid(C4–C9)}}$ and $C18{-}H18C\cdots\pi_{\mathrm{centroid(C12–C17)}}$ hydrogen bonds between methoxy and the benzene rings to form a 1-D infinite chain along the *a*-axis (Supplementary material) [36]. With the help of $O{-}H\cdots N$, $C{-}H\cdots O$, and $C{-}H\cdots\pi$ hydrogen bonds interactions, the packing diagrams form a rectangle-like large cave structure along the *a*-axis, as illustrated in the "Supplementary material" [28, 37].

3.4. *Crystal structure of* $\{[(NiL)(n\text{-}BuOH)]_2(\mu\text{-}OAc)_2Ni\}\cdot n\text{-}BuOH$

The molecular structure of the cluster is shown in figure 2. Selected bond lengths and angles are given in table 3. The cluster crystallizes in the triclinic system, space group *P*-1 with a linear trinuclear array of three Ni(II)'s coupled by both double μ-phenoxo oxygen atoms of L^{2-} and simultaneously two acetates in the *syn–syn* bridging mode. All six-coordinate Ni(II)'s have slightly distorted octahedra. The two terminal Ni(II) (Ni2 and $Ni2^{\#1}$) are located in the N_2O_2 coordination sphere of L^{2-} (figure 1). One oxygen (O8) from the μ-acetato bridge and one oxygen (O9) from the *n*-BuOH molecule are also coordinated to Ni2. Consequently, the dihedral angle between the coordination plane of O5–Ni2–N2 and that of O3–Ni2–N1 is about 11.85(3)°, indicating slight distortion toward octahedral geometry from the square planar structure, which is the same as the environment of $Ni2^{\#1}$. The coordination sphere of the central Ni(II) (Ni1) contains quadruple μ-phenoxo oxygen atoms (O3, O5, $O3^{\#1}$, and $O5^{\#1}$) from two L^{2-} and double μ-acetato oxygen atoms (O7 and $O7^{\#1}$) that adopt a familiar μ–O–C–O. All six oxygen atoms coordinated to Ni1 constitute octahedral geometry.

There is an inversion center through Ni1 and the distance of Ni1–O7 (2.022(2) Å) is shorter than Ni1–O5 (2.094(2) Å) (table 3), indicating a weak steric effect. Similar

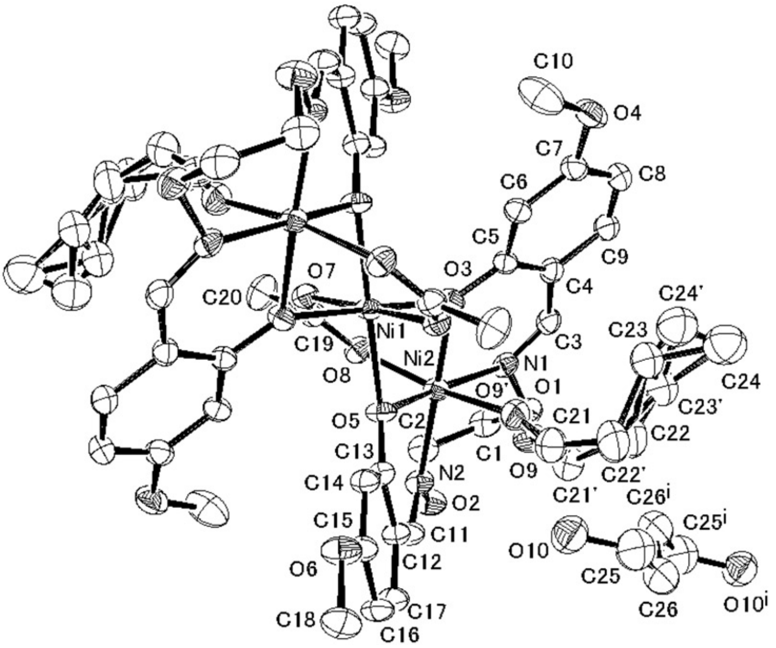


Figure 2. Molecular structure and atom numberings of the cluster (hydrogen atoms are omitted for clarity).

Table 3. Selected bond distances (Å) and angles (°) for the cluster.

Ni1–O7	2.022(3)	Ni1–O7$^{\#1}$	2.022(3)	Ni1–O3	2.087(3)
Ni1–O3$^{\#1}$	2.087(3)	Ni1–O5$^{\#1}$	2.093(3)	Ni1–O5	2.093(3)
Ni2–O5	1.998(3)	Ni2–O3	2.000(3)	Ni2–N1	2.030(4)
Ni2–O8	2.037(3)	Ni2–N2	2.042(4)	Ni2–O9′	2.200(15)
Ni2–O9	2.285(12)				
O7–Ni1–O7$^{\#1}$	180.00	O7–Ni1–O3	89.66(12)	O7$^{\#1}$–Ni1–O3	90.34(12)
O7–Ni1–O3$^{\#1}$	90.34(12)	O7$^{\#1}$–Ni1–O3$^{\#1}$	89.66(12)	O3–Ni1–O3$^{\#1}$	180.00
O7–Ni1–O5$^{\#1}$	91.22(12)	O7$^{\#1}$–Ni1–O5$^{\#1}$	88.78(12)	O3–Ni1–O5$^{\#1}$	100.61(11)
O3$^{\#1}$–Ni1–O5$^{\#1}$	79.39(11)	O7–Ni1–O5	88.78(12)	O7$^{\#1}$–Ni1–O5	91.22(12)
O3–Ni1–O5	79.39(11)	O3$^{\#1}$–Ni1–O5	100.61(11)	O5$^{\#1}$–Ni1–O5	180.00
O5–Ni2–O3	83.78(11)	O5–Ni2–N1	167.31(14)	O3–Ni2–N1	89.73(13)
O5–Ni2–O8	92.52(12)	O3–Ni2–O8	89.51(13)	N1–Ni2–O8	98.34(14)
O5–Ni2–N2	88.38(13)	O3–Ni2–N2	171.41(14)	N1–Ni2–N2	97.27(15)
O8–Ni2–N2	94.36(14)	O5–Ni2–O9′	77.1(4)	O3–Ni2–O9′	77.0(5)
N1–Ni2–O9′	90.8(4)	O8–Ni2–O9′	163.7(5)	N2–Ni2–O9′	97.9(5)
O5–Ni2–O9	88.8(3)	N2–Ni2–O9	84.0(3)	N1–Ni2–O9	80.5(3)
O8–Ni2–O9	177.9(3)	C3–N1–Ni2	124.1(3)	O9′–Ni2–O9	18.5(3)
C5–O3–Ni1	133.7(3)	C11–N2–Ni2	122.8(3)	O1–N1–Ni2	121.3(3)
C13–O5–Ni2	124.5(2)	Ni2–O3–Ni1	95.85(12)	O2–N2–Ni2	126.0(3)
C19–O7–Ni1	129.5(3)	C13–O5–Ni1	133.8(3)	C5–O3–Ni2	127.8(3)
C21′–O9′–Ni2	125.7(15)	C19–O8–Ni2	126.8(3)	Ni2–O5–Ni1	95.72(12)
C21–O9–Ni2	134.3(10)	O3–Ni2–O9	92.3(3)	C22′–O9′–Ni2	171.5(13)

Symmetry transformations used to generate equivalent atoms: $^{\#1}$ $-x+1$, $-y+1$, $-z+1$.

elongations of M–O bonds have been observed in [M(Salen)]$_2$ [38]. The Ni2–N2 bond (2.042(4) Å) is slightly longer than Ni2–N1 (2.030(4) Å), which is attributed to the *n*-BuOH coordinating to Ni2 and Ni2$^{\#1}$ resulting in the larger steric hindrance. The Ni1–Ni2 distance (3.034(3) Å) is significantly longer than all the Ni–O and Ni–N bonds

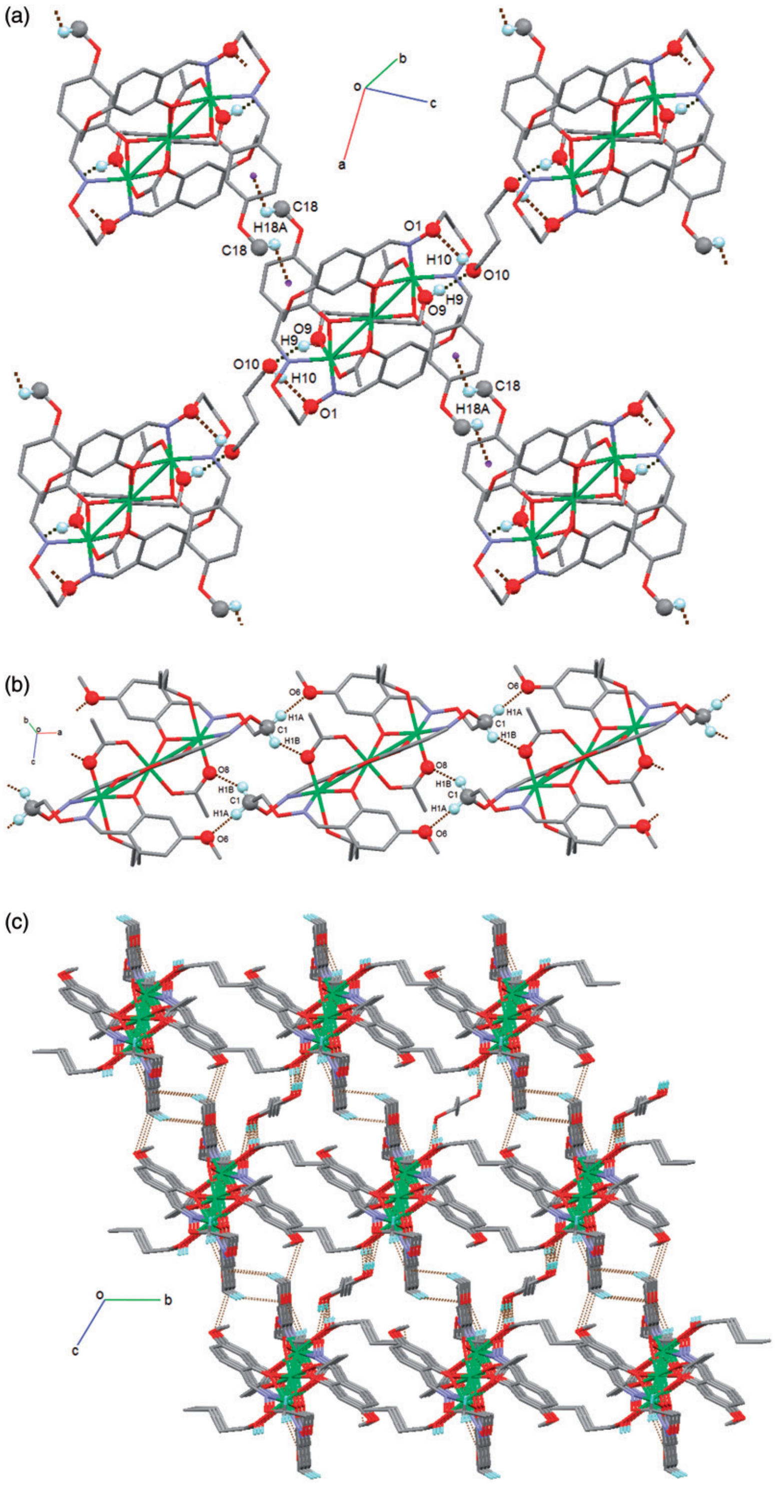


Figure 3. The supramolecular structure of the Ni(II) cluster (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity). (a) View of the 2-D layer motifs parallel to the *ac* plane; (b) View of the 1-D chain motif along the *a*-axis; (c) View of the 3-D supramolecular network motifs.

Table 4. Intermolecular hydrogen bonds for the cluster (Å, °).

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠DHA
O9–H9···O10	0.82	1.98	2.803(3)	176
O10–H10···O1	0.82	2.21	3.024(3)	177
C1–H1A···O6	0.97	2.42	3.322(2)	154
C1–H1B···O8	0.97	2.68	3.487(5)	140
C18–H18A···$\pi_{\text{centroid(C12–C17)}}$	0.96	3.45	4.067(3)	124
O9$^{\#1}$–H9$^{\#1}$···O7	0.82	2.60	3.250(5)	137

(2.084–2.134 Å), indicating weak inter-metal interaction, similar to that previously reported for a salen-type cluster, $\{[\mathrm{Ni(Salpr)NC_5H_5}]_2(\mu\mathrm{OAc})_2\mathrm{Ni}\}$ [39].

The cluster contains one non-coordinated *n*-BuOH molecule and there are complicated hydrogen bonding interactions (figure 3), hydrogen bond data are given in table 4. In the cluster, as shown in figure 3, each disordered *n*-butanol interlinks two neighboring clusters through intermolecular O10–H10···O1 and O9–H9···O10 hydrogen bonds into an infinite chain. Synchronously, the neighboring clusters are further interlinked by a pair of intermolecular C18–H18A···$\pi_{\text{centroid(C12–C17)}}$ hydrogen bonding interactions between –CH moiety of the methoxy and the benzene of L^{2-} to form the other 1-D infinite chain [40–42]. Thus, the clusters and crystallizing disordered *n*-butanols are interlinked by intermolecular hydrogen bonds to form an infinite 2-D layer supramolecular structure parallel to the *ac* crystallographic plane (figure 3a). This linkage is further stabilized *via* two pairs of intermolecular C1–H1A···O6 and C1–H1B···O8 hydrogen bonds between –CH of the *O*-alkyl chain and O6 of methoxy of L^{2-} and O5 of μ-acetate, to form a 1-D infinite chain along the *a*-axis (figure 3b). The crystal packing of the cluster shows that a 3-D supramolecular network was formed through intermolecular O–H···O, C–H···O, and C–H···π hydrogen bond interactions (figure 3c).

4. Conclusion

A new salen-type bisoxime 5,5′-dimethoxy-2,2′-[(ethylenedioxy)bis(nitrilomethylidyne)] diphenol ($\mathrm{H_2L}$) and its trinuclear Ni(II) cluster, $\{[(\mathrm{NiL})(n\text{-BuOH})]_2(\mu\text{-OAc})_2\mathrm{Ni}\}\cdot n\text{-BuOH}$, have been synthesized and structurally characterized. The structure of $\mathrm{H_2L}$ contains a pair of crystallographically independent but chemically identical molecules. With O–H···N, C–H···O, and C–H···π hydrogen bonding interactions, a self-assembling infinite 2-D layer supramolecular structure is formed. A 3-D supramolecular network structure is formed in the cluster through intermolecular O–H···O, C–H···O, and C–H···π hydrogen bonding interactions.

Supplementary material

Further details of the crystal structure investigation(s) may be obtained from the Cambridge Crystallographic Data Centre, Postal Address: CCDC, 12 Union Road, CAMBRIDGE CB2 1EZ, UK. Telephone: (44) 01223 762910; Facsimile: (44) 01223

336033; Email: deposit@ccdc.cam.ac.uk on quoting the depository number CCDC Nos: 757718, 712167 for H_2L and 757718 for the cluster.

Acknowledgments

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