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Synthesis, crystal structure and spectral properties of a supramolecular trinuclear nickel(II) complex with 5-methoxy-4′-bromo-2,2′-[ethylenedioxybis(nitrilomethylidyne)]diphenol

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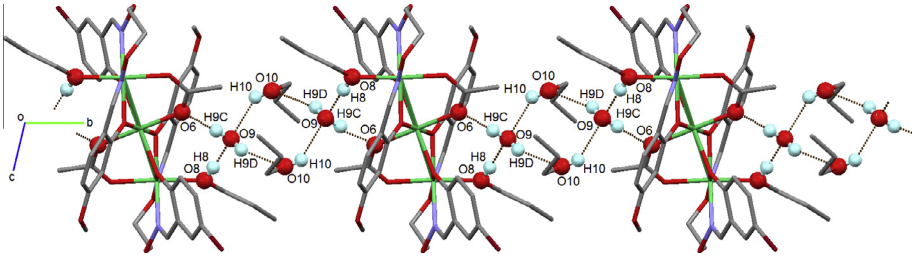
HIGHLIGHTS

- A new asymmetric Salamo-type bisoxime has been synthesized firstly.
- A novel complex {[NiL(*n*-PrOH)(*μ*-OAc)]$_2$Ni}·*n*-PrOH·H_2O has been synthesized and characterized structurally.
- The hydrogen-bonding interactions have stabilized the Ni(II) complex molecules to form an infinite 1D supramolecular chain-like structure.
- New results are very important to modern coordination and supramolecular chemistry.

GRAPHICAL ABSTRACT

A supramolecular trinuclear Ni(II) complex {[NiL(*n*-PrOH)(*μ*-OAc)]$_2$Ni}·*n*-PrOH·H_2O with 5-methoxy-4′-bromo-2,2′-[ethylenedioxybis(nitrilomethylidyne)]diphenol (H_2L), has been synthesized and characterized structurally. Two *n*-propanol molecules are coordinated to the two terminal Ni(II) ions which are linked by two acetate groups coordinated to the central Ni(II) ion forming Ni—O—C—O—Ni bridges. Four *μ*-phenoxo oxygen atoms from two deprotonated L^{2-} units also bridge the three Ni(II) ions, forming a trinuclear structure. There are also one non-coordinated *n*-propanol and one water molecule. In the crystal structure, the Ni(II) complex is linked by intermolecular hydrogen bonds into an infinite 1D supramolecular chain.



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ABSTRACT

A novel trinuclear Ni(II) complex [{NiL(*n*-PrOH)(*μ*-OAc)}$_2$Ni]·*n*-PrOH·H_2O with an asymmetric Salamo-type ligand, 5-methoxy-4′-bromo-2,2′-[ethylenedioxybis(nitrilomethylidyne)]diphenol (H_2L), has been synthesized and characterized by elemental analyses, IR, UV/Vis and fluorescence spectra and molar conductance measurement. The crystal structure of the Ni(II) complex has been determined by single-crystal X-ray diffraction. Two acetate groups coordinating to three Ni(II) ions through Ni—O—C—O—Ni bridges and four *μ*-phenoxo oxygen atoms from two [NiL(*n*-PrOH)] units also coordinating to Ni(II) ions. In the Ni(II) complex, two *n*-propanol molecules are coordinated to the two terminal Ni(II) ions having slightly distorted octahedral coordination geometries and form a trinuclear structure, There are also one non-coordinated *n*-propanol and one non-coordinated water molecule. In the crystal structure, the Ni(II) complex is linked by intermolecular hydrogen bonds into an infinite 1D supramolecular chain-like structure.

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Introduction

Transition metal complexes with Salen-type ligands are now an important research field [1,2]. These complexes can be used to obtain different catalysts [3,4], photonic devices [5–7], magnetic materials [8–10] and models of biological systems [11,12]. Supramolecular complexes, in which the central metal ions are bridged through Salen-type ligands are of much current interest, owing to their enormous variety of structures, as well as their potential applications [13–18]. To generate target complexes by design, an advisable selection of ligands is required in order to control the supramolecular interactions [19]. Regarding the symmetric Salamo-type ligand, an asymmetric variant is important because electronic and steric effect of the ligands may be controlled by introduction of different substituents into the two benzene rings [20]. It has been reported that transition metal complexes derived from asymmetric Salen-type ligands may exhibit better enantioselectivities when compared with their symmetric counterparts [21]. Solvent effect can lead to better ways to control supramolecular interactions [22]. With this in mind, we have designed and synthesized an asymmetric Salamo-type ligand, 5-methoxy-4′-bromo-2,2′-[ethylenedioxybis(nitrilomethylidyne)]diphenol (H_2L), and its supramolecular trinuclear Ni(II) complex [{NiL(*n*-PrOH)(μ-OAc)}$_2$ Ni]·*n*-PrOH·H_2O in *n*-propanol solution.

Experimental

Materials

2-Hydroxy-4-methoxybenzaldehyde ($\geqslant$99%) and 5-bromo-2-hydroxybenzaldehyde ($\geqslant$99%) were purchased from Alfa Aesar and used without further purification. The other reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory.

Methods

The methods are the same as literature early [23].

Synthesis of the ligand H_2L

H_2L was synthesized according to an analogous method reported earlier [24]. The reaction steps involved in the synthesis of the asymmetric Salamo-type ligand (H_2L) is shown in Scheme 1.

A solution of 2-[*O*-(1-ethyloxyamide)]oxime-5-methoxyphenol (0.45 g, 2 mmol) in ethanol (20 mL) was added to a solution of 2-hydroxy-5-bromobenzaldehyde (0.40 g, 2 mmol) in ethanol (20 mL). The mixture was stirred at 55 °C for 5 h. After cooling to room temperature, the precipitates were collected on a suction filter and washed successively with ethanol and ethanol/hexane (1:4). The product was dried in *vacuo*, and a colorless flocculent crystalline solid was obtained. Yield: 73.8%; M. p. 112–114 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ/ppm) δ = 3.90 (s, 3H), 4.46–4.49 (m, 4H), 6.86 (d, *J* = 8.6 Hz, 1H), 6.90 (t, *J* = 7.8 Hz, 1H), 6.99 (d, *J* = 7.8 Hz, 1H), 7.15 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.24 (d, *J* = 2.5 Hz, 1H), 7.36 (dd, *J* = 8.6, 2.5 Hz, 1H), 8.15 (s, 1H), 8.22 (s, 1H), 9.70 (s, 1H), 9.76 (s, 1H). Anal. Calcd for $C_{17}H_{17}BrN_2O_5$ (%): C 49.89; H 4.19; N 6.85. Found (%): C 49.75; H 4.32; N 6.99.

Synthesis of the nickel(II) complex

A solution of $Ni(OAc)_2 \cdot 4H_2O$ (3.73 mg, 0.015 mmol) in *n*-propanol (2 mL) was added dropwise to a solution of H_2L (4.10 mg, 0.010 mol) in acetonitrile (2 mL) at room temperature. The color of the solution turned to green immediately, and was stirred for 2 h at room temperature. The mixture was filtered and the filtrate was allowed to stand at room temperature for about three weeks. The solvent was partially evaporated and several green prismatic single crystals suitable for X-ray crystallographic analysis were obtained. Yield: 31.5%; Anal. Calcd for $C_{50}H_{72}Br_2N_4Ni_3O_{20}$ ([{NiL(*n*-PrOH)(μ-OAc)}$_2$Ni]·*n*-PrOH·H_2O) (%): C 43.36; H 5.24; N 4.05; Ni 12.71. Found (%): C 43.52; H 5.41; N 3.89; Ni 12.55.

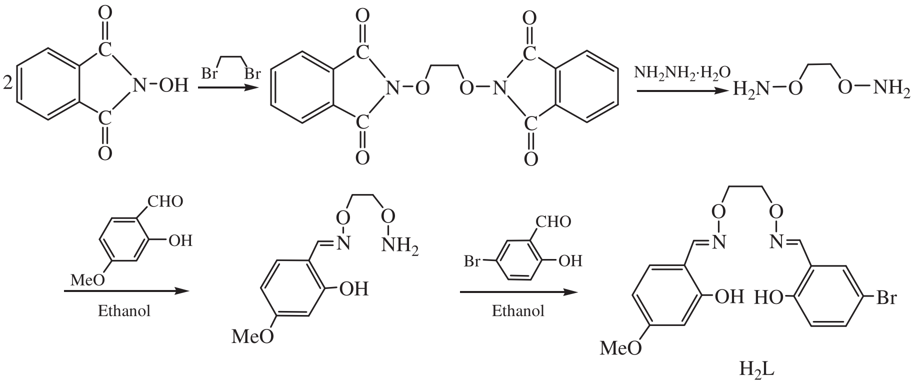
X-ray crystallography

Details of the data collection and refinements of the Ni(II) complex are given in Table 1. A single crystal of the Ni(II) complex with the approximate dimensions of 0.18 × 0.10 × 0.09 mm was placed on a Bruker Smart 1000 CCD area detector. The reflections were collected using a graphite monochromated Mo Kα radiation (λ = 0.71073 Å) at 298(2) K. The structure was solved by using the program SHELXL-97 and Fourier difference techniques, and refined by the full-matrix least-squares method on F^2. All hydrogen atoms were added in calculated positions. The non-hydrogen atoms were refined anisotropically. All hydrogen atoms were added theoretically.

Results and discussion

Molar conductances

The Ni(II) complex is soluble in DMF and DMSO, slightly soluble in THF, but not soluble in ethanol, methanol, acetonitrile, acetone, ethyl acetate and hexane. The molar conductance value at 25 °C in 1.0×10^{-3} mol dm^{-3} DMF solution is 5.7 S cm^2 mol^{-1}, indicating a non-electrolyte [25], which implies that all Ni(II) ions and acetates



Scheme 1. Synthetic route to the asymmetrical Salamo-type ligand H_2L.

Table 1
Crystal data and structure refinement for the Ni(II) complex.

Molecular formula	$C_{50}H_{72}Br_2N_4Ni_3O_{20}$
Formula weight	1385.07
T (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P-1$
a (Å)	10.7475(9)
b (Å)	12.1999(11)
c (Å)	12.4027(14)
α (°)	102.016(2)
β (°)	112.000(3)
γ (°)	90.9150(10)
V (Å^3)	1466.8(2)
Z	1
D_c (Mg m^{-3})	1.568
Absorption correction	Semi-empirical from equivalents
$F(000)$	714
Crystal size (mm)	$0.18 \times 0.10 \times 0.09$
θ Range for data collection (°)	2.78–25.02
Index ranges	$-12 \leqslant h \leqslant 12$, $-14 \leqslant k \leqslant 13$, $-14 \leqslant l \leqslant 14$
Completeness to $\theta = 25.02$ (%)	99.8
Reflections collected	9325
Independent reflections	5177[R(int) = 0.0616]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5177/0/429
Goodness-of-fit on F^2	1.041
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0767$, $wR_2 = 0.1626$
R indices (all data)	$R_1 = 0.1482$, $wR_2 = 0.2186$
$(\Delta\rho)_{max}$ (e Å^{-3})	0.830
$(\Delta\rho)_{min}$ (e Å^{-3})	−0.716

in the Ni(II) complex are firmly held in the coordination sphere in solution.

IR spectra

IR spectra of H_2L and its corresponding Ni(II) complex exhibit various bands in the 100–4000 cm^{-1} region. The most important bands are given in Table 2.

The free ligand exhibits characteristic Ar—O and C=N stretching bands at 1263 and 1630 cm^{-1}, respectively, which are shifted to lower frequencies (1215 and 1612 cm^{-1}) for the Ni(II) complex. This lowering of energy results from the Ni—O and Ni—N interactions upon complexation, which is similar to previously reported Ni(II) complexes [26,27]. The IR spectrum of the Ni(II) complex shows the expected O—H stretching band and H—O—H bending mode at ca. 3417 and 1637 cm^{-1}, which are the evidence for the existence of n-propanol and water molecules [28].

The far-infrared spectrum of the Ni(II) complex was obtained in the region 500–100 cm^{-1} to identify frequencies due to the Ni—O and Ni—N bonds. Weak bands at 478 and 409 cm^{-1} are assigned to ν(Ni—N) and ν(Ni—O) [29], as pointed out by Percy and Thornton [30]. The metal-oxygen and metal-nitrogen frequency assignments are at times very difficult.

UV–vis absorption spectra

UV–vis absorption spectra of H_2L and its corresponding Ni(II) complex in 5.0×10^{-5} mol L^{-1} DMF solution are shown in Fig. 1. The absorption of the Ni(II) complex is obviously different from that of H_2L owing to complexation. For the free ligand there are two intense peaks centered at around 283 and 312 nm, assigned to π–π* transitions of the benzene rings of the benzaldehyde and oxime groups [31]. The absorption at 283 is slightly shifted bathochromically to 285 nm and the absorption at 312 nm is absent in the spectrum of the Ni(II) complex. A new intense absorption peak is observed at 351 nm for the Ni(II) complex, which is assigned to the n–π* charge transfer from the filled pπ orbital of the bridging phenolic oxygen to vacant d-orbital of the Ni(II) ions.

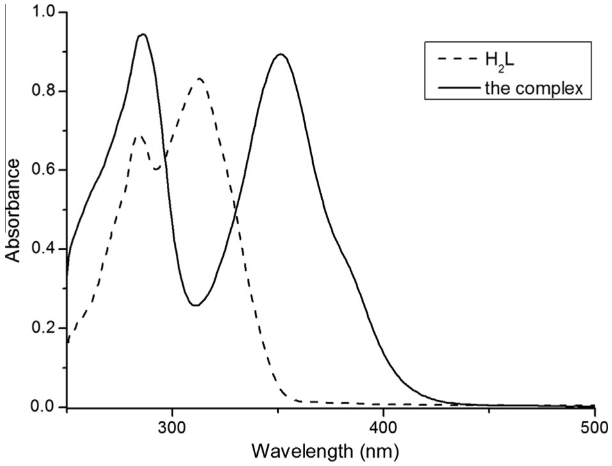


Fig. 1. UV/Vis absorption spectra of H_2L and its corresponding Ni(II) complex in DMF ($c = 5 \times 10^{-5}$ mol L^{-1}).

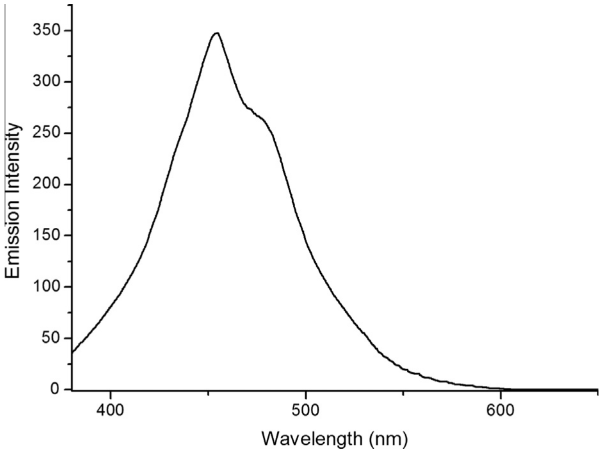


Fig. 2. Emission spectrum of the Ni(II) complex ($\lambda_{ex} = 351$ nm) in DMF solution at room temperature ($c = 5 \times 10^{-5}$ mol L^{-1}).

Fluorescence spectra

The emission spectrum of the Ni(II) complex in dilute DMF at room temperature is shown in Fig. 2. The Ni(II) complex shows an intense photoluminescence with maximum emission at ca. 455 nm upon excitation at 351 nm. Compared with the free ligand,

Table 2
Main IR bands for H_2L and its corresponding Ni(II) complex (cm^{-1}).

Compound	$\nu_{(C=N)}$	$\nu_{(Ar—O)}$	$\nu_{(O—H)}$	$\nu_{(C=C)\text{benzene rings keleton}}$	$\nu_{(Ni—N)}$	$\nu_{(Ni—O)}$
H_2L	1630	1263	3415	1575, 1515, 1477	-	-
Complex	1612	1215	3417	1535, 1469, 1440	478	409

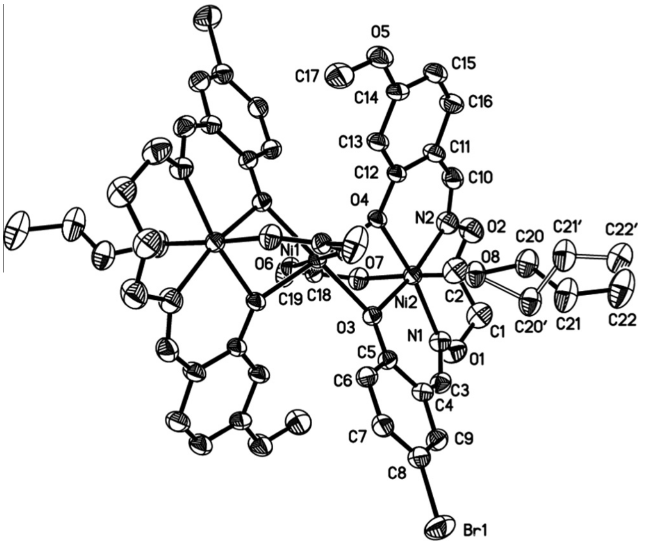


Fig. 3. ORTEP drawing of the Ni(II) complex with atom numbering (Displacement ellipsoids for non-H atoms are drawn at the 30% probability level, the cocrystallizing *n*-propanol and water molecules are omitted).

strong fluorescence intensity of the Ni(II) complex is observed, indicating that fluorescent characteristic has been influenced by the introduction of the Ni(II) ions [32].

Crystal structure of the Ni(II) Complex

ORTEP-style drawing and atom numbering of the Ni(II) complex is shown in Fig. 3. Selected bond lengths and bond angles are listed in Table 3. The Ni(II) complex crystallizes in the triclinic system, space group *P*−1 with three Ni(II) atoms, two deprotonated L^{2-} units, two *μ*-acetato anions, two coordinated *n*-propanol molecules, one non-coordinated *n*-propanol molecule and one non-coordinated water molecule resulting in a trinuclear Ni(II) complex [33–39]. The coordination sphere of terminal Ni(II) (Ni2 and $Ni2^{\#1}$) atoms is completed by N_2O_2 donors, an oxygen (O7) atom from the *μ*-acetato anion and an oxygen (O8) atom from the disordered coordinated *n*-propanol molecule in the axial position. The Ni(II) (Ni1) atom is located on a centre of inversion, and coordinated by four *μ*-phenoxo oxygen (O3, O4, $O3^{\#1}$ and $O4^{\#1}$) atoms from two deprotonated L^{2-} units, and two *μ*-acetato oxygen (O6 and $O6^{\#1}$) atoms. Thus, all of three Ni(II) atoms in the Ni(II) complex are hexa-coordinated, with slightly distorted octahedral coordination geometries.

The terminal Ni(II) (Ni2 or $Ni2^{\#1}$) atom is 0.038(3) Å out of the N_2O_2 equatorial plane designed by atoms (N1—O3—O4—N2) with the dihedral angle between the two representative planes, N1—Ni2—O3 and N2—Ni2—O4, being 3.37(5)°. The central Ni(II)

Table 4
The data for hydrogen-bonding interactions (Å, °).

D—H···A	d(D—H)	d(H···A)	d(D···A)	∠D—H···A	Symmetry code
O8—H8···O9	0.82	1.81	2.63(1)	178	*x*, *y*, *z*
O9—H9C···O6	0.85	1.87	2.72(1)	176	1 − *x*, −*y*, 1 − *z*
O9—H9D···O10	0.85	2.02	2.75(6)	144	*x*, *y*, *z*
O10—H10···O9	0.82	2.14	2.72(5)	128	1 − *x*, 1 − *y*, 1 − *z*
C2—H2B···O7	0.97	2.37	3.22(1)	146	*x*, *y*, *z*
C13—H13···O6	0.93	2.59	3.21(1)	125	1 − *x*, −*y*, 1 − *z*

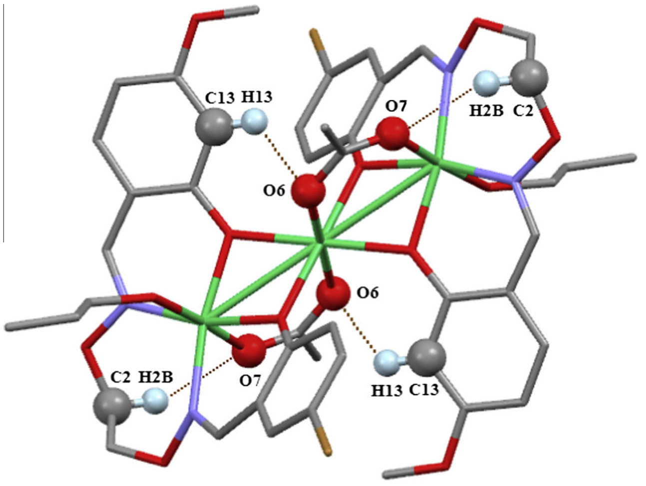


Fig. 4. View of the intramolecular hydrogen-bonding interactions of the Ni(II) complex.

Table 3
Selected bond lengths [Å] and bond angles [°] for the Ni(II) complex.

Bond	Dist.	Bond	Dist.	Bond	Dist.
$Ni1—O4^{\#1}$	2.049(5)	$Ni1—O3^{\#1}$	2.092(5)	Ni2—O4	2.026(5)
Ni1—O4	2.049(5)	Ni1—O3	2.092(5)	Ni2—N2	2.046(8)
Ni1—O6	2.087(5)	Ni2—O3	2.017(5)	Ni2—N1	2.086(7)
$Ni1—O6^{\#1}$	2.087(5)	Ni2—O7	2.019(6)	Ni2—O8	2.105(6)
Bond	Angles	Bond	Angles	Bond	Angles
$O4^{\#1}—Ni1—O4$	180.0(1)	O3—Ni2—O7	92.4(2)	C3—N1—Ni2	123.4(6)
$O4^{\#1}—Ni1—O6$	91.7(2)	O3—Ni2—O4	79.3(2)	O1—N1—Ni2	128.6(5)
O4—Ni1—O6	88.3(2)	O7—Ni2—O4	91.7(2)	C10—N2—Ni2	124.7(7)
$O4^{\#1}—Ni1—O6^{\#1}$	88.3(2)	O3—Ni2—N2	166.7(3)	O2—N2—Ni2	127.1(6)
$O4—Ni1—O6^{\#1}$	91.7(2)	O7—Ni2—N2	91.1(3)	C5—O3—Ni2	125.6(5)
$O6—Ni1—O6^{\#1}$	180.0(1)	O4—Ni2—N2	87.7(3)	C5—O3—Ni1	134.1(5)
$O4^{\#1}—Ni1—O3^{\#1}$	77.1(2)	O3—Ni2—N1	88.1(2)	Ni2—O3—Ni1	97.8(2)
$O4—Ni1—O3^{\#1}$	102.9(2)	O7—Ni2—N1	89.5(3)	C12—O4—Ni2	125.8(5)
$O6—Ni1—O3^{\#1}$	90.6(2)	O4—Ni2—N1	167.4(3)	C12—O4—Ni1	132.8(5)
$O6^{\#1}—Ni1—O3^{\#1}$	89.4(2)	N2—Ni2—N1	104.8(3)	Ni2—O4—Ni1	98.9(2)
$O4^{\#1}—Ni1—O3$	102.9(2)	O3—Ni2—O8	89.0(2)	C18—O6—Ni1	130.1(5)
O4—Ni1—O3	77.1(2)	O7—Ni2—O8	176.3(2)	C18—O7—Ni2	129.0(6)
O6—Ni1—O3	89.4(2)	O4—Ni2—O8	91.9(2)	C20—O8—Ni2	128.0(10)
$O6^{\#1}—Ni1—O3$	90.6(2)	N2—Ni2—O8	88.3(3)	C20′—O8—Ni2	135(2)
$O3^{\#1}—Ni1—O3$	180.0(1)	N1—Ni2—O8	87.2(3)		

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

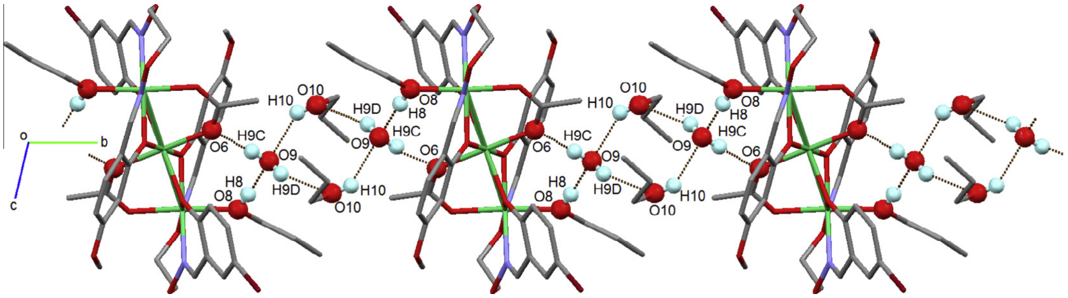


Fig. 5. View of the 1D chain motif of the Ni(II) complex units (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity).

(Ni1) atom lies in its equatorial plane designed by atoms (O3—O4—O3—O4), as well as the dihedral angle between the N_2O_2 equatorial plane and the O_2O_2 equatorial plane is 24.27(4)°. Furthermore, the distances of Ni1 and Ni2 (or Ni2$^{\#1}$) atoms to the six donors have a small difference (2.049–2.092 Å and 2.017–2.105 Å). The Ni1—Ni2 distance (3.096(3) Å) is significantly longer than all of the Ni—O and Ni—N bonds, indicating weak inter-metal interaction, which is essentially similar to a previously reported Salen-type complex of {[Ni(Salpr)NC_5H_5]$_2$(μ-OAc)$_2$Ni} [16]. The Ni—N bonds (2.046(8) and 2.086(7) Å) around the terminal Ni(II) (Ni2 and Ni2$^{\#1}$) atoms are slightly longer than Ni—O bonds (2.017(5) and 2.026(5) Å) owing to the larger steric hindrance.

Supramolecular interaction

In the crystal structure, there are abundant hydrogen bonding (O8—H8···O9, O9—H9C···O6, O9—H9D···O10, O10—H10···O9, C2—H2B···O7 and C13—H13···O6) interactions between the Ni(II) complex and the crystallizing *n*-propanol and water molecules. Hydrogen bond data are summarized in Table 4. There are two pairs of intramolecular C2—H2B···O7 and C13—H13···O6 hydrogen-bonding interactions in the Ni(II) complex (Fig. 4). The oxygen (O9) atom of the water molecule is hydrogen-bonded to the —O8H8 group of the coordinated *n*-propanol molecule and the —O10H10 group of the *n*-propanol molecule, while its —O9H9C and —O9H9D groups are hydrogen-bonded to the oxygen (O6) atom of the bridging acetate anion and the oxygen (O10) atom of the second *n*-propanol molecule.

The components of the crystal are linked by intermolecular hydrogen bonds into an infinite 1D chain along the *b* axis (Fig. 5).

Conclusions

We have designed and synthesized a supramolecular trinuclear Ni(II) complex with an asymmetric Salamo-type ligand. The Ni(II) complex has been characterized by physicochemical methods and single-crystal X-ray diffraction. All three Ni(II) atoms are hexa-coordinated by two deprotonated L^{2-} units, two μ-acetato anions and two coordinated *n*-propanol molecules. The supramolecular structure is stabilized by hydrogen bonding of *n*-propanol and water molecules, compared with other solvent (such as ethanol and methanol) molecules, which could be useful for the development of novel transition metal complexes and deserve further study based on their potential applications.

Acknowledgements

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2014.04.168.

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