Contents lists available at ScienceDirect

Inorganica Chimica Acta

journal homepage: www.elsevier.com/locate/ica

Research paper

Tetranuclear cobalt(II) and nickel(II) complexes with an unsymmetrical salamo-like ligand: Structural characterization, Hirshfeld analysis and fluorescent properties

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ARTICLE INFO

Keywords: Salamo-like donor-N₂O₄ ligand Tetranuclear metal(II) complex Synthesis Crystal structure Hirshfeld surface analysis

ABSTRACT

Two newly designed tetranuclear transition metal(II) complexes, $[{Co(L)(\mu-OAc)Co(CH_3OH)_2}_2]$ and $[{Ni(L) (DMF)(\mu-OAc)Ni}_2]$ derived from an unsymmetrical salamo-like donor-N₂O₄ ligand (H₃L: 6-hydroxy-6'-methoxy-2, 2'-[ethylenediyldioxybis(nitrilomethylidyne)]diphenol) were synthesized and characterized by elemental analysis, FT-IR, UV-vis, fluorescence spectroscopy, X-ray crystallography and Hirshfeld surface analysis. The Co (II) complex includes two fully deprotonated (L)³⁻ moieties, two μ -acetato ligands and four coordinated methanol molecules, bearing a highly symmetrical tetranuclear structure. The terminal Co(II) ions are penta-coordinated and have slightly twisted triangular bipyramidal geometries, and the central Co(II) ions are hexa-coordinated and possess slightly twisted octahedral geometries. The Co(II) complex was self-assembled by adequate intermolecular hydrogen bonding interactions giving rise to an infinite two-dimensional supramolecular structure. The Ni(II) complex adopts a symmetric tetranuclear structure including two fully deprotonated (L)³⁻ units, two coordinated DMF molecules, and two μ -acetato ligands. All the Ni(II) ions are hexa-coordinated and have slightly twisted octahedral geometries. At the same time, the fluorescent properties and Hirshfeld surfaces analyses of the Co(II) and Ni(II) complexes were also investigated in detail.

1. Introduction

Salen and its analogues (R–CH=N–(CH₂)₂–N=CH–R) and their transition metal(II) complexes have been extensively investigated in modern coordination chemistry for several decades [1–7], and their potential applications have been explored in biological systems [8–11], electrochemistry [12], nonlinear optical [13–15] and magnetic materials [16,17], luminescent materials [18,19] supramolecular architectures [20–24] and so on.

As derivatives of salen, in recent years, our research mostly concentrated on the syntheses of salamo-like ligands [25–31]. These compounds have been investigated in forming transition metal(II) complexes with interesting properties [32,33]. Some work has been devoted to the synthesis and characterization of mono- [34], di- and multi-nuclear transition metal(II) complexes bearing salamo-like ligands and their derivatives [35,36]. There are few investigations on unsymmetric salamo-like ligands, and it is expected that multi-nuclear transition metal(II) complexes can be obtained by introducing some groups such as alkoxy or hydroxyl substituents at the 3-positions of the salicylaldehyde derivatives in the salamo-like ligands. Herein, as part of our ongoing interest in salamo-like transition metal(II) complexes, X-ray crystal structures, spectroscopic properties and Hirshfeld surfaces analyses of two newly designed and synthesized transition metal(II) complexes [{Co(L)(μ -OAc)Co(CH₃OH)₂}] and [{Ni(L)(DMF)(μ -OAc)Ni}₂] derived from an unsymmetrical salamo-like donor-N₂O₄ ligand H₃L have been investigated in detail.

2. Experimental

2.1. Materials and physical measurements

3-Hydroxysalicylaldehyde (99%) and 3-methoxysalicylaldehyde (98%) were purchased from Alfa Aesar and used without further purification. The other solvents and reagents were analytical grade reagents.

C, H and N analyses were performed via a GmbH VarioEL V3.00 automatic elemental analyzer. Elemental analyses for metal(II) were conducted using an IRIS ER/S·WP⁻¹ ICP atomic emission spectrometer.

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https://doi.org/10.1016/j.ica.2019.119238

Received 29 September 2019; Received in revised form 29 October 2019; Accepted 29 October 2019 Available online 31 October 2019

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Scheme 1. Synthetic route to the ligand precursor H₃L.

¹H NMR spectra were recorded using a Bruker AVANCE DRX-400 spectrometer. Melting points were determined via a microscopic melting point instrument made in Beijing Tektronix Instruments Limited Company. FT-IR spectra were gained via a VERTEX70 FT-IR spectrophotometer, with samples prepared as CsI (500–100 cm⁻¹) and KBr (4000–500 cm⁻¹) pellets. UV/Vis absorption spectra were obtained via a Shimadzu UV-2550 spectrometer. X-ray single crystal structures were determined on a SuperNova, Dual Eos four-circle diffractometer. Fluorescence spectra were gained via a F-7000 FL spectrophotometer.

2.2. Preparation of the ligand H_{3L}

The major reaction steps involved in the synthesis of H_3L are given in Scheme 1. 1,2-Bis(aminooxy)ethane was synthesized following the literature [37].

2-[*O*-(1-Ethyloxyamide)]oxime-6-methoxyphenol was synthesized via the reaction of 3-methoxysalicylaldehyde (304.3 mg, 2.0 mmol) in methanolic solution (30 mL) with 1,2-bis(aminooxy)ethane (92.3 mg, 1.0 mmol) in methanolic solution (20 mL). The solution was stirred at ca. 55 °C for about 12 h, then the solution was concentrated under reduced pressure and the residue was purified by column chromatography (SiO₂, chloroform/ethyl acetate, 30:1) to afford a colorless flocculent crystalline solid. Yield: 65%. *Anal. Calc.* for $C_{10}H_{14}N_2O_4$: C, 53.09; H, 6.24; N, 12.38. Found: C, 52.95; H, 6.20; N, 12.29%.

The ligand precursor H₃L: 6-Hydroxy-6'-methoxy-2, 2'-[ethylenediyldioxybis(nitrilomethylidyne)]diphenol was prepared by the reaction of 2-[O-(1-ethyloxyamide)]oxime-6-methoxyphenol (452.4 mg, 2.0 mmol) in ethanol (20 mL) with 3-hydroxysalicylic aldehyde (276.3 mg. 3.0 mmol) in ethanol (20 mL). The mixture was subjected to heating at 65 °C for 4 h, then the resulting pale yellow solid was collected. Yield: 78%. *Anal. Calc.* for C₁₇H₁₈N₂O₆: C, 58.96; H, 5.24; N, 8.09. Found: C, 58.87; H, 5.28; N, 8.02%. ¹H NMR (400 MHz, CDCl₃) δ 9.92 (s, 1H), 9.70 (s, 1H), 8.24 (d, J = 13.3 Hz, 2H), 7.03–6.66 (m, 6H), 5.60 (s, 1H), 4.49 (s, 3H), 3.91 (s, 4H).

2.3. Syntheses of the Co(II) and Ni(II) complexes

To a methanolic solution (4 mL) of cobalt(II) acetate dihydrate (7.96 mg, 0.04 mmol), was added dropwise an acetone solution (4 mL) of H₃L (6.92 mg, 0.02 mmol), and the mixed solution color changed to dark-brown immediately. The mixed solution was filtered and the filtrate was allowed to stand for about four weeks. After partial solvent evaporation, single crystals suitable for X-ray diffraction analysis were obtained. Yield, 56%. *Anal. Calc.* for $[{Co(L)(\mu-OAc)Co(CH_3OH)_2}_2] (C_{42}H_{52}Co_4N_4O_{20})$: C, 43.17; H, 4.49; N, 4.79; Co, 20.17. Found: C, 43.18; H, 4.47; N, 4.76; Co, 20.18%.

To a methanolic solution (4 mL) of nickel(II) acetate dihydrate (8.76 mg, 0.04 mmol) was added dropwise a dichloromethane/DMF (v/v, 10:1) solution (4 mL) of H₃L (6.92 mg, 0.02 mmol), and the mixed solution color changed to yellow immediately. The mixed solution was filtered and the filtrate was allowed to stand for about one week. After partial solvent evaporation, single crystals suitable for X-ray diffraction analysis were obtained. Yield: 49%. *Anal. Calc.* for [{Ni(L)(DMF)(μ -OAc)Ni}₂] (C₄₄H₄₉N₆Ni₄O₁₈): C, 44.65; H, 4.09; N, 7.10; Ni, 19.83. Found: C, 44.67; H, 4.07; N, 7.06; Ni, 19.84%.

2.4. X-ray structure determination of the Co(II) and Ni(II) complexes

The crystal diffractometer with a monochromatic beam of Mo Kα radiation (0.071073 nm) produced using a graphite monochromator from a sealed Mo X-ray tube was used to obtain the crystal data of the Co(II) and Ni(II) complexes at 293.01(2) and 293.42(10) K, respectively. The LP factor semi-empirical absorption corrections were applied using the SADABS program. The structures were solved by direct methods (SHELXS-2014) [38]. All hydrogen atoms were added theoretically and difference-Fourier map revealed the positions of the remaining atoms. All non-hydrogen atoms were refined anisotropically via a full-matrix least-squares procedure on F^2 with SHELXL-2014 [39]. The crystal data and experimental parameters relevant to the structure determinations are listed in Table 1.

3. Results and discussion

3.1. IR spectra analyses

The FT-IR spectra of H₃L and the Co(II) and Ni(II) complexes demonstrate various absorption bands in the 400–4000 cm⁻¹ region (Table 2). The free ligand H₃L shows a broad characteristic band of the OH group at 3436 cm⁻¹. This band is weakened in the IR spectra of the Co(II) and Ni(II) complexes, which is indicative of the fact that the phenolic OH groups of H₃L have been deprotonized and coordinated to

Table 1

Crystal and refine	nement parameter	data for	the Co(II)	and Ni(II)	complexes.
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Complex	Co(II)	Ni(II)
Empirical formula	$C_{42}H_{52}Co_4N_4O_{20}$	C44H48N6Ni4O18
Molecular weight	584.30	101.74
Color	light-brown	clear light green block
Crystal size (mm)	0.21 imes 0.15 imes 0.14	0.22 imes 0.18 imes 0.17
Habit	Block-shaped	Block-shaped
Crystal system	Triclinic	Monoclinic
Space group	P - 1	<i>P</i> 1 2 ₁ /n 1
Unit cell dimension		
a (Å)	9.9509(4)	13.3288(2)
b (Å)	10.7756(6)	13.4470(3)
c (Å)	12.2089(7)	14.5060(3)
α (°)	75.073(5)	90
β (°)	86.887(4)	112.3730(10)
γ(°)	68.260(5)	90
V (Å ³)	1173.74(11)	2404.23(8)
Ζ	2	30
$D_{\text{calc}} (\mathbf{g} \cdot \mathbf{cm}^{-3})$	1.653	2.108
$\mu ({\rm mm^{-1}})$	1.473	5.783
F(0 0 0)	600	1500
θ range for data collection (°)	3.46 to 26.01	3.030 to 27.123
Index ranges	$-12 \le h \le 12$,	$-17 \le h \le 17$,
	$-13 \le k \le 12$,	$-17 \le k \le 17,$
	$-15 \le l \le 15$	$-18 \le l \le 18$
Reflections collected	7721	46,544
Completeness to (%) (θ)	99.79 (26.01)	99.6 (25.242)
Data/restraints/parameters	4616/6/326	5298/0/329
Final R_1 , w R_2 indices	0.0889, 0.0812	0.0361, 0.0999
R_1 , w R_2 indices (all data)	0.0484, 0.0889	0.0515, 0.1091
Largest diff. peak and hole $(e \dot{A}^{-3})$	0.558, -0.440	1.038, -0.464

Major IR spectroscopic bands of $H_{3}L$ and its Co(II) and Ni(II) complexes (cm⁻¹).



Fig. 1. UV-vis spectra of H₃L and its Co(II) and Ni(II) complexes.



Fig. 2. (a) Molecular structure of the Co(II) complex (hydrogen atoms are omitted for clarity). (b) Coordination polyhedra for the Co(II) ions of the Co(II) complex.

the Co(II) and Ni(II) atoms [40,41]. For the Co(II) and Ni(II) complexes, these bands weakened are expected due to the substitution of hydrogen atoms for the Co(II) and Ni(II) atoms upon coordination [28,42]. Besides, an absorption band at 3416 cm⁻¹ in the Co(II) complex could be ascribed to the –OH group of coordinated methanol molecules [30]. A characteristic strong C=N stretching band of H₃L emerges at 1613 cm⁻¹, while those of the Co(II) and Ni(II) complexes emerge at 1596 and 1606 cm⁻¹, respectively [43]. The C=N stretching frequencies are shifted to lower frequencies by *ca*. 17 and 7 cm⁻¹ upon complexation, indicating that the Co(II) and Ni(II) ions are coordinated by the N₂O₄ donor atoms of the ligand (L)³⁻ units. Thus, it provides evidence for the coordination of deprotonated H₃L to the Co(II) and Ni



Fig. 3. View of the intramolecular hydrogen bonds of the Co(II) complex unit (for clarity purpose, hydrogen atoms are omitted except those forming hydrogen bonds).

(II) ions. The free ligand precursor H_3L also exhibits an Ar–O stretching frequency at 1253 cm⁻¹, while those of the Co(II) and Ni(II) complexes appear at 1208 and 1245 cm⁻¹, respectively [44]. The Ar–O stretching frequencies of the phenoxy groups are shifted to low frequencies, which could be evidence of the Co–O or Ni–O bond formation between the Co (II) or Ni(II) ions and the oxygen atoms of the phenoxy groups [45].

The far-IR spectra (500–100 cm⁻¹) of the Co(II) and Ni(II) complexes were obtained to identify the M-O and M-N stretching frequencies. The $\nu_{\rm M(II)-O}$ bands at 423 and 421 cm⁻¹ in the Co(II) and Ni (II) complexes can be assigned to $\nu_{\rm Co-O}$ and $\nu_{\rm Ni-O}$, while the $\nu_{\rm M(II)-N}$ bands at 511 and 539 cm⁻¹ are attributed to $\nu_{\rm Co-N}$ and $\nu_{\rm Ni-N}$, respectively [46].

3.2. UV-Vis absorption spectral analyses

The UV–Vis absorption spectra of H₃L, Co(II) and Ni(II) complexes in ethanolic solution $(1.0 \times 10^{-5} \text{ mol/L})$ at 298 K are presented in Fig. 1.

It can be seen that the absorption peaks of the Co(II) and Ni(II) complexes are obviously different from those of H₃L. The absorption spectrum of H₃L includes two relatively intense peaks centered at 271 and 319 nm. The former absorption peak at *ca*. 271 nm can be assigned to the π - π * transitions of the phenyl rings, and latter absorption peak at *ca*. 319 nm can be assigned to the intra-ligand π - π * transition of the oxime group. Upon coordination of the ligand, the π - π * transitions of the phenyl rings in the Co(II) and Ni(II) complexes are bath-ochromically shifted to 278 and 280 nm, respectively, which indicates coordination of the ligand precursor H₃L, a new weak broad absorption peak emerges at *ca*. 371 nm in the UV–Vis spectrum of the Ni (II) complex, which can be assigned to a π - π * charge transfer transition from the filled p-orbital of the bridging phenoxo oxygen atoms to the vacant d-orbital of the Ni(II) ions [47].

3.3. Description of the crystal structure of the Co(II) and Ni(II) complexes

X-ray crystallographic analyses reveal that the Co(II) and Ni(II) complexes form two different crystal structures, which are different compared to common tetranuclear structures of salamo-like complexes reported earlier [48–50]. The crystal structures of the Co(II) and Ni(II) complexes and the coordination polyhedra of the Co(II) and Ni(II) ions are shown in Figs. 2 and 5. Selected bond lengths and angles are summarized in Tables S1 and S2.

3.3.1. Crystal structure of the Co(II) complex

As presented in Fig. 2, the Co(II) complex includes four Co(II) ions, two fully deprotonated $(L)^{3-}$ moieties, four coordinated MeOH molecules and two μ -acetato ligands. The Co(II) complex possesses a highly symmetrical tetranuclear structure. The penta-coordinated terminal Co (II) ion (Co1) lies in the N₂O₂ coordination sphere (N1, N2, O1 and O5)



Fig. 4. View of the intermolecular hydrogen bonding interactions of the Co(II) complex.

 Table 3

 Hydrogen bond parameters for the Co(II) complex.

D–X···A	d(D–X)	d(X…A)	d(D…A)	∠D–X…A	Symmetry code
09–H9…01 09–H9…02 010–H10…09 C8–H8A…07 C8–H8B…010 C9–H9A…04	0.87 0.87 0.87 0.97 0.97 0.97	1.84 2.43 1.97 2.41 2.34 2.60	2.625(3) 3.132(3) 2.772(3) 3.245(4) 3.172(4) 3.405(4)	149 137 153 144 144 141	1-x, 2-y, 1-z 1-x, 1-y, 1-z 1-x, 1-y, -z



Fig. 5. (a) Molecular structure of the Ni(II) complex (hydrogen atoms are omitted for clarity). (b) Coordination polyhedra for the Ni(II) ions of the Ni(II) complex.

 Table 4

 Hydrogen bond parameters for the Ni(II) complex.

D–X…A	d(D–X)	d(X…A)	d(D…A)	∠D–X…A	Symmetry code
C6–H8A…N2	0.97	2.59	2.937(5)	101	1 - x, 1 - y, 1 - z
C20–H20…O1	0.93	2.50	2.853(5)	103	
C22–H20…O5	0.93	2.54	3.078(5)	117	
C20–H20…O2	0.93	2.37	3.2795)	166	



Fig. 6. View of the intramolecular hydrogen bonds of the Ni(II) complex unit (for clarity purpose, hydrogen atoms are omitted except those forming hydrogen bonds).

of the fully deprotonated $(L)^{3-}$ unit, and is coordinated further to one oxygen atom (O7) from the μ -acetato ligand, adopting a twisted triangular bipyramidal geometry which is deduced by calculating the value of $\tau = 0.693$. Unlike the terminal Co(II) ion, the hexa-coordinated central Co(II) ion (Co2) is surrounded by three oxygen atoms (O6, O6 and O5) from the fully deprotonated $(L)^{3-}$ moiety, one oxygen atom (O8) of the μ -acetato ligand and two oxygen atoms (O9 and O10) from two coordinated methanol molecules adopting a twisted octahedral geometry.

There are four pairs of intramolecular hydrogen bonding interactions (O9-H9...O1, O9-H9...O2, O10-H10...O9 and C8-H8A...O7) and two pairs of intermolecular hydrogen bonding interactions (O8-H8B...O10 and C9-H9A...O4) [51] (Figs. 3 and 4). The intermolecular and intramolecular hydrogen bonding interactions are summarized in Table 3. As shown in Fig. 4, the supramolecular structure of the Co(II)



Fig. 7. Emission spectra of $H_{3}L$ (c = 1 \times 10 $^{-5}$ M, λex = 370 nm) and its Co(II) and Ni(II) complexes.

complex is linked further by intermolecular hydrogen bonding interactions (O8-H8B···O10 and C9-H9A···O4), which play a crucial role in constructing and stabilizing the 2D supramolecular chain structure.

3.3.2. Crystal structure of the Ni(II) complex

As depicted in Fig. 5, the Ni(II) complex also has a symmetrical tetranuclear structure. In the crystal structure of the Ni(II) complex, the hexa-coordinated terminal Ni(II) ion (Ni1) lies in the N₂O₂ coordination sphere (N1, N2, O1 and O5) of the fully deprotonated (L)^{3–} moiety, and is coordinated further to one μ -acetato oxygen atom (O7) and one oxygen atom (O9) from the coordinated DMF molecule. The Ni(II) ion (Ni1) adopts a slightly twisted octahedral geometry. Unlike the Ni(II) ion (Ni1), the hexa-coordinated central Ni(II) ion (Ni2) is coordinated to the four oxygen atoms (O1, O2, O5 and O6) from a fully deprotonated (L)^{3–} moiety, the oxygen atoms (O2^{#2}) from another fully deprotonated (L)^{3–} moiety and one μ -acetato oxygen atom (O8), and adopts a slightly twisted octahedral geometry.

As illustrated in Table 4 and Fig. 6, there are only four pairs of intramolecular hydrogen bonding interactions (C(8)-H(8A)…N2, C(20)-H(20)…O1, C(20)-H(20)…O5 and C(20)-H(20)…O2) [52].

3.4. Fluorescence properties

The fluorescence spectra of H₃L, and the Co(II) and Ni(II) complexes were recorded at room temperature and are depicted in Fig. 7. The ligand precursor H₃L exhibits a relatively strong emission peak at *ca*. 400 nm upon excitation at 370 nm, and it could be attributed to the intra-ligand π – π * transition. The Co(II) and Ni(II) complexes exhibit lower photoluminescence with maximum emission at *ca*. 393 and 397 nm when excited with 370 nm, respectively, and the absorption peaks are hypsochromically shifted, which could be assigned to ligandto-metal charge transfer (LMCT) [8]. Compared with the ligand precursor H₃L, emission intensities of the Co(II) and Ni(II) complexes are obviously reduced, indicating that the Co(II) and Ni(II) ions are involved in fluorescence quenching.

3.5. Hirshfeld surfaces analyses

The Hirshfeld surfaces [53] of the Co(II) and Ni(II) complexes are illustrated in Fig. 8, showing surfaces that have been mapped over d_{norm} . The corresponding location in shape index exists the complementary region of the red concave surface surrounded by receptors and the blue convex surface surrounding receptors, further confirming the hydrogen bonding. As for the large amount of white region in the d_{norm} surfaces, it is suggested that there is a weaker and farther contact between molecules, rather than hydrogen bonds. Fig. 9 shows the 2D plots generated [54] corresponding to the O…H, C…H and H…H interactions from the Hirshfeld surfaces of the Co(II) and Ni(II) complexes.

As depicted in Fig. 9a for the Co(II) complex, the H···H interactions appearing at (1.15 Å, 1.15 Å) account for 53.6% of the total area of Hirshfeld surfaces. The C···H/H···C interactions in the range of (1.60 Å, 0.90 Å) appear as a pair of symmetrical wings, accounting for 12% of the total area of Hirshfeld surfaces. The proportions of O···H/H···O interactions comprise 11.1% of the total Hirshfed surfaces for each molecule of the Co(II) complex. As depicted in Fig. 9b for the Ni(II) complex, the interactions of H···H appearing at (1.15 Å, 1.15 Å) account for 56% of the total area of Hirshfeld surfaces. The C···H/H···C interactions in the range of (1.60 Å, 1.05 Å) account for 10.6% of the total area of Hirshfeld surfaces. The proportions of O···H/H···C interactions in the range of (1.60 Å, 1.05 Å) account for 10.6% of the total area of Hirshfeld surfaces for each molecule of the Ni(II) complex. It is because of the existence of these weaker hydrogen bonds that the Co(II) and Ni(II) complexes can be stable.



Fig. 8. Hirshfeld surfaces analyses mapped with curvedness, *d_{norm}* and shape index of the Co(II) and Ni(II) complexes.



Fig. 9. Fingerprint plots of the Co(II) complex (a) and Ni(II) complex (b): full and resolved into full and resolved into O...H, C...H and H...H contacts showing the percentages of contacts contributed to the total Hirshfeld surface area of molecule.

4. Conclusion

In summary, we have reported the successful syntheses and characterization of two newly designed tetranuclear metal(II) complexes $[{Co(L)(\mu-OAc)Co(CH_3OH)_2}_2]$ and $[{Ni(L)(DMF)(\mu-OAc)Ni}_2]$ derived from an unsymmetrical Salamo-like ligand precursor H₃L. The pentacoordinated Co(II) ion (Co1) in the Co(II) complex adopts slightly twisted triangular bipyramidal configuration, while the hexa-coordinated Co(II) ion (Co2) is in a slightly twisted octahedral configuration. The supramolecular structure of the Co(II) complex is formed by intermolecular hydrogen bonding interactions, resulting in a selfassembled infinite 2D supramolecular network. The hexa-coordinate Ni (II) ions in the Ni(II) complex adopt a slightly twisted octahedral configuration. Meanwhile, the Co(II) and Ni(II) complexes exhibit weak fluorescence when excited with 370 nm, and the maximum emission wavelengths at 393 and 397 nm, respectively.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (21761018), Special Funds for Discipline Construction of Gansu Agricultural University (LXYXK201801) and the Program for Excellent Team of Scientific Research in Lanzhou Jiaotong University (201706), which are gratefully acknowledged.

Appendix A. Supplementary data

CCDC-1952219 and 1952218 contains the supplementary crystallographic data for the Co(II) and Ni(II) complexes. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (Telephone: +44-01223-762910; Fax: +44-1223-336033; or E-mail: deposit@ccdc.cam.ac.uk). Supplementary data to this article can be found online at https://doi. org/10.1016/j.ica.2019.119238.

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