Synthesis and crystal structures of three isophthalato-bridged macrocyclic nickel(II) complexes

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Abstract. Three dinuclear isophthalato-bridged nickel(II) complexes formulated as $[Ni(rac-L)]_2(\mu-IPA)(ClO_4)_2$ (1), $[Ni(RR-L)]_2(\mu-IPA)(ClO_4)_2$ (2) and $[Ni(SS-L)]_2(\mu-IPA)(ClO_4)_2$ (3) (L = 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyc-lotetradecane, IPA = isophthalic acid) have been isolated and characterized. Single crystal X-ray diffraction analyses revealed that the Ni(II) atoms have six-coordinated distorted octahedral environments, and the isophthalato ligand bridges two Ni(II) centres in a bis bidentate fashion to form dimers in all three complexes. The monomers of { $[Ni(SS-L)]_2(\mu-IPA)$ }²⁺ are connected through intermolecular hydrogen bonds to generate one-dimensional left-handed helical chains in complex 3. The homochiral natures of complexes 2 and 3 have been confirmed by CD spectroscopy.

Keywords. Isophthalato-bridged; macrocyclic nickel(II) complexes; crystal structure; circular dichroism spectra.

1. Introduction

The design and synthesis of extended bridged polymetallic complexes continue to be the focus of much attention owing to their potential applications in magneto- and materials chemistry.¹⁻⁷ Many extended bridged dinuclear complexes such as μ terephthalate,^{8–11} μ -oxalate,^{12–17} μ -N₃,^{18–21} μ -butene dicarboxylate²² and μ - succinate²³ have been synthesized. In our previous report,^{11,23} we used rigid terephthalic acid and flexible succinic acid ligand bridging macrocyclic nickel(II) complexes to construct one-dimensional hydrogen bonded helical chains. In continuation of our research on the constructions of chiral helical chains, we employ [Ni $(rac-L)^{2+}/[Ni(RR-L)]^{2+}/[Ni(SS-L)]^{2+}$ (L = 5,5,7,12, 12,14-hexamethyl-1,4,-8,11-tetraazacyclotetradecane) (scheme 1) and angular isophthalic acid ligand as building blocks to construct supramolecular isomers, and one-dimensional left-handed helical chains of $\{[Ni(SS-L)]_2(\mu-IPA)\}^{2+}$ are formed through intermolecular hydrogen bonds in $[Ni(SS-L)]_2(\mu$ -IPA) $(ClO_4)_2$.

2. Experimental

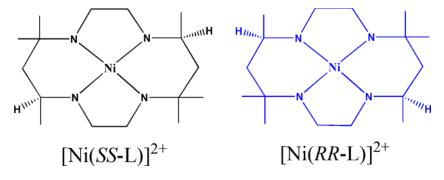
2.1 Materials and methods

The macrocyclic ligand (L) and its nickel(II) complex were prepared according to the literature method^{24,25} and separated as the racemic form of *rac*-L. The nickel(II) complex $[Ni(rac-L)](ClO_4)_2$ was prepared according to the previously reported methods.²⁶ All other chemicals were commercially sourced and used without further purification. Elemental analyses were determined using an Elementar Vario EL elemental analyser. IR spectra were recorded in the 4000 to 400 cm⁻¹ region using KBr pellets and a Bruker EQUINOX 55 spectrometer. The solid (KBr pellets) and solution circular dichroism (CD) spectra were recorded on a JASCO J-810 spectropolarimeter.

2.2 X-ray structure determination

Single-crystal data for 1–3 were collected on a Bruker Smart 1000 CCD diffractometer, with Mo-K α radiation ($\lambda = 0.71073$ Å). Empirical absorption corrections were applied by using the SADABS program.²⁷ The structures were solved using direct methods, which yielded the positions of all non-hydrogen atoms. These were refined first isotropically and then anisotropically.

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Scheme 1. Coordination geometries of $[Ni(SS-L)]^{2+}$ and $[Ni(RR-L)]^{2+}$.

All the hydrogen atoms of the ligands were placed in calculated positions with fixed isotropic thermal parameters and included in the structure factor calculations in the final stage of full-matrix least-squares refinement. All calculations were performed using the SHELXTL suite of computer programs.²⁸ The crystallographic data for complexes 1–3 are summarized in table 1. Selected bond lengths and angles are listed in table 2. The hydrogen bond parameters for complexes 1 and 3 are given in table 3.

acid (IPA, 0.16 g, 1 mmol) and NaOH (0.08 g, 2 mmol) in water. The resulting blue solution was evaporated slowly at room temperature to give blue crystals of complex **1**. Yield: 0.35 g (32%). Elemental Anal. Found: C, 43.41; H, 7.36; N, 10.25%. Calcd for $C_{40}H_{82}N_8Cl_2O_{15}Ni_2$: C, 43.54; H, 7.49; N, 10.16%. IR (KBr): 3416, 3262, 2968, 1612, 1533, 1416, 1283, 1088, 964, 820, 729 and 625 cm⁻¹.

2.4 Synthesis of $[Ni(RR-L)]_2$ (μ -IPA)(ClO₄)₂(2)

2.3 Synthesis of $[Ni(rac-L)]_2(\mu-IPA)(ClO_4)_2(1)$

An acetonitrile solution (5 mL) of $[\text{Ni}(rac-\text{L})](\text{ClO}_4)_2$ (0.54 g, 1 mmol) was added to a solution of isophthalic Use of $[Ni(RR-L)](CIO_4)_2$ in a procedure analogous to that detailed for the preparation of complex **1** afforded 0.28 g (26%) of blue-violet prism-shaped crystals suitable for X-ray analysis. Elemental Anal.

Table 1. Crystal data and structure refinement.

Compound	1	2	3
Empirical formula	C40H82Cl2N8Ni2O15	C ₄₀ H ₇₈ Cl ₂ N ₈ Ni ₂ O ₁₃	C ₄₀ H ₈₂ Cl ₂ N ₈ Ni ₂ O ₁₅
Formula weight	1103.46	1067.42	1103.46
Temperature (K)	296(2)	173(2)	173(2)
Crystal system	Monoclinic	Orthorhombic	Orthorhombic
Space group	P2(1)/c	P2(1)2(1)2(1)	P2(1)2(1)2(1)
a/ Å	15.198(2)	14.717(2)	15.038(4)
<i>b</i> / Å	16.132(2)	16.851(3)	16.807(4)
c/ Å	21.625(3)	21.227(3)	21.097(5)
β / °	97.749(2)	90	90
$V / Å^3$	5253.2(13)	5264.3(15)	5332(2)
Ζ	4	4	4
$D_{\rm c}$ / g· cm ⁻³	1.395	1.347	1.375
$\mu/\text{ mm}^{-1}$	0.887	0.880	0.874
F(000)	2352	2272	2352
Crystal size (mm)	$0.42 \times 0.38 \times 0.21$	$0.46 \times 0.41 \times 0.21$	$0.47 \times 0.42 \times 0.20$
θ range for data collection	1.35-27.53	1.54-27.10	1.55-27.05
Reflections collected/unique	43469/11857(0.0577)	27136/11584(0.0774)	15581/10715(0.0401)
Completeness to θ	98.1	99.7	97.2
Goodness-of-fit on F^2	1.040	1.038	1.023
Final <i>R</i> indices $[I > 2\sigma(I)]$	0.0507, 0.1289	0.0708, 0.1670	0.0537, 0.1012
<i>R</i> indices (all data)	0.0985, 0.1690	0.1863, 0.2098	0.1068, 0.21182
Absolute structure parameter		-0.04	-0.032
Max. peak/hole (e. $Å^{-3}$)	0.490/-0.743	0.632/-0.471	0.649/-0.434

		1			
Ni(1)–N(1)	2.139(3)	Ni(1)–N(2)	2.082(3)	Ni(1)–N(3)	2.139(3)
Ni(1) - N(4)	2.096(3)	Ni(2)–N(5)	2.113(3)	Ni(2)–N(6)	2.092(3)
Ni(2)–N(7)	2.135(3)	Ni(2)–N(8)	2.096(3)	Ni(1) - O(1)	2.122(2)
Ni(1)–O(2)	2.151(3)	Ni(2)–O(3)	2.115(2)	Ni(2) - O(4)	2.187(2)
N(2)–Ni(1)–N(4)	104.96(11)	N(4)-Ni(1)-N(1)	91.60(11)	N(4)-Ni(1)-O(1)	97.32(10)
N(2)-Ni(1)-N(1)	85.47(11)	O(1)-Ni(1)-N(1)	98.13(10)	N(3)-Ni(1)-O(2)	98.16(10)
N(2)-Ni(1)-N(3)	92.04(11)	N(3)-Ni(1)-N(1)	175.19(11)	N(1)-Ni(1)-O(2)	86.22(10)
N(2)-Ni(1)-O(1)	157.34(11)	N(2)-Ni(1)-O(2)	96.14(10)	N(6)-Ni(2)-N(8)	102.34(11)
N(4) - Ni(1) - N(3)	85.05(11)	N(4)-Ni(1)-O(2)	158.56(10)	N(6)-Ni(2)-N(5)	85.51(11)
O(1)-Ni(1)-N(3)	85.75(10)	O(1)-Ni(1)-O(2)	62.01(9)	N(8)-Ni(2)-N(5)	92.14(11)
N(6) - Ni(2) - N(7)	92.50(11)	N(6) - Ni(2) - O(4)	155.72(10)	N(6) - Ni(2) - O(3)	95.85(10)
N(8) - Ni(2) - N(7)	85.03(10)	N(5)-Ni(2)-O(4)	100.73(11)	N(8) - Ni(2) - O(3)	161.75(10)
N(5)-Ni(2)-N(7)	176.13(11)	O(3) - Ni(2) - O(4)	61.31(9)	N(5)-Ni(2)-O(3)	87.88(11)
O(3)–Ni(2)–N(7)	95.64(10)	N(7)-Ni(2)-O(4)	82.42(10)	N(8)–Ni(2)–O(4)	100.86(10)
NT (4) NT (4)	2 122/0	2			2 100 (C)
Ni(1)-N(1)	2.133(6)	Ni(1)-N(2)	2.078(5)	Ni(1) - N(3)	2.109(6)
Ni(1)–N(4)	2.106(6)	Ni(1)–O(1)	2.106(5)	Ni(1)–O(2)	2.220(5)
Ni(2)-N(5)	2.160(7)	Ni(2)-N(6)	2.053(7)	Ni(2)-N(7)	2.121(9)
Ni(2)–N(8)	2.089(6)	Ni(2)–O(3)	2.097(5)	Ni(2)–O(4)	2.165(5)
N(2)–Ni(1)–O(1)	161.0(2)	O(1)-Ni(1)-N(1)	98.67(19)	N(1)–Ni(1)–O(2)	83.4(2)
N(2)-Ni(1)-N(4)	103.0(2)	N(4)-Ni(1)-N(1)	90.6(2)	N(6)-Ni(2)-N(8)	103.4(3)
O(1)-Ni(1)-N(4)	95.6(2)	N(3)-Ni(1)-N(1)	174.6(2)	N(6)–Ni(2)–O(3)	157.3(2)
N(2)-Ni(1)-N(3)	91.3(2)	N(2)-Ni(1)-O(2)	101.1(2)	N(8)–Ni(2)–O(3)	99.2(2)
O(1) - Ni(1) - N(3)	85.9(2)	O(1)-Ni(1)-O(2)	61.26(17)	N(6)-Ni(2)-N(7)	91.4(4)
N(4) - Ni(1) - N(3)	86.1(2)	N(4)-Ni(1)-O(2)	154.6(2)	N(8)-Ni(2)-N(7)	86.2(3)
N(2)-Ni(1)-N(1)	85.3(2)	N(3)-Ni(1)-O(2)	101.3(2)	O(3) - Ni(2) - N(7)	88.4(3)
N(6)–Ni(2)–N(5)	86.0(3)	N(6)-Ni(2)-O(4)	96.6(3)	N(7)-Ni(2)-O(4)	101.4(3)
N(8) - Ni(2) - N(5)	89.8(3)	N(8) - Ni(2) - O(4)	158.5(2)	N(5)-Ni(2)-O(4)	83.7(2)
O(3) - Ni(2) - N(5)	95.9(2)	O(3)–Ni(2)–O(4)	61.3(2)	N(7)-Ni(2)-N(5)	174.5(3)
N(1)–Ni(1)	2.112(4)	3 N(2)–Ni(1)	2.067(4)	N(3)–Ni(1)	2.125(4)
N(4) - Ni(1)	2.094(4)	N(2) = N(1) N(5) - Ni(2)	2.126(5)	N(6) - Ni(2)	2.094(4)
N(7) - Ni(2)	2.152(4)	N(8) - Ni(2)	2.089(4)	N(0)=N(2) Ni(1)=O(1)	2.094(4) 2.112(3)
N(1) - N(2) Ni(1) - O(2)	2.132(4) 2.181(3)	N(0)=N(2) Ni(2)=O(4)	2.117(3)	Ni(2) - O(3)	2.112(3) 2.189(3)
N(1)=O(2) N(2)=Ni(1)=N(4)	102.86(16)	N(2) - N(1) - N(3)	90.88(15)	N(1)-Ni(1)-O(2)	85.91(14)
N(2)-Ni(1)-O(1)	158.41(14)	N(2)=N(1)=N(3) N(4)=Ni(1)=N(3)	85.48(16)	N(1) = N(1) = O(2) N(3) = Ni(1) = O(2)	100.38(14)
N(2) = Ni(1) = O(1) N(4) = Ni(1) = O(1)	98.16(13)	N(4) - N(1) - N(3) N(1)-Ni(1)-N(3)	172.85(16)	N(3) - Ni(1) - O(2) N(8) - Ni(2) - N(6)	100.38(14)
N(2)-Ni(1)-N(1)	84.77(15)	N(1) = N(1) = N(3) N(2) = Ni(1) = O(2)	98.58(14)	N(8)-Ni(2)-O(4)	158.30(15)
	89.95(16)		157.69(14)		97.82(15)
N(4)-Ni(1)-N(1) N(8) $Ni(2)$ $N(5)$	91.81(19)	N(4)-Ni(1)-O(2) N(8) $Ni(2)$ $N(7)$	84.91(18)	N(6)-Ni(2)-O(4) N(5) $Ni(2)$ $N(7)$	173.69(16)
N(8)–Ni(2)–N(5) N(6)–Ni(2)–N(5)	85.32(16)	N(8)–Ni(2)–N(7) N(6)–Ni(2)–N(7)	90.23(16)	N(5)–Ni(2)–N(7) N(8)–Ni(2)–O(3)	97.65(15)
N(5)-Ni(2)-O(3)	100.07(15)	N(6) - Ni(2) - O(3)	157.72(15)	N(3) - N(2) - O(3) N(7) - Ni(2) - O(3)	85.73(15)
O(1)-Ni(1)-N(1)	100.07(13) 100.25(14)	O(1)-Ni(1)-O(2)	61.23(11)	O(4)-Ni(2)-O(3)	61.04(13)
O(1) - Ni(1) - N(1) O(1) - Ni(1) - N(3)	85.87(13)	O(1) = Ni(1) = O(2) O(4) = Ni(2) = N(5)	88.39(16)	O(4) - Ni(2) - O(3) O(4) - Ni(2) - N(7)	96.65(15)
O(1) - IV(1) - IV(3)	05.07(15)	$O(+)^{-1}(2)^{-1}(3)$	00.39(10)	$O(+)^{-1}(2)^{-1}(7)$	90.05(15)

Table 2. Selected bond lengths (Å) and bond angles (°).

Found: C, 45.29; H, 7.45; N, 10.39%. Calcd for $C_{40}H_{78}N_8Cl_2O_{13}Ni_2$: C, 45.01; H, 7.36; N, 10.50%. IR (KBr): 3441, 3250, 2970, 1605, 1541, 1408, 1273, 1090, 962, 727 and 627 cm⁻¹.

2.5 Synthesis of $[Ni(SS-L)]_2(\mu$ -IPA)(ClO₄)₂ (3)

Use of $[Ni(SS-L)](ClO_4)_2$ in a procedure analogous to that detailed for the preparation of complex **1** afforded 0.32 g (29%) of blue-violet prism-shaped crystals suitable for X-ray analysis. Elemental Anal.

Found: C, 43.32; H, 7.23; N, 10.27%. Calcd for $C_{40}H_{82}N_8Cl_2O_{15}Ni_2$: C, 43.54; H, 7.49; N, 10.16%. IR (KBr): 3433, 3255, 2972, 1608, 1570, 1406, 1270, 1092, 974, 748 and 625 cm⁻¹.

3. Results and Discussion

3.1 Crystal structure description

Single crystal X-ray structural analysis displays that all three compounds show similar structures,

		1		
D-HA	d(D–H)	d(HA)	d(DA)	<(DHA)
N(6)-H(6A)O(3W)	0.91	2.10	2.991(4)	167.3
O(1W)–H(1WA)O(2W) ^{#1}	0.858(10)	1.99(3)	2.783(5)	153(5)
O(1W)–H(1WB)O(2) ^{#2}	0.856(9)	2.099(10)	2.896(4)	155(3)
O(2W)-H(2WB)O(7) ^{#3}	0.861(10)	2.21(3)	3.022(7)	157(7)
O(2W)-H(2WA)O(4)#4	0.863(10)	1.948(17)	2.798(4)	168(6)
O(3W)–H(3WA)O(1W)	0.868(10)	1.850(17)	2.701(5)	166(5)
O(3W)–H(3WB)O(5) ^{#5}	0.868(10)	2.184(18)	3.026(7)	163(5)
		3		
D-HA	d(D–H)	d(HA)	d(DA)	<(DHA)
O(1W)-H(1E)O(2W)	0.852(10)	1.93(2)	2.758(6)	163(7)
O(1W)–H(1D)O(3) ^{#6}	0.854(10)	1.999(19)	2.839(6)	168(6)
O(2W)-H(2D)O(2) ^{#6}	0.849(10)	1.937(15)	2.776(5)	169(6)

Table 3. Hydrogen bond parameters (Å, °) for complexes 1 and 3.

Symmetry codes: #1 -*x*+1, *y*-1/2, -*z*+1/2; #2 *x*, *y*-1, *z*; #3 -*x*, *y*+1/2, -*z*+1/2; #4 *x*+1, *y*, *z*; #5 *x*, -*y*+1/2, *z*+1/2; #6 *x*+1, *y*, *z*.

which contain one $[(NiL)_2(\mu-IPA)]^{2+}$ and two CIO_4^- (figures 1, 3 and 4). Two $[NiL]^{2+}$ units are bridged by one IPA^{2-} anion to form the dimeric cation in $[(NiL)_2(\mu-IPA)]^{2+}$. In the dimer, the two Ni(II) atoms display a distorted octahedral coordination geometry by coordination with four nitrogen atoms of the macrocyclic ligand in a folded conformation, plus two carboxylate oxygen atoms of IPA^{2-} in *cis*-positions. The Ni–N bond lengths [2.082(3)-2.139(3) Å for 1, 2.053(7)-2.160(7) Å for 2 and 2.067(4)-2.152(4) Å for 3] are slightly shorter than the Ni–O bond lengths [2.115(2) and 2.187(2) Å for 1, 2.097(5)-2.220(5) Å for 2, and 2.112(3)-2.189(3) Å for 3] (table 2). Complexes 2 and 3 constitute a pair of stereoisomers and their macrocyclic ligands adopt a *RR* and *SS* configuration, respectively. The monomers of $\{[Ni(rac-L)]_2(\mu-IPA)\}^{2+}$ are connected through N– $H\cdots O$ (2.991(4)Å) and O– $H\cdots O$ (2.701(5) and 2.896(4)Å) intermolecular hydrogen bonds between the carboxylate oxygen atom (O2) of IPA²⁻, water (O1w and O3w), and the nitrogen atom (N6) of L, giving a one-dimensional zigzag chains, and the zigzag chains are further aggregated through O– $H\cdots O$ (2.798(4)–3026(7)Å) intermolecular hydrogen bonds between the carboxylate oxygen atom (O4) of IPA²⁻, water (O2w), and the oxygen atoms (O5 and O7)

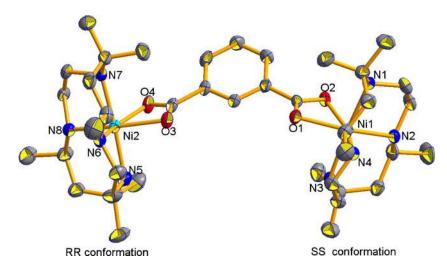


Figure 1. ORTEP diagram of $\{[Ni(rac-L)]_2(\mu-IPA)\}^{2+}$ with 50% probability displacement ellipsoids (H atoms, ClO_4^- and water molecule are omitted for clarity).

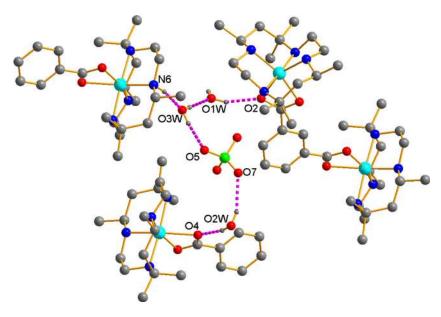


Figure 2. The intermolecular hydrogen bondings (dashed lines) in complex 1.

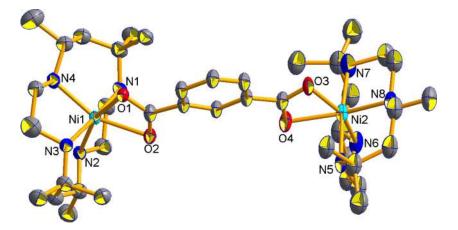


Figure 3. ORTEP diagram of $\{[Ni(RR-L)]_2(\mu-IPA)\}^{2+}$ with *RR* conformation and 30% probability displacement ellipsoids (H atoms, ClO_4^- and water molecule are omitted for clarity).

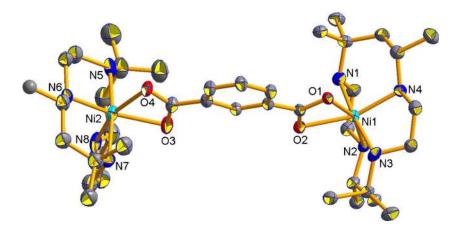


Figure 4. ORTEP diagram of $\{[Ni(SS-L)]_2(\mu-IPA)\}^{2+}$ with SS conformation and 50% probability displacement ellipsoids (H atoms, ClO_4^- and water molecule are omitted for clarity).

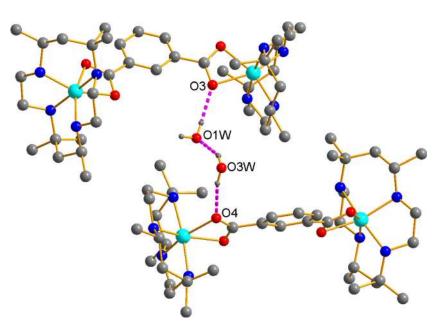


Figure 5. The intermolecular hydrogen bondings (dashed lines) in complex 3.

of ClO₄⁻, forming a two-dimensional sheet in complex 1 (figure 2 and table 3). While the monomers of $\{[Ni(SS-L)]_2(\mu-IPA)\}^{2+}$ are linked through O-H···O (2.758(6)–2.839(6) Å) intermolecular hydrogen bonds between the carboxylate oxygen atoms (O3 and O4) of IPA²⁻, water (O1w and O3w), giving a onedimensional left-handed helical chain in complex 3 (figures 5, 6 and table 3), similar to those found in previously reported.^{11,23} Each helix in the chain contains two $\{[Ni(SS-L)]_2(\mu-IPA)\}^{2+}$ monomers, with a pitch of 15.04 Å. Unfortunately a one-dimensional right-handed helical chain was not found in complex 2. Both compounds 2 and 3 crystallize in the same chiral space groups, $P2_12_12_1$. The absolute structure parameters of -0.04(2) and 0.032(16), respectively, confirm the homochiral nature of complexes 2 and 3.

3.2 UV-vis and CD spectra

The UV-vis spectra show an absorption peak near 230 nm for both complexes 2 and 3 (figure 7). As shown in figure 8, the bulk crystals of 2 show negative and positive Cotton effects near $\lambda = 440$ and 570 nm, respectively, while the bulk crystals of 3 show positive and negative Cotton effects of the opposite sign near the corresponding wavelengths. The results of CD measurements therefore confirm the chiral nature of complexes 2 and 3, in agreement with the X-ray crystal structures.

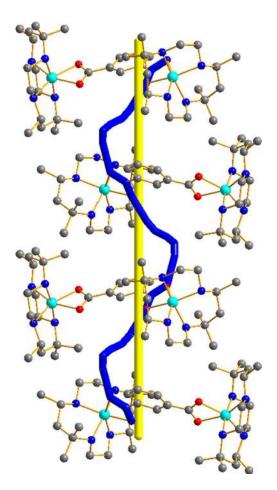


Figure 6. Side view of one-dimensional hydrogen bonded left-handed helical chain in complex 3.

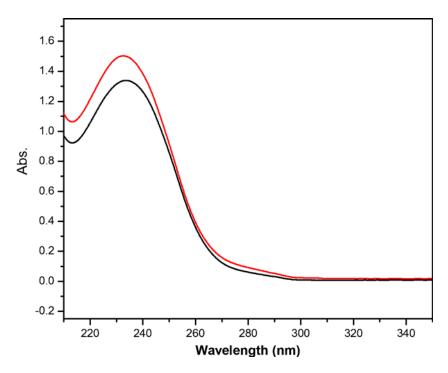


Figure 7. The UV-vis spectra of 2 and 3.

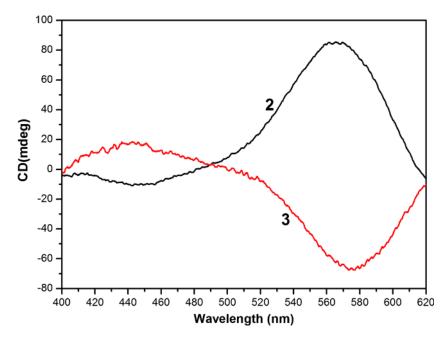


Figure 8. The solid CD_. spectra of 2 and 3.

4. Conclusion

In this paper, three dinuclear isophthalato-bridged nickel(II) complexes $[Ni(rac-L)]_2(\mu-IPA)(CIO_4)_2$ (1), $[Ni(RR-L)]_2(\mu-IPA)(CIO_4)_2$ (2) and $[Ni(SS-L)]_2(\mu-IPA)(CIO_4)_2$ (3) have been described. All the compounds are bridged by IPA^{2-} anions to form dimeric

structures, in which the two Ni(II) atoms display a distorted octahedral coordination geometry. The {[Ni(*SS*-L)]₂(μ -IPA)}²⁺ monomers are connected through intermolecular hydrogen bonds to generate one-dimensional left-handed helical chains. The results demonstrate that hydrogen bonds and shape of bridged ligand can affect the construction of helical structures.

Supplementary Information

Crystallographic data for 1, 2 and 3 have been deposited with the Cambridge Crystallographic Data Centre as supplemental publication numbers CCDC 989829, 989830 and 989831, respectively. Copies of the data can be obtained free of charge via http://www.ccdc.cam.ac. uk.

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