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Thermal Behavior, and Biological Activity of a New Heterotrinuclear Complex: [(NiL(NCS)(H<sub>2</sub>O))<sub>2</sub>Cd(DMF)<sub>2</sub>]

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Accepted author version posted online: 13 Jan 2015.

To cite this article: Alper Yardan, Ahmet Karahan, N. Burcu Arslan, Cigdem Hopa, Mukadderat Gokmen, Canan Kazak & Raif Kurtaran (2015) Synthesis, Characterization, Crystal Structure, Thermal Behavior, and Biological Activity of a New Heterotrinuclear Complex: [(NiL(NCS)(H<sub>2</sub>O))<sub>2</sub>Cd(DMF)<sub>2</sub>], Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry, 45:8, 1224-1233, DOI: <u>10.1080/15533174.2013.862679</u>

To link to this article: <u>http://dx.doi.org/10.1080/15533174.2013.862679</u>

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# Synthesis, Characterization, Crystal Structure, Thermal Behavior, and Biological Activity of a New Heterotrinuclear Complex: [(NiL(NCS)(H<sub>2</sub>O))<sub>2</sub>Cd(DMF)<sub>2</sub>]

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Received 26 August 2013; accepted 2 November 2013

A new trinuclear heterometallic nickel(II)-cadmium(II)-nickel(II) complex,  $[(NiL(NCS)(H_2O))_2Cd(DMF)_2]$  (DMF: dimethylformamide) (1), has been synthesized by using NiL as the so-called metallo ligand where  $H_2L = N,N'$ -bis(salicylidene)-1,3-diaminopropane, cadmium nitrate, and amonium thiocyanate. The title complex was characterized by various methods including elemental analyses, IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, TG-DTA, and X-ray diffraction techniques. The complex crystallizes in monoclinic space group P2<sub>1/c</sub>, with unit cell dimensions a = 10.6217(3), b = 13.3487(4), c = 16.0043(4) Å,  $\beta = 93.823(2)^{\circ}$ . The complex was screened for antimicrobial activities by the disc diffusion using DMF as solvent. The minimum inhibitory concentration values were calculated.

Keywords: Schiff base ligands, hetero trinuclear complexes, thermal analysis, single crystal XRD, biological activity

#### Introduction

Research on coordination polynuclear complexes of transition metals containing Schiff base ligands, continue to attract intense interest because of their importance in catalysis, molecular electronics, photochemistry.<sup>[1-3]</sup> At the same time heteropolymetallic complexes are active fields of research including chemistry, material science and nanostructure studies.<sup>[4-8]</sup> Tetradentate ONNO type Schiff base ligands derived from N,N'-bis(salicylidene)-1,3-diaminopropane (H<sub>2</sub>L), salen have been used for complexation in recent years. Salen-type Schiff base complexes themselves can act as ligand-complexes and can chelate a second metal substrate using phenolate oxygen atoms of Schiff base ligand to construct extended homo- or heteropolynuclear complexes.<sup>[9,10]</sup> Recently, phenoxo bridged heterometallic complexes have been great interest to the chemists due to their magnetic interactions.<sup>[11-13]</sup> A survey of literature reveals that although heterotrinuclear complexes containing both nickel(II) and cadmium(II) centers have not received adequate attention, mono-and polynuclear Schiff base complexes of Ni(II) and Cd(II) have received enormous attention due to their extensive use in the design and preparation of molecule based materials.<sup>[14–17]</sup>

In this study, we report the synthesis and structural characterization of the new linear hetero trinuclear complex of general formula  $[(Ni(L)(NCS)(H_2O))_2Cd(DMF)_2]$  has been synthesized. Scheme 1 demonstrates the synthetic pathway for  $[(NiL(NCS)(H_2O))_2Cd(DMF)_2]$ .

#### Experimental

#### Materials and Measurements

All chemical reagents and solvents used were purchased from Merck, Aldrich, or Carlo Erba and used without further purification. The elemental analyses for complex were carried out at the Eurovector 3018 CHNS analyzer. IR spectra were obtained by using IR grade KBr disks on a Perkin Elmer 1600 Series FT-IR spectrophotometer in the range of 4000–400 cm<sup>-1</sup>. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR analysis were performed using a Bruker Ultrashield Superconducting 400 MHz liquid NMR spectrometer. Thermogravimetry/differential thermal analysis (TG/DTA) measurements were run on a Perkin Elmer Diamond.

#### Synthesis of the [(NiL(NCS)(H<sub>2</sub>O))<sub>2</sub>Cd(DMF)<sub>2</sub>]

N,N'-bis(salicylidene)-1,3-propanediamine ( $H_2L$ ) and was prepared by reacting basic ethanolic solution of salicylaldehyde and

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Sch. 1. Synthetic pathway for [(NiL(NCS)(H<sub>2</sub>O))<sub>2</sub>Cd(DMF)<sub>2</sub>].

1,3-diamino propane as described previously in the literature (Scheme 1).<sup>[18]</sup> NiL was prepared by reacting basic ethanolic solution of H<sub>2</sub>L with NiCl<sub>2</sub> as described previously in the literature.<sup>[18]</sup> A solution of Cd(NO<sub>3</sub>)<sub>2</sub>.4H<sub>2</sub>O (1 mmol; 0.308 g) in methanol (10 mL) was added slowly with constant stirring to a DMF solution (10 mL) of the NiL (2 mmol; 0.678 g). NH<sub>4</sub>SCN (2 mmol; 0.194 g) dissolved in water (10 mL) was added dropwise to the mixture. The mixture was stirred at boiling point under air atmosphere. The resultant mixture filtered and left to stand at room temperature for four days. Dark-green crystals were collected by filtration and dried in open air. Yield: 0.84 g

**Table 1.** Crystal and experimental data for the complex 1

		Cui-O
Chemical formula	C <sub>42</sub> H <sub>50</sub> Cd N <sub>8</sub> Ni <sub>2</sub> O <sub>8</sub> S <sub>2</sub>	Cd1-O
CCDC No.	769387	Nil-N2
Formula weight	1088.84	Nil-N
Crystal system, space group	Monoclinic, $P2_{1/c}$	Nil-O2
a (Å)	10.6217(3)	Ni1-Ol
b (Å)	13.3487(4)	O3-C1
c (Å)	16.0043(4)	N2-C1
β	93.823(2)	N2-C1
Volume (Å <sup>3</sup> )	2264.13(11)	N1-C7
Z	2	
Calculated Density ( $Mg.m^{-3}$ )	1.597	O1-Cd
(F <sub>000</sub> )	1116	O1-Cd
Absoption coefficient $(mm^{-1})$	1.441	O1-Cd
Wavelength	0.71073	O2-Cd
Temperature (K)	293(2)	O1-Cd
h, k, l index (°)	-13/13, -16/16, 20/20	O1-Cd
$\theta_{\min}$ - $\theta_{\max}(^{\circ})$	1.92-26.63	O2-Cd
Number of reflections used	3804 (I > $2\sigma$ (I))	O2-Cd
Independent reflections	4735	O1-Cd
Number of parameters	294	O1-Cd
R <sub>int</sub>	0.0750	O2-Cd
R; $R_w(I > 2\sigma(I))$	0.0265; 0.0603	O3-Cd
S	0.950	N2-Ni
$\Delta \rho_{\rm min.}; \Delta \rho_{\rm max.} (e/{\rm \AA}^3)$	-0.48, 0.33	N2-Ni

(77%).  $C_{42}H_{50}CdN_8Ni_2O_8S_2$  (1088.84): Anal. Calcd. C 46.33; H 4.63; N 10.29%; Found C 46.15; H 4.81; N 10.41%.

#### Crystal Structure Determination

The data collection was performed at 293 K on a STOE IPDS II diffractometer equipped with a graphite monochromated Mo–K $\alpha$  radiation ( $\lambda = 0.71073$  Å). The structure was solved by direct methods using SHELXS-97<sup>[19]</sup> and refined

Table 2. Bond lengths (Å) and angles (°) in the metal coordination spheres of complex 1

_	-		
Cd1-O1	2.2411(13)	N1-C8	1.466(3)
Cd1-O2	2.3181(15)	N3-C18	1.158(3)
Cd1-O3	2.3611(16)	N4-C19	1.301(3)
Ni1-N2	2.0231(18)	N4-C20	1.454(4)
Nil-N1	2.025(2)	N4-C21	1.455(3)
Ni1-O2	2.0358(14)	Ni1-N3	2.127(2)
Nil-Ol	2.0376(15)	Ni1-O4	2.1347(19)
O3-C19	1.246(3)	S1-C18	1.630(3)
N2-C11	1.271(3)	O1-C17	1.326(2)
N2-C10	1.466(3)	O2-C1	1.325(3)
N1-C7	1.273(3)		
O1-Cd1-O1	180.0	N1-Ni1-O2	90.48(7)
O1-Cd1-O2	107.71(5)	N2-Ni1-O1	90.02(7)
O1-Cd1-O2	72.29(5)	N1-Ni1-O1	173.06(7)
O2-Cd1-O2	180.0	O2-Ni1-O1	82.65(6)
O1-Cd1-O3	87.65(5)	N2-Ni1-N3	87.93(8)
O1-Cd1-O3	92.35(5)	N1-Ni1-N3	88.74(8)
O2-Cd1-O3	90.52(6)	O1-Ni1-N3	92.77(7)
O2-Cd1-O3	89.48(6)	N2-Ni1-O4	89.30(8)
O1-Cd1-O3	92.35(5)	N1-Ni1-O4	89.10(9)
O1-Cd1-O3	87.65(5)	O2-Ni1-O4	88.53(7)
O2-Cd1-O3	90.52(6)	01-Ni1-O4	89.73(7)
O3-Cd1-O3	180.0	N3-Ni1-O4	176.27(9)
N2-Ni1-N1	96.80(8)	O2-Ni1-N3	94.53(7)
N2-Ni1-O2	172.37(7)		

Table 3. Hydrogen bonding distances (Å) and angles (°) for complex 1

D-HA	d(D-H)	$d(H\cdots A)$	d(DvA)	$\angle (D-H\cdots A)$
$\overline{ O(4)-HvO(3)^{i} } O(4)-H\cdots S(1)^{ii}$	0.809	1.920	2.690	158.54
	0.886	2.417	3.294	170.03

Symmetry codes: i-x, -y, 2-z; ii -x, -1/2 + y, 2.5 - z.

by a full-matrix least-squares procedure using the program SHELXL-97.<sup>[20]</sup> All non-hydrogen atoms were easily found from the difference Fourier map and refined anisotropically. All carbon and hydrogen atoms were included using a riding model. Molecular graphics were prepared using ORTEP 3.<sup>[21]</sup>

#### **Biological Activity of the Complex**

#### Microorganisms Used

A total of 3 Gram-negative and 3 Gram-positive organisms were used in the study. The isolates *Escherichia coli* ATCC 25292, *Pseudomonas fluorencens* ATCC 49642, *Enterobacter sakazakii* ATCC BAA-894, *Staphylococcus aureus* ATCC 6538, *Bacillus cereus* ATCC 11778, *Listeria monocytogenes* ATCC 764.

#### Antibiotic Gentamicin Used (Control)

The antibiotic standard used in this study was Gentamicin G10 susceptibility test discs (10 mg per disc) which were obtained from Oxoid Ltd., Basingstoke, England.

#### Antimicrobial Screening

Antimicrobial activities of complexes were determined by the disc diffusion method of CLSI (2006) on Mueller-Hilton agar (Oxoid, CM337). All tests were performed in duplicate to check the results. Suspension of the tested microorganisms (10<sup>6</sup> CFU/ $\mu$ L) was spread on the solid media plates. Test solution was prepared in DMF. Then antimicrobial susceptibity blank discs (Oxoid Ltd., Basingstoke, England) were soaked with 10  $\mu$ L of the stock solutions and placed on the inoculated plates, they were incubated at 35°C for 24 h for bacteria. The diameters of the inhibition zones were measured in millimeters. The diameter of the zone of clearance (including the diameter of the disk) was measured to the nearest whole milimeter and interpreted on the basis of CLSI guideline.<sup>[22]</sup>

#### Determination of Minimum Inhibitory Concentration

The minimum inhibitory concentration (MIC) of the metal complex against the test microorganisms were employed to the macrodilution broth susceptibility assay. Determination of the results of MIC was done according to the method of the turbidity of the inocula was adjusted to match that of 0.5 McFarland standards. The tubes were incubated in aerobically at  $35^{\circ}$ C for 20 h and at the end of the incubation period, the bacterial growth in the test tubes was checked and the last tube of growth observed concentration MIC was determined.<sup>[22]</sup>



Fig. 1. FT-IR spectrum of (a) H<sub>2</sub>L ligand and (b) complex 1.



**Fig. 2.** <sup>1</sup>H-NMR spectra of ligand ( $H_2L$ ).



Fig. 3. <sup>1</sup>H-NMR spectra of complex 1.



**Fig. 4.** <sup>13</sup>C-NMR spectra of ligand ( $H_2L$ ).

#### **Results and Discussion**

#### IR and NMR Spectra

The H<sub>2</sub>L ligand and the complex 1 FT-IR studies are shown in Figure 1. The interest of the IR spectrum of the title complex lies mainly in the bands due to the thiocyanate ion, water molecule and carbonyl group from the DMF ligand. The spectrum was compared with the free ligand's one (H<sub>2</sub>L) which shows significant bands at 1611 cm<sup>-1</sup> for C=N stretching vibration, 2752–2748 cm<sup>-1</sup> for phenolic O—H stretching and 1294 cm<sup>-1</sup> for phenolic C—O (Figure 1). The mononuclear complex (NiL) does not show bands from the aromatic O–H stretching and the C = N stretching frequency is shifted to upper wavelength from 1611 to 1639 cm<sup>-1</sup>. Also, medium intense bands assignable to Ni—N (542 cm<sup>-1</sup>) and Ni—O (470 cm<sup>-1</sup>) stretching vibrations can be observed.<sup>[23,24]</sup> The FT-IR spectra of the complex 1 clearly show the characteristic medium and

broad absorptions in the 3366  $\text{cm}^{-1}$  region due to the presence of water (Figure 1). This very broad band can also indicate the presence of strong hydrogen bonds, which is substantiated by crystal structure. The  $\nu$ (C=N) stretching vibration for Nbonded thiocyanate usually appears below 2100 cm<sup>-1</sup> and our complex 1 shows a strong band at 2071  $\text{cm}^{-1}$  (Figure 1) supporting the presence of terminally N-bonded thiocyanate groups to the Ni atom.<sup>[16,25]</sup> Also, the  $\nu$ (C=N) stretching vibrations of azomethine groups of the heterotrinuclear Schiff base complexes are observed a strong band at 1639 cm<sup>-1</sup> (Figure 1). This situation indicates the nitrogen atoms of the azomethine groups are coordinated to the metal ion. These data are consistent with the literature values.<sup>[26,27]</sup> The  $\nu$ (C=O) stretching vibration of the DMF molecules can be assigned to the band at  $1554 \text{ cm}^{-1}$  in the IR spectrum, which is lower than that in free DMF molecules  $(1655 \text{ cm}^{-1})$  due to the coordination of the DMF oxygen atoms to the Ni(II) ion,<sup>[28]</sup> which is consistent with the results of X-ray analysis.



Fig. 6. TG/DTA curves of the [(NiL(NCS)(H<sub>2</sub>O)<sub>2</sub>Cd(DMF)<sub>2</sub>].



Fig. 7. The ORTEP drawing of the complex 1 with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

H-NMR spectra of complex were recorded in DMF solution at 400 MHz and chemical shifts are in units of ppm relative to TMS. Peak of ligand azomethine proton was observed 8.58 ppm in <sup>1</sup>H-NMR spectra (Figure 2). Otherwise, complex azomethine proton signals shifted 7.91 ppm (Figure 3). This situation has shown that azomethine nitrogen joined coordination. Intramolecular hydrogen bonding accounts for 13.5 ppm frequency of the phenolic hydrogen signals in the H<sub>2</sub>L ligand. The absence of peak 13.5 ppm in the complex spectra indicates loss of phenolic hydroxy proton on complexation.<sup>[29]</sup>

In <sup>13</sup>C-NMR spectrum, ligand imine signals (166.1 ppm) are shifted to upfield (163.7) after complexation which means that the shifts are due to coordination of the ligand to metal atom by the azomethine nitrogen (Figures 4 and 5).<sup>[30,31]</sup>

### **TG-DTA** Curves

To examine thermal stability of 1, thermogravimetric and differential thermal analyses (TG-DTA) were made between 20 and  $1200^{\circ}$ C in the static atmosphere of nitrogen. TG-DTA curves (Figure 6) indicate that complex **1** is not decomposite up to 83°C and then gradual decomposition occurs in three successive steps within the temperature range 83–1200°C with endothermic effects at 113, 370, and 529°C. The first weight loss between 83 and 145°C is associated with the release of water and DMF molecules (observed 17.3%; calcd. 16.7%).<sup>[32,33]</sup> Second and third steps involve the loss of the ligand L and thiocyanate groups (observed 51.4%; calcd. 56.6%) and probably the formation of Ni<sub>2</sub>CdO<sub>4</sub> as the final residue at 1200°C (observed 31.7%; calcd. 27%).

#### Crystal Structure

The molecular structure of complex 1 obtained by X-ray diffraction studies is shown in Figures 7 and 8. Also data collection and crystal data are given in Table 1, and important bond lengths and bonding angles around the coordination sphere are given in Table 2. The X-ray structural study reveals that complex 1 consists of heterometallic assembly of Ni(II) and Cd(II). The central cadmium atom, which is located on the inversion centre, has a distorted octahedral coordination geometry with six oxygen donor sets, four from the ligands in the equatorial planes and two from the DMF groups in the apical positions. The Cd-O bond distances range from 2.241 to 2.361Å. The bond angles around Cd are in the range of 72.29–180.0°. The greatest deviation of bond angles from the ideal octahedral angles is found for O1–Cd– O2 and O1<sup>i</sup>–Cd–O2, 72.29° (5) and 107.71° (5), respectively. The distortion from an ideal octahedral geometry is a consequence of the limited opening of the tetradentate ligands, which reduces the bridging O1–Cd–O2 angle. All other bond angles are close to the ideal values. The isothiocyanato moiety is almost linear N5–C8–S1 178.3° (2). The maximum deviation from linearity of SCN<sup>-</sup> ion is 1.7°.

The terminal Ni atoms are also six-coordinated by two imine nitrogen atoms, two deprotonated phenoxo oxygen atoms from the ligand, an oxygen atom of water molecule, and a nitrogen atom from terminal thiocyanate anion. The axial positions are occupied with the O atom of the water molecule [Ni–O4 = 2.1347 (19) Å] and N atom of the SCN<sup>-</sup> ion coordinate to the

Ni atom to complete the sixth-coordination [Ni–N5 = 2.127 (2) Å]. Ni-Cd atoms are equatorially bridged by two phenolic oxygen atoms of ligand with angles of Ni1–O1–Cd1 = 102.62° (6) and Ni1–O2–Cd1 = 100.07° (6). The Ni $\cdots$ Cd distance is 3.342 Å. The possible hydrogen bonds indicate intermolecular hydrogen bonds. These hydrogen bonds are rather weak interactions as can be seen from the details given in Table 3. O–H $\cdots$ O intermolecular interactions interlink these chains along the *bc*-axis.

#### **Biological Activity**

Screening for antimicrobial activities of the stock solutions of the Schiff base and the metal complexes were performed qualitatively using the disc diffusion method. The metal complex 1, NiL and the H<sub>2</sub>L yielded clear inhibition zones around the disc.<sup>[34]</sup> These showed activity against *E. coli* ATCC 25292, *P. fluorescens* ATCC 49642, and *S. aureus* ATCC 6538, while NiL was showed strong activity against



Fig. 8. Packing of the atoms in the unit cell.

Table 4. Antimicrobial screening of complex 1 (mm)

Microorganism	Stock solution				
	Diameter of inhibition zone (mm)				
	Complex 1	NiL	$H_2L$	Control*	
Escherichia coli ATCC 25292	14	11	9	23	
Pseudomonas fluorescens ATCC 49642	13	11	8	24	
Enterobacter sakazakii ATCC 11778	10	12	9	24	
Staphylococcus aureus ATCC 6538	12	11	7	23	
Bacillus cereus ATCC 7064	11	10	7	24	
Listeria monocytogenes ATCC 764	9	9	8	24	

\*Gentamicin.

*E. sakazakii* ATCC 11778 and these complexes and the free ligand showed weak activity against *L. monocytogenes* ATCC 764 (Table 4). If the inhibition zone measures 2 and 3 mm, then the complex has a good bactericidal action on the tested bacterium. If the inhibition zone more than 3 mm across, then it is considered very effective, but if there is no inhibition zone then the complex has no activity on the bacterium, and will not be retained for treatment.<sup>[35]</sup>

The antimicrobial activities of free ligand ( $H_2L$ ) and the complex 1 on the inoculated plates have been screened against *Escherichia coli* ATCC 25292, *Pseudomonas fluorescens* ATCC, *Enterobacter sakazakii* ATCC 11778, *Staphylococcus aureus* ATCC 6538, *Bacillus cereus* ATCC 7064 ve *Listeria monocytogenes* ATCC 764 by the Agar Disc Diffusion Method and the control antibiotic disc (gentamicin). The metal complex yielded clear inhibition zones around the disc. The complex 1 showed strong activity against *E. coli* ATCC 25292 and showed weak activity against *L. mononctogenes* ATCC 764 (Table 4).

As shown Table 5, the MIC values of the compounds that show positive results in the screening process were determined as a quantitative measurement of antibacterial activity. The activities of the complex have been compared with the activity of standard antibiotic gentamicin. The results revealed that the complex is most effective against E. coli ATCC 25292, P. fluorescens ATCC, S. aureus ATCC 6538 with MIC value of 16  $\mu$ g/mL while the complex has lower MIC value of 32  $\mu$ g/mL against bacteria of *E. sakazakii* ATCC 11778, B. cereus ATCC 7064 ve L. monocytogenes ATCC 764. Though there is sufficient increase in the bacteriostatic activity of the complex 1, it could not reach the effectiveness of the conventional antibacterial agent, Gentamicin. The results of this study are with similar to the our previous study.<sup>[34]</sup> This study suggests that the antibacterial activity of the ligand is enhanced when it is chelated with metal ions. The increase in the antimicrobial activity of metal chelates

Control (including Micro Complex NiL H<sub>2</sub>L organisms Sources culture) \*\* Escherichia coli ATCC 25292 16 32 64 \*\* ATCC 49642 16 32 64 Pseudomonas fluorescens Enterobacter ATCC 11778 32 64 128 \*\* sakazakii \*\* ATCC 6538 32 64 Staphylococcus 16 aureus \*\* Bacillus cereus ATCC 7064 32 64 128 ATCC 764 32 128 \*\* Listeria 64 monocytogenes

**Table 5.** Antimicrobial activity (MIC) of the complexes ( $\mu$ g/mL)

\*\*Growth determination.

may be due to the effect of the metal ions on the normal cell process.

#### Conclusion

The heterometallic trinuclear Ni(II)-Cd(II)-Ni(II) complex from N,N'-bis(salicylidene)-1,3-diaminopropane ( $H_2L$ ) has been synthesized in dimethylformamide medium and investigated using elemental analyses, infrared and electronic spectra, <sup>1</sup>H- and <sup>13</sup>C-NMR spectra, XRD, and thermal analysis. The phenolate oxygen atoms of Schiff base ligand bridged nickel and cadmium atoms. The distorted octahedral coordination sphere of nickel is completed by aqua molecule and isothiocyanate ion. Although the pseudohalide ions are attractive as coligands due to their bridging metal ions to each other, and neutralize the charges of the complexes, in this complex isothiocyanate ions are bonded to the nickel atoms as a terminal ligand. The most recent by Pearson,<sup>[36]</sup> suggest that S in SCN<sup>-</sup> is soft and will prefer to coordinate with soft acids whereas N in SCN<sup>-</sup> is hard and coordinates with hard acids. So nickel ions are coordinated with the hardest part of the NCS<sup>-</sup> ion. The biological activities of the characterized complex 1 were also studied and compared with H<sub>2</sub>L and NiL. Studies along these lines are currently in progress in our laboratory.

#### Funding

The financial support of scientific research Council of Balikesir University (Project No. 2007/10) is gratefully acknowledged.

#### **Supplementary Material**

CCDC 769387 contains the supplementary crystallographic data. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road,

Cambridge CB21EZ, UK; fax: (+44) 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk

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