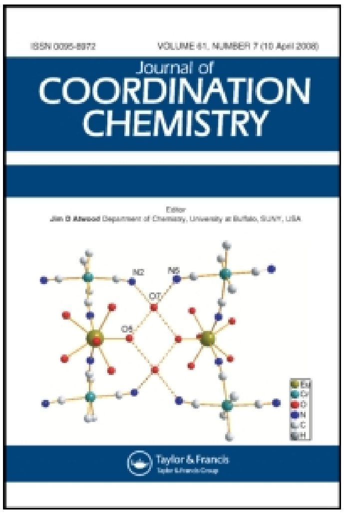

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Synthesis, structures, and characterization of copper(II), nickel(II), and cobalt(III) metal complexes derived from an asymmetric bidentate Schiff-base ligand

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Synthesis, structures, and characterization of copper(II), nickel(II), and cobalt(III) metal complexes derived from an asymmetric bidentate Schiff-base ligand

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An asymmetric bidentate Schiff-base ligand (2-hydroxybenzyl-2-furylmethyl)imine (L–OH) was prepared. Three complexes derived from L–OH were synthesized by treating an ethanolic solution of the appropriate ligand with an equimolar amount of metallic salt. Three complexes, $Cu_2(L–O^-)_2Cl_2$ (**1**), $Ni(L–O^-)_2$ (**2**) and $Co(L–O^-)_3$ (**3**), have been structurally characterized through elemental analysis, IR, UV spectra and thermogravimetric analysis. Single crystal X-ray diffraction shows metal ions and ligands reacted with different proportions 1 : 1, 1 : 2 and 1 : 3, respectively, so copper(II), nickel(II), and cobalt(III) have different geometries.

Keywords: X-ray crystallography; Copper(II) complex; Nickel(II) complex; Cobalt(III) complex; Bidentate Schiff-base ligand

1. Introduction

Salicylaldehyde Schiff base has been used extensively in complexes of transition and main group metals [1]. Schiff-base complexes with mononuclear and dinuclear metals have been studied in great detail for their coordination, spectroscopic properties, catalysis properties [2–6] and bioinorganic properties [7–11]. Asymmetric Schiff-base ligands offer many advantages over their symmetrical counterparts in the elucidation of the composition and geometry of metal ion binding sites in metalloproteins, and selectivity of natural systems with synthetic materials [12]. Understanding the coordination chemistry of these complexes, which is affected by the strain of the ligands, has an important role on the above properties [13, 14]. In this report, an asymmetric Schiff-base ligand, (2-hydroxybenzyl-2-furylmethyl)imine (L-OH), has been synthesized. We describe the transition metal complexes, thermogravimetric analysis and their spectroscopic properties. The crystal structures of **1**, **2** and **3** are also reported by single crystal X-ray diffraction.

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2. Experimental

2.1. *Materials and physical measurements*

All reagents and solvents used were purchased from commercial sources of analytical grade. The C, H and N analyses were taken with a Perkin–Elmer model 2400 elemental analyzer. IR spectra were recorded on an EQUINOX 55 IR spectrophotometer using KBr pellets. ^{1}H NMR spectra of $CDCl_3$ solution were recorded on a Bruker ARX400. Thermogravimetric analysis was performed under nitrogen with a heating rate of $10^\circ C\,min^{-1}$ using a NETZSCH STA 499C thermal analyzer. UV-Vis spectra were measured on a UV-1700 spectrophotometer in the range 200–800 nm.

2.2. *Synthesis*

2.2.1. Synthesis of Schiff-base ligand, (2-hydroxybenzyl-2-furylmethyl)imine (L–OH). 2–Furylmethylamine (0.9705 g, 10.0 mmol) in methanol (8 mL) was added to a solution of an equimolar amount of salicylaldehyde (1.221 g, 10.0 mmol) in methanol (6 mL), and the mixed solution immediately turned yellow. After stirring at room temperature for 12 h, the solvent was removed by rotary evaporation to obtain a yellow hygroscopic liquid. Several attempts to recrystallize the compound were unsuccessful, so the ligand was then used for metalation without further purification [15, 16]. IR: ν(–OH) 3421 cm^{-1}, ν_s(C=N) 1632 cm^{-1}, ν_s(Ar–O) 1279 cm^{-1}, ν_{as}(C–O–C) 1212 cm^{-1}, ν_s(C–O–C) 1148 cm^{-1}, ω(Ar–H) 755 cm^{-1}. ^{1}H NMR ($CDCl_3$): $\delta = 13.15$ (s, 1H, Ar–OH), 8.38(s, 1H, –CH=N–), 7.33(s, 1H, Ar–H), 7.29(t, 1H, Ar–H), 7.27 (d, 1H, furyl), 6.97(d, 1H, Ar–H), 6.88(t, 1H, Ar–H), 6.35(t, 1H, furyl), 6.28(d, 1H, furyl), 4.76(q, 2H, CH_2).

2.2.2. Synthesis of $Cu_2(L\text{–}O^-)_2Cl_2$ (1). L–OH (0.0603 g, 0.3 mmol) in ethanol (5 mL) was added to a solution of $CuCl_2 \cdot 2H_2O$ (0.0511 g, 0.3 mmol) in ethanol (4 mL) dropwise. Stirring at room temperature, the resulting solution was filtered after 16 h and the solution was kept standing for about 4 days at room temperature to give dark-green crystals (m.p. >573K) suitable for X-ray diffraction. The yield was 56%. Anal. Calcd for $C_{24}H_{20}N_2O_4Cl_2Cu_2$: C, 48.17; H, 3.37; N, 4.68%. Found: C, 48.75; H, 3.09; N, 4.45%. IR: ν_s(C=N) 1621 cm^{-1}, ν_s(Ar–O) 1301 cm^{-1}, ν_{as}(C–O–C) 1210 cm^{-1}, ν_s(C–O–C) 1145 cm^{-1}, ω(Ar–H) 760 cm^{-1}.

2.2.3. Synthesis of $Ni(L\text{–}O^-)_2$ (2). L-OH (0.0603 g, 0.3 mmol) was dissolved in diethyl ether and $Ni(NO_3)_2 \cdot 6H_2O$ (0.0872 g, 0.3 mmol) in methanol. Using three-layer diffusion, dark-green crystals (m.p. >573K) suitable for X-ray diffraction were grown after about 2 weeks. The yield was 37%. Anal. Calcd for $C_{24}H_{20}N_2O_4Ni$: C, 62.74; H, 4.39; N, 6.10%. Found: C, 62.38; H, 4.04; N, 6.45%. IR: ν_s(C=N) 1612 cm^{-1}, ν_s(Ar–O) 1329 cm^{-1}, ν_{as}(C–O–C) 1214cm^{-1}, ν_s(C–O–C) 1143 cm^{-1}, ω(Ar–H) 758 cm^{-1}.

2.2.4. Synthesis of $Co(L\text{–}O^-)_3$ (3). Complex **3** was prepared in a manner similar to **1** (m.p. >573K) using cobalt nitrate. The yield was 43%. Anal. Calcd for $C_{36}H_{31}N_3O_6Co$: C, 65.46; H, 4.73; N, 6.36%. Found: C, 65.89; H, 4.34; N, 6.45%. IR: ν_s(C=N)

Table 1. Crystal data and structure refinement.

	1	**2**	**3**
Chemical formula	$C_{24}H_{20}N_2O_4Cl_2Cu_2$	$C_{24}H_{20}N_2O_4Ni$	$C_{36}H_{30}N_3O_6Co$
Formula weight	595.94	458.08	659.56
Temperature (K)	296(2) K	296(2)	273(2)
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2(1)/c$	C2/c	$P\bar{1}$
a (Å)	11.7358(9)	20.284(2)	8.9989(5)
b (Å)	12.7163(10)	5.7565(7)	10.6121(6)
c (Å)	8.3612(6)	17.358(2)	16.3435(10)
α (°)	90	90	94.0890(10)
β (°)	108.0410(10)	94.766(2)	94.8850(10)
γ (°)	90	90	93.5780(10)
V (Å^3)	1186.44(16)	2019.8(4)	1547.44(15)
Z	3	4	2
Radiation (λ, Å)	Mo-Kα (0.71073)	Mo-Kα (0.71073)	Mo-Kα (0.71073)
D_{Calcd} (Mg cm^{-3})	1.675	1.510	1.416
Absorption coefficient (mm^{-1})	2.052	0.996	1.155
Crystal size (mm^3)	0.46 × 0.25 × 0.11	0.38 × 0.29 × 0.12	0.48 × 0.28 × 0.18
θ range for data collection (°)	2.43–25.10	2.01–25.09	1.93–25.10
Index ranges	$-14 \le h \le 14$, $-9 \le k \le 15$, $-9 \le l \le 9$	$-24 \le h \le 24$, $-5 \le k \le 6$, $-20 \le l \le 20$	$-7 \le h \le 10$, $-12 \le k \le 12$, $-19 \le l \le 19$
Reflections collected	5834	4799	7835
Independent reflections (R_{int})	2106 (0.0221)	1797 (0.0159)	5405 (0.0139)
Goodness-of-fit on F^2	1.119	1.097	1.083
Final R indices	$R_1 = 0.0331$, $wR_2 = 0.0909$	$R_1 = 0.0333$, $wR_2 = 0.0921$	$R_1 = 0.0359$, $wR_2 = 0.952$
R indices (all data)	R_1=0.0422, $wR_2 = 0.0940$	$R_1 = 0.0382$, $wR_2 = 0.0953$	$R_1 = 0.0454$, $wR_2 = 0.0993$
Largest difference in peak and hole (e Å^{-3})	0.642 and −0.358	0.523 and −0.311	0.383 and −0.347

1626 cm^{-1}, ν_s(Ar–O) 1323 cm^{-1}, ν_{as}(C–O–C) 1209 cm^{-1}, ν_s(C–O–C) 1146 cm^{-1}, ω(Ar–H) 738 cm^{-1}.

2.3. *Crystal structure measurements*

Single crystals of **1** and **3** were obtained from ethanolic solution, respectively, by slow evaporation. Single crystals of **2** were obtained from the method of three-layer diffusion. Single-crystal X-ray diffraction data for **1**, **2** and **3** were collected from single crystals on a Bruker SMART APEX CCD X-ray diffractometer with graphite-monochromated Mo-Kα (λ = 0.71073 Å) radiation using ω scan mode. The determination of integral intensities and global cell refinement were performed with Bruker SAINT. The data collection, processing, and refinement of unit cell parameters were performed with SMART and SAINT programs. The structure was solved by direct methods (SHELXTL-97 [19]) and refined by the full-matrix least-squares method against F^2 in the anisotropic approximation (SHELXTL-97 [20]). Some crystallographic details are listed in table 1. Crystallographic data (excluding structure factors) for the structures reported in this article have been deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC 615407 for **1**, CCDC 615408 for **2** and CCDC 615409 for **3**. Copies of the data can be obtained free of

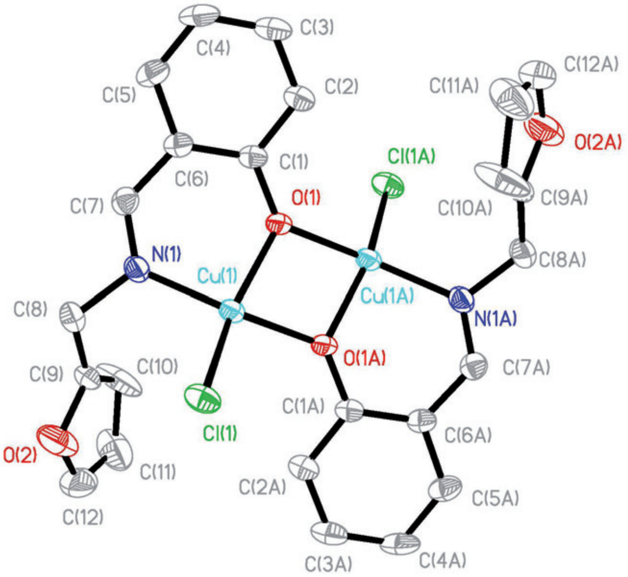

Figure 1. Perspective view of the complex **1**, showing 30% probability ellipsoids for the non-hydrogen atoms and the atom-labeling scheme, all hydrogen atoms have been omitted for clarity.

charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

3. Results and discussion

3.1. *Synthesis and characterization*

IR spectra of L–OH and corresponding complexes are very similar. In L–OH there is a strong absorption band at 1632 cm^{-1} assigned to the C=N stretching vibration, indicating the formation of the Schiff-base products [17, 18]. In **1**–**3**, absence of the phenolic O–H vibration indicates that ligand L-OH is deprotonated in complexes. Shift of C–O stretch of phenol to higher wavenumber confirms that metal ions are bound to the phenolic oxygen, and shifts to lower wavenumbers support the participation of the C=N group. The C–O–C vibration in furyl rings does not shift indicating that oxygen is non-coordinating, in agreement with the crystal structure.

We wanted a tridentate Schiff-base ligand using 2-furylmethylamine, but the furyl group is not coordinated because conjugation of the aromatic ring decreases the electron density of oxygen atom.

3.2. *Description of the crystal structures*

3.2.1. Structure description of complex 1. The molecular structure of **1** is shown in figure 1. Selected bond distances and angles are listed in table 2.

Table 2. Selected bond lengths (Å) and angles (°) for $Cu_2(L\text{-}O^-)_2Cl_2$ (**1**).

Bond lengths (Å)			
Cu(1)–O(1)	1.935(2)	Cu(1)–Cl(1)	2.2048(9)
Cu(1)–N(1)	1.936(3)	Cu(1)–O(1A)	1.965(2)
Cu(1A)–O(1)	1.965(2)	Cu(1A)–Cl(1A)	2.2048(9)
Bond angles (°)			
O(1)–Cu(1)–N(1)	92.59(10)	N(1)–Cu(1)–Cu(1A)	127.39(8)
O(1)–Cu(1)–O(1A)	77.24(9)	O(1A)–Cu(1)–Cu(1A)	38.27(6)
N(1)–Cu(1)–O(1A)	153.78(11)	Cl(1)–Cu(1)–Cu(1A)	133.85(3)
O(1)–Cu(1)–Cl(1)	152.53(8)	C(7)–N(1)–Cu(1)	123.8(2)
N(1)–Cu(1)–Cl(1)	98.40(9)	O(1A)–Cu(1)–Cl(1)	101.57(7)

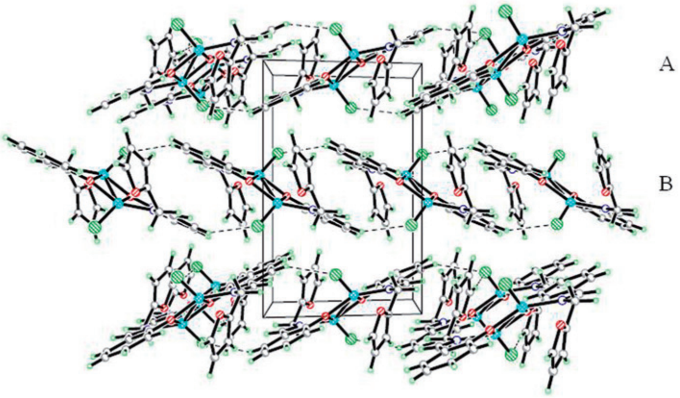

Figure 2. Perspective view of stacking array of complex **1** along *a* axis.

The X-ray structure of **1** shows that two copper atoms have the same configuration, defined by the two phenolate oxygen atoms O(1) and O(1A), which link two copper atoms as a bridge, the one imino nitrogen atom N(1) and the one chlorine atom Cl(1). The sum of plane angles Cu(1)–O(1)–Cu(1A), Cu(1)–O(1A)–Cu(1), O(1A)–Cu(1A)–O(1) and O(1A)–Cu(1)–O(1) is 360°, so the four atoms Cu(1), Cu(1A), O(1) and O(1A) are coplanar; bond lengths of Cu(1)–O(1), Cu(1A)–O(1A), Cu(1A)–O(1) and Cu(1)–O(1A) are the same. The geometry about each four-coordinate copper atom can be described as a distorted trigonal pyramid with N(1), O(1) and O(1A) making up the undersurface and Cl(1) as the vertex. No classical hydrogen bonds occur in the crystal structures of **1** and the C(12)–Cl(1) and C(5)–Cl(1) distances are 3.651(3) Å and 3.759(6) Å, respectively. Complex **1** arranges in an ABAB fashion forming a three-dimensional stratiform structure, with molecules in every layer connected by weak interaction of C–H $\cdots$ Cl bond [21] (figure 2).

3.2.2. Structure description of complex 2.

The molecular structure of **2** is shown in figure 2. Selected bond distances and angles are listed in table 3.

In **2**, the coordination geometry around the nickel is square planar. The two Ni–O bonds are *trans*, with equivalent bond lengths of Ni(1)–O(1) (1.8272(18) Å) and Ni(1)–O(1A) (1.8272(18) Å). The two N donor atoms are also "trans" [22], with Ni(1)–N(1)

Table 3. Selected bond lengths (Å) and angles (°) for Ni(L–O$^-$)$_2$ (**2**).

Bond lengths (Å)			
Ni(1)–O(1A)	1.8272(18)	Ni(1)–N(1A)	1.9192(18)
Ni(1)–O(1)	1.8272(18)	Ni(1)–N(1)	1.9193(18)
Bond angles (°)			
O(1A)–Ni(1)–O(1)	180.0	O(1A)–Ni(1)–N(1)	87.64(8)
O(1A)–Ni(1)–N(1A)	92.36(8)	O(1)–Ni(1)–N(1)	92.36(8)
O(1)–Ni(1)–N(1A)	87.64(8)	N(1A)–Ni(1)–N(1)	180.0

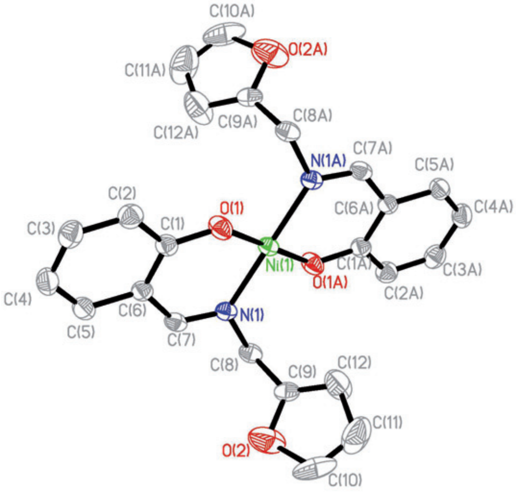

Figure 3. Perspective view of the complex **2**, showing 30% probability ellipsoids for the non-hydrogen atoms and the atom-labeling scheme, all hydrogen atoms have been omitted for clarity.

and Ni(1)–N(1A) bond lengths (1.9193(18) Å and 1.9192(18) Å) almost the same. Bond angles of O(1A)–Ni(1)–O(1) and N(1A)–Ni(1)–N(1) are 180°, so **2** becomes an invert centrosymmetric structure with the symmetric center at the nickel atom.

3.2.3. Structure description of complex 3.

The molecular structure of **3** is shown in figure 3. Selected bond distances and angles are listed in table 4.

The coordination sphere about cobalt consists of three phenolate oxygens and three imino nitrogen atoms (O_3N_3) from three ligands. Each ligand is bidentate using a phenolate oxygen and a pyridine nitrogen. The geometry about the cobalt atom is close to octahedral. The sum of the plane angles around cobalt atom, 359.8°, is very close to 360°, so Co(1), O(3), O(6), N(1) and N(2) atoms are nearly coplanar with the Co in the plane, and the bond angles within the 'plane' vary [23]. The bond angle of O(2)–Co(1)–N(3) is 176.84(7)°, so O(2), Co(1), and N(3) compose the upright axis in the octahedron. Average bond length of Co–O (1.8918 Å) is slightly shorter than the average bond length of Co–N (1.9424 Å). $Co(NO_3)_2$ was used to synthesize the complex in the experiment, but Co(II) was oxidized in the reaction.

Table 4. Selected bond lengths (Å) and angles (°) for Co(L–O$^-$)$_3$ (**3**).

Bond lengths (Å)			
Co(1)–O(3)	1.8858(15)	Co(1)–N(1)	1.9438(18)
Co(1)–O(6)	1.8885(15)	Co(1)–N(2)	1.9398(18)
Co(1)–O(2)	1.9012(14)	Co(1)–N(3)	1.9436(18)
Bond angles (°)			
O(3)–Co(1)–O(6)	174.13(7)	O(2)–Co(1)–N(2)	84.77(7)
O(3)–Co(1)–O(2)	90.22(7)	O(3)–Co(1)–N(3)	90.37(7)
O(6)–Co(1)–O(2)	88.14(6)	O(6)–Co(1)–N(3)	91.58(7)
O(3)–Co(1)–N(2)	94.34(7)	O(2)–Co(1)–N(3)	176.84(7)
O(6)–Co(1)–N(2)	91.12(7)		

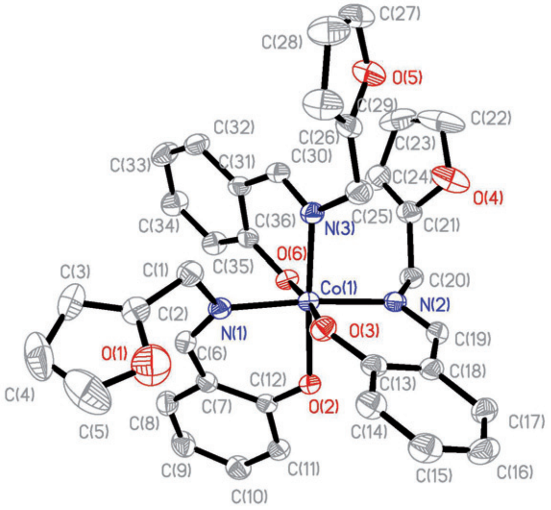

Figure 4. Perspective view of complex **3**, showing 30% probability ellipsoids for the non-hydrogen atoms and the atom-labeling scheme, all hydrogen atoms have been omitted for clarity.

3.3. *Thermogravimetric analysis*

Thermogravimetric analyses of **1**, **2** and **3** under nitrogen were examined.

Complex **1** shows a first weight loss of 29.66% in the temperature range 180–350°C, corresponding to removal of one ligand (Calcd 33.57%); the peak temperature in DSC curve is at 227.8°C. **1** continues to lose weight gradually and has a keen-edged exothermic peak at 711.2°C in the DSC curve.

Complex **2** undergoes two major stages of weight loss. First, one ligand molecule is removed between 150 and 350°C, leading to a total weight loss of about 42.50% (Calcd 43.68%) and giving an endothermic peak at 246.6°C in the DSC curve. The second weight loss occurs between 350–750°C, corresponding to removal of another ligand with weight loss of 41.12% (Calcd 43.68%) with one broad exothermic peak at 410–550°C in the DSC curve. The value of the residue (13.28%, Calcd 12.81%) agrees with the value calculated for metallic nickel.

Complex **3** undergoes three major weight loss stages. First, one ligand is removed between 180 and 280°C, leading to a weight loss of 30.58% (Calcd 30.31%) and giving an endothermic peak at 213.3°C in the DSC curve. The second weight loss occurs between 280 and 370°C with 15.37%, Calcd 15.15% corresponding to loss of half of one ligand. Finally, the third weight loss occurs between 370 and 830°C, with weight loss of 42.37%, Calcd 45.46% for removal of one and a half ligands; one broad exothermic peak between 390–570°C is in the DSC curve. The complex decomposes to metallic cobalt and the value of the residue is 9.54% (Calcd 8.92%).

Peaks in the DSC curves of **1**, **2** and **3** have the same interpretation, with the narrow endothermic peaks corresponding to melting of complexes and the exothermic peak corresponding to decomposition.

3.4. *UV-visible spectra*

The UV spectra of L–OH and **1**, **2** and **3** are measured in anhydrous ethanol. Ligand and complexes exhibit very similar UV spectra. In the UV spectrum of L–OH, two anterior peaks are at 222 and 256 nm, attributed to the π–π^* transitions of benzene ring. A peak attributed to the C=N group transition is observed at 316 nm [2, 17]. The two anterior peaks are red-shifted to 224, 268 nm; 225, 260 nm; and 224, 254 nm in **1**, **2** and **3**, respectively. The other peaks are also red-shifted to 368, 335 and 388 nm, respectively. The C=N group transition shifts more than the π–π^* transitions of benzene ring, indicating that the C=N group takes part in coordination [24].

The absorptions in **1**, **2** and **3** are all red-shifted and broader than in the ligand because formation of M(II)–N (M = Cu, Ni, Co) affect the π–π conjugation system [25].

4. Conclusions

We have reported three different crystals derived from LOH, with different metal ions in 1 : 1, 1 : 2 and 1 : 3 (M^{n+}: L–O^-) proportions. In order to keep charge balance, phenolic OH are deprotonated and bond to the metals as oxygen anions [26]. Due to having the same ligand, the complexes have similar IR and UV-visible spectra and thermogravimetric analyses.

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