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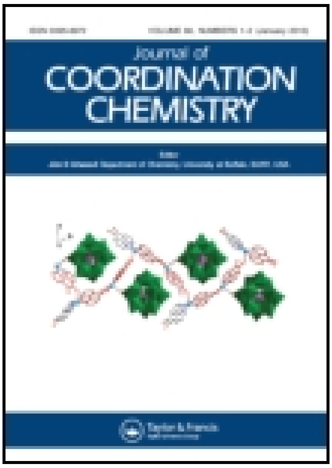

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Nickel(II), copper(II), and cobalt(II) complexes derived from a new unsymmetrical ONS donor Schiff base ligand: synthesis, characterization, crystal structure, and catalytic activities

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A series of Ni(II), Cu(II), and Co(II) complexes, ML_2, of a new thioether containing ONS donors have been synthesized, where L = deprotonated Schiff base. The analytical, spectral (FTIR, ^{1}H NMR, UV–vis), conductivity, and magnetic studies showed that the metal complexes possess octahedral geometry and are non-electrolytes. The structures of ligand as well as nickel(II) complex, NiL_2, **2**, were determined by single-crystal X-ray diffraction studies. The copper complex shows very good catalytic activities towards oxidation of organic thioethers to the corresponding sulfoxide predominantly using H_2O_2 as the oxidant.

Ni(II), Cu(II), and Co(II) complexes, ML_2, with a new thioether containing ONS donors were synthesized, where L = deprotonated Schiff base. The analytical, spectral (FTIR, ^{1}H NMR, and UV-vis), conductivity, and magnetic studies show that the metal complexes possess octahedral geometry and are non-electrolytes. The coordination mode of ligand, **1**, and nickel(II) complex, NiL_2, **2**, was determined by single-crystal X-ray diffraction studies. Here, the nickel is coordinated to two oxygens, two nitrogens, and two sulfurs of two tridentate ligands with slightly distorted octahedral environment around nickel. The copper complex shows very good catalytic activities towards oxidation of organic thioethers to the corresponding sulfoxide predominantly using H_2O_2 as the oxidant.

Keywords: ONS donor ligand; Crystal structure; Thioether; Catalytic activity; Schiff base

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1. Introduction

Schiff bases have led to a renewed interest in coordination chemistry as they can be easily synthesized, have good coordinating ability to metals, and have potential antibacterial, anticancer, and antifungal properties [1]. Chelating ligands with ONS donors are important in coordination chemistry owing to their stability, chemical properties, electrochemical activities, and broad biological activities [2]. Transition metal complexes with such ligands have versatile structural and functional properties and applications in catalysis, material research, etc. [3]. Coordination chemistry of copper with ONS donor ligands is interesting as their structures, spectral properties, redox properties, and medical applications mimic those of sulfur-containing proteins [4]. New complexes containing nickel provide better understanding of biological systems and also develop new homogeneous catalysts for selective oxidation [5]. Nickel complexes with ONS donors are effective in Co-dehydrogenase, saline alcoholysis, and also have fungi toxicities [6]. Cobalt complexes are important due to their anticancer properties, cytotoxic activities, etc. [7].

Metal complex-catalyzed conversion of sulfides to sulfoxides is significant in synthetic chemistry, offering effective synthesis of important compounds that have industrial and medical applications [8]. Transition metal complexes like oxido-peroxido tungsten, vanadium, and molybdenum give good selectivity towards sulfoxide formation from sulfide [9–12]. Also, nanoparticle-supported vanadium complexes are effective as heterogeneous catalysts in sulfide oxidation [13]. Copper Schiff base complexes also give predominantly sulfoxides from sulfides [14]. Nanoparticle-supported manganese complexes are used in the oxidation of thiol to disulfide [15].

Herein, we report the synthesis and characterization of a new ONS donor Schiff base, bromo, nitro, N-[2-(benzylthio) phenyl] salicylaldimine and its nickel(II), copper(II), and cobalt(II) complexes. Crystal structures of ligand and nickel(II) complex are solved by single-crystal X-ray diffraction studies. In this paper, we describe Schiff base copper complex-catalyzed oxidation of sulfides to their sulfoxides in the presence of H_2O_2, a process delivering a high yield and desirable reaction time.

2. Experimental

2.1. *Materials and methods*

Nickel chloride hexahydrate, copper nitrate trihydrate, and cobalt chloride hexahydrate were purchased from Merck, India. 3-Bromo-5-nitro salicylaldehyde was purchased from Alfa Aesar. 2-(Benzylthio)aniline was prepared according to the literature method [16]. Elemental analyses were recorded on a Perkin-Elmer Model 240C elemental analyzer. Electrical conductivities were measured on a CM-180 conductivity meter (Elico, India). Electronic spectra were measured on a Cary 100 Bio UV–visible spectrophotometer. Magnetic susceptibilities were measured on a conventional Gouy balance using freshly prepared $Hg[Co(NCS)_4]$ as calibrant by Magway MSB MK1 Magnetic susceptibility balance, Sherwood Scientific, Cambridge, UK. Infrared spectra of the ligand and complexes were recorded on an IR-affinity-I FTIR spectrometer SHIMADZU as KBr pellets. Melting points were recorded on a Veego melting point apparatus and were uncorrected. The ^1H NMR spectra were recorded on a Bruker 400 DRX spectrometer in $CDCl_3$ solution using TMS as the internal standard. All other reagents and solvents like methanol, ethanol, DMF, DMSO, dichloromethane (DCM), and acetonitrile were of commercial grade and employed as received or purified by standard methods prior to use. All the reactions were carried out at room temperature in open atmosphere.

2.2. *Synthesis of HL*

2-(Benzylthio)aniline (0.215 g, 1 mM) was dissolved in ethanol and 3-bromo-5-nitro salicylaldehyde (0.233 g, 1 mM) was dissolved in ethanol and added dropwise to the above solution with continuous stirring. The solution was then refluxed for another 30 min and the color of the solution changed to yellow. Then, the solution was kept undisturbed for six hours at room temperature. The formed orange crystalline product was filtered off, washed several times with ethanol, and dried in vacuum (10^{-2} torr) (purity was checked by TLC). Needle-like crystals suitable for XRD analysis were grown by recrystallization from ethanol. Yield 91%; m.p. 198 °C. IR (KBr pellet, cm^{-1}): 3460(m), 1603(s), 1444(s), 1329(s), 735(s). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 11.39 (1H, s, OH), 8.55 (1H, s, CH=N), 8.35 (1H, s, Ar–H), 8.25 (1H, s, Ar–H), 6.91–7.42 (9H, m, Ar–H) and 4.11 (2H, s, CH_2). UV–vis (DCM, λ_{max}, nm): 310 (13,193), 473 (1625). Anal. Calcd for $C_{20}H_{15}O_3N_2SBr$: C, 54.18; H, 3.38; N, 6.32; S, 3.61. Found (%): C, 53.95; H, 3.27; N, 6.38; S, 3.59.

2.3. *Synthesis of complexes*

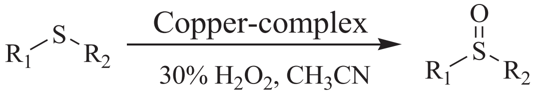
2.3.1. Preparation of NiL_2 (2). The ligand, **1** (0.884 g, 2 mM), was dissolved in boiling methanol (30 mL) followed by the addition of methanolic 2 mM sodium hydroxide. To the above solution, a solution of nickel chloride hexahydrate (0.237 g, 1 mM) dissolved in methanol (30 mL) was added. The solution was stirred for 40 min. The color of the solution changed to brown-black. The solution was kept undisturbed for 24 h. The black-brown needle-like crystal formed, suitable for X-ray diffraction, was filtered off, washed several times with 25% methanol–water solution to remove the impurities, and dried under vacuum (10^{-2} torr) (purity was checked by TLC). Yield 67.7%; m.p. 274 °C. IR (KBr, cm^{-1}): 1600(s), 1482(s), 1304(s), 756(s), 555(m), 510(m). UV–vis (DCM, λ_{max}, nm): 301 (1150), 387 (1445), 601 (232). Anal. Calcd for $C_{40}H_{28}O_6N_4S_2Br_2Ni$: C, 50.92; H, 2.97; N, 5.94; S, 3.39. Found (%): C, 50.98; H, 2.87; N, 5.81; S, 3.28.

2.3.2. Preparation of CuL_2 (3). The preparation of **3** follows the same procedure as that of **2** except that copper nitrate trihydrate (0.241 g, 1 mM) was used. When the color of the solution changed to dark green, it was kept undisturbed for 24 h and green microcrystals were obtained (purity was checked by TLC). Yield 68.6%; m.p. 252 °C. IR (KBr, cm^{-1}): 1598(s), 1455(s), 1315(m), 750(s), 566(m), 524(m). UV–vis (DCM, λ_{max}, nm): 263 (1681), 363 (2071), 617 (117). Anal. Calcd for $C_{40}H_{28}O_6N_4S_2Br_2Cu$: C, 50.66; H, 2.95; N, 5.91; S, 3.37. Found (%): C, 50.54; H, 2.86; N, 5.96; S, 3.32.

2.3.3. Preparation of CoL_2 (4). The preparation of **4** follows the same procedure as that of **2** and **3** except that cobalt chloride hexahydrate (0.237 g, 1 mM) was used. When the color of the solution changes to black, it was kept undisturbed for 2 days. Black microcrystals were obtained (purity was checked by TLC). Yield 58.7%; m.p. 264 °C. IR (KBr, cm^{-1}): 1592(s), 1470(s), 1318(m), 752(s), 560(s), 524(m). UV–vis (DCM, λ_{max}, nm): 289 (2264), 372 (1766), 598 (652). Anal. Calcd for $C_{40}H_{28}O_6N_4S_2Br_2Co$: C, 50.91; H, 2.97; N, 5.94; S, 3.39. Found (%): C, 50.98; H, 2.87; N, 5.83; S, 3.35.

2.4. *Oxidation of thioether*

The present work also describes the catalytic oxidation of sulfide to sulfoxide and sulfone using the synthesized copper(II) complex. The H_2O_2-induced catalytic activity of copper(II) complex towards oxidation of sulfide under atmospheric conditions is shown in equation (1). A 30% hydrogen peroxide solution (5 mM) was added to a solution containing the sulfide (2 mM), complex (0.5 M %), and 2 ml CH_3CN [14]. The reaction mixture was stirred at room temperature until the completion of reaction and it is monitored using TLC. After complete conversion of the reactant, the product was extracted with EtOAc and washed with water. The organic layer was dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by chromatography (eluting with 1:1 hexane/EtOAc). Here, we have used hydrogen peroxide as an oxidant in the presence of the copper (II) complex for the oxidation of sulfide to their sulfoxide with high yield. A series of solvents were employed for the reaction; acetone, DCM, and methanol give good yield with slightly longer reaction times. The conversion was calculated on the basis of the isolated yields shown in table 1.



...(1)

2.5. *X-ray crystallography*

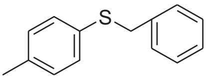
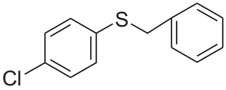
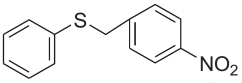
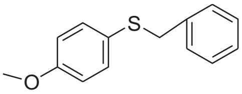
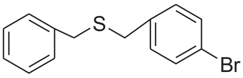
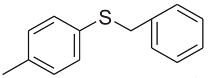
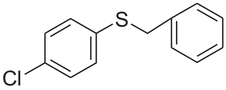
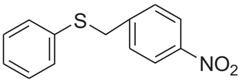
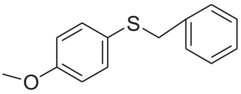
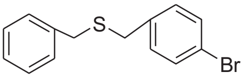
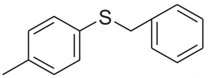
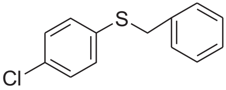
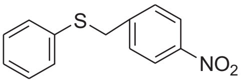
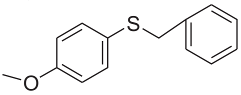
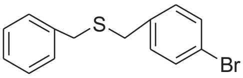
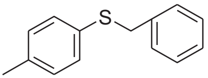
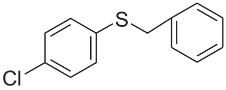
Single-crystal X-ray diffraction data for **1** and **2** were collected at 100 K with Mo Kα radiation (λ = 0.71073 Å) using a Bruker Smart Apex II CCD diffractometer equipped with a graphite monochromator. SMART [17] software was used for data collection and also for indexing the reflections and determining the unit cell parameters. Collected data were integrated using SAINT [17]. Structures were solved by direct methods and refined by full-matrix least-squares calculations using SHELXTL [18] software. Absorption corrections were done by multiscan method (SADABS) [17]. All non-H atoms were refined in the anisotropic approximation against F^2 of all reflections. The H-atoms were placed at their calculated positions and refined in the isotropic approximations. We have used SIMU, DELU, and ISOR commands to find reasonable ADP restraints for C18, C8, and O3.

3. Results and discussion

3.1. *Synthesis and formulation*

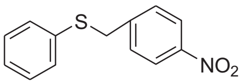
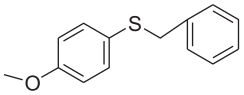
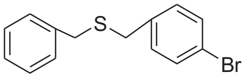
The new ONS donor Schiff base, HL, was synthesized by the reaction of 2-(benzylthio)aniline and 3-bromo-5-nitro salicylaldehyde in ethanol. The ligand was characterized by ^1H NMR, FTIR, and elemental analyses. Neutral nickel(II), copper(II), and cobalt(II)

Table 1. Oxidation of sulfide catalyzed by CuL_2 in different solvents in the presence of 30% H_2O_2.[a]

Entry	Substrate	Solvent	Time (h)	Sulfoxide[b] yield (%)	Sulfone[b] yield (%)
1		CH_3CN	5	92	6
2		CH_3CN	4	90	7
3		CH_3CN	5	88	9
4		CH_3CN	3	90	5
5		CH_3CN	4	89	8
1		CH_3COCH	8	90	8
2		CH_3COCH	7	89	9
3		CH_3COCH	8	85	12
4		CH_3COCH	6	86	10
5		CH_3COCH	7	85	9
1		CH_2Cl_2	7	91	7
2		CH_2Cl_2	6	88	9
3		CH_2Cl_2	7	87	10
4		CH_2Cl_2	5	88	8
5		CH_2Cl_2	6	82	10
1		CH_3OH	9	89	8
2		CH_3OH	8	87	10

(*Continued*)

Table 1. (*Continued*).

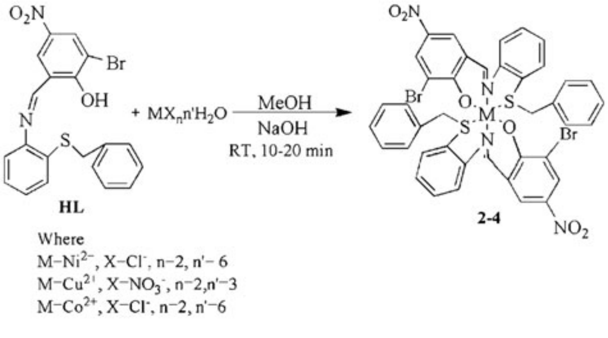
Entry	Substrate	Solvent	Time (h)	Sulfoxide[b] yield (%)	Sulfone[b] yield (%)
3		CH_3OH	9	85	9
4		CH_3OH	7	88	9
5		CH_3OH	8	8	10

[a]Reaction condition: substrate (2 mM), complex **1** (0.5 M %), and 30% H_2O_2 (5 mM) were stirred at room temperature in solvent (2 mL).
[b]Isolated yield.

complexes were obtained by the reaction of ligand HL with the corresponding metal salt. Elemental analyses indicate that metal ions react with the ligand in a 1 : 2 M ratio to afford the neutral complexes ML_2, where L represents the deprotonated mononionic (ONS) ligand. They are crystalline solids, stable in air, and have melting points in the range 238–296 °C. They are insoluble in organic solvents such as acetone, but soluble in DMF, DCM, and DMSO. The elemental analyses data of ligands **2**–**4** are in agreement with the structure of the ligand and complexes, respectively. The reaction scheme for the synthesis of metal complexes is given in scheme 1.

3.1.1. FTIR spectra. The FTIR spectrum of **1** exhibits a broad band at 3460 cm^{-1} due to ν(O–H), a strong band at 1603 cm^{-1} due to ν(C=N) and other strong bands from ν(C=C) to ν(C–S) at 1444 and 735 cm^{-1}, respectively.

The band for ν(OH) disappeared in the spectra of complexes indicating coordination of phenolic oxygen with metal. The complexes exhibit a band for ν(C=N) in the range 1592–1600 cm^{-1} showing a shift to lower frequencies by 11–3 cm^{-1} compared with ligand, which indicates coordination of ligand with metal via nitrogen. Moreover, phenolic ν(CO) stretch, which is shifted by 20–32 cm^{-1} towards lower wavenumbers in the complexes, indicates coordination of the phenolic oxygen [19]. Again, ν(C–S) in



Scheme 1. Synthesis of metal complexes.

complexes shifted to higher frequencies from 735 to 752–756 cm^{-1}, which is evidence of the M–S bond [20]. The appearance of new bands at 566–555 and 524–510 cm^{-1} in complexes supported coordination with oxygen and nitrogen, respectively [19, 20].

3.1.2. Electronic spectra. The ligand shows sharp bands at 473 nm and 310 nm, which are assigned to n–π* transition of the imine moiety and π–π* transition of phenyl rings, respectively. The electronic spectrum of **2** exhibited three bands, 601, 387, and 301 nm, which may be assigned to d–d, n–π*, and π–π* transitions, respectively. The electronic spectrum of **3** shows a band at 617 nm due d–d transition and at 363 nm which may be due to n–π* band of the imine. Complex **3** has another band centered at 263 nm which is assigned to π–π* transition of phenyl rings [5, 21]. Complex **4** shows bands at 598, 372, and 289 which may be assigned to d–d, n–π*, and π–π* transitions, respectively.

3.1.3. Molar conductivity. The molar conductivities of **2** are at 24.5 s cm^2 M^{-1}, **3** at 19.4 s cm^2 M^{-1}, and **4** at 29.2 s cm^2 M^{-1}. Molar conductivities of 19.4–29.2 s cm^2 M^{-1} in acetonitrile revealed that all the complexes are non-electrolytes [22].

3.1.4. Magnetic measurement. The magnetic moment (μ_{eff}) for **2** is 2.79 BM, close to the spin-only value of 2.83 BM expected for a d^8 system in O$_h$ symmetry. The value obtained

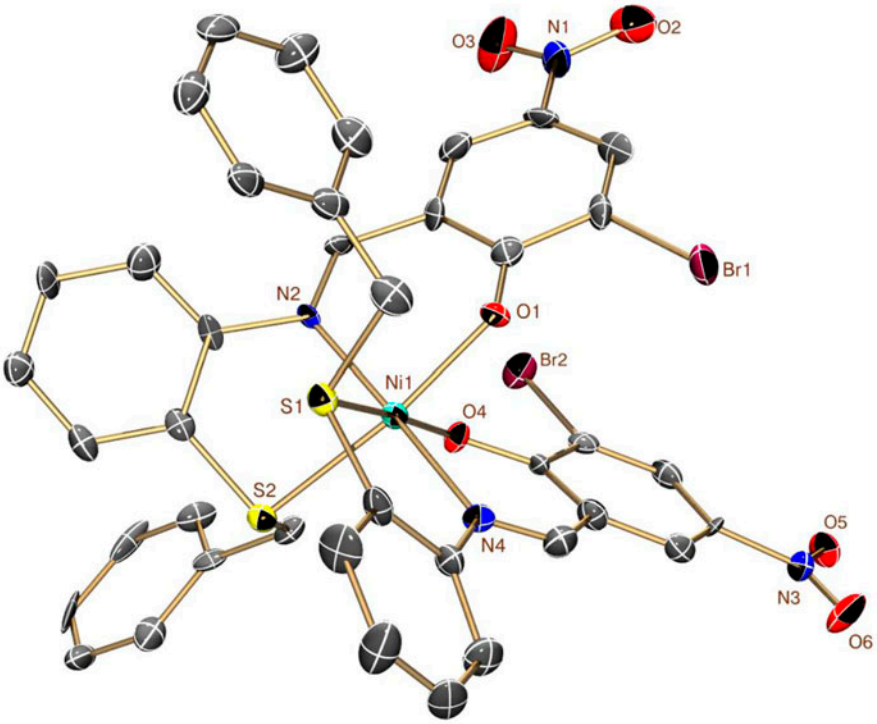


Figure 1. Molecular structure of nickel complex shown with 50% probability ellipsoids. Hydrogens are omitted for clarity.

for **3** is in the expected range (1.61 BM) for a d^9 configuration [5]. The magnetic moment of **4** is 4.96 BM, consistent with the value suggested for high-spin octahedral Co(II) complex.

3.2. *Molecular structure*

The ORTEP view with atom numbering of nickel complex is shown in figure 1. The crystal structure of the ligand from unsubstituted aldehyde and its metal complexes is reported by Kalita *et al.* [5]. Here, we report a ligand from substituted aldehyde. In this case, the benzyl part is tilted outside from the basal plane. This may be due to the presence of bulky bromine and nitro. In **1**, N-methylenebenzenamine and 2-hydroxy-3-bromo-5-nitro phenyl rings are planar. The bromine and nitro of the ligand repel the benzyl part of the ligand, resulting in the benzyl part being tilted outside from the coordination sphere. The imine double bond has a *trans* geometry, indicating that coordination of the ligand with metal ions must be through ONS donors. The C=N bond length is in the expected range for a typical C=N bond length of imines. $N_2S_2O_2$ donor set meridionally made the coordination sphere. The nickel coordinated with two deprotonated oxygens, two thioether sulfurs, and two imine nitrogens of the tridentate ligand to form the chelate complex. The two oxygens bonded to nickel are *cis*. The two thioether sulfurs also occupy *cis* positions in the basal plane and the two imine nitrogens are in axial positions [23]. The distortion around nickel in the complex

Table 2. Crystal structure and structure refinement details for ligand and nickel complex.

Compound	Ligand	Nickel complex
CCDC entry no.	980688	980689
Empirical formula	$C_{20}H_{15}BrN_2O_3S$	$C_{40}H_{28}Br_2N_4NiO_6S_2$
Formula weight	443.31	943.31
T (K)	100(2)	100(2)
λ (Å)	0.71073	0.71073
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	$Pca2_1$
Unit cell dimensions		
a (Å)	8.6540(2)	21.1839(7)
b (Å)	13.9908(3)	11.0378(4)
c (Å)	14.7051(3)	16.4508(6)
α (°)	90.00	90.00
β (°)	95.2770(10)	90.00
γ (°)	90.00	90.00
V (Å^3)	1772.89(7)	3846.6(2)
Z	4	4
D_{Calcd} (Mg/m^3)	1.661	1.629
μ (mm^{-1})	2.461	2.744
F(0 0 0)	896	1896
Crystal size (mm^3)	0.58 × 0.49 × 0.41	0.37 × 0.13 × 0.11
θ Range for data collection (°)	2.01–38.78	1.92–27.48
Index ranges	$-15 \leq h \leq 15$ $-24 \leq k \leq 22$ $-25 \leq l \leq 25$	$-19 \leq h \leq 27$ $-5 \leq k \leq 14$ $-21 \leq l \leq 13$
Completeness (%)	98.8	85.0
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Max and min transmissions	0.4319 and 0.3294	0.7523 and 0.4301
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	10,037/0/248	8816/0/496
Goodness-of-fit on F^2	1.063	1.002
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0321$, $wR_2 = 0.0741$	$R_1 = 0.0613$, $wR_2 = 0.1334$
R indices (all data)	$R_1 = 0.0956$, $wR_2 = 0.0944$	$R_1 = 0.1220$, $wR_2 = 0.1955$

Table 3. Selected bond lengths (Å) and angles (°) for **2**.

Ni1–S1	2.399(3)	S1–Ni1–O4	175.0(2)
Ni1–S2	2.464(3)	S1–Ni1–O1	95.1(2)
Ni1–N2	2.028(9)	S1–Ni1–N4	85.1(3)
Ni1–N4	2.026(8)	S1–Ni1–N2	92.4(2)
Ni1–O1	2.010(7)	S1–Ni1–S2	88.11(10)
Ni1–O4	1.998(7)	S2–Ni1–N2	81.6(2)
S1–C33	1.750(11)	N4–Ni1–S2	97.3(3)
S1–C34	1.849(11)	N4–Ni1–N2	177.3(4)
S2–C13	1.772(11)	O1–Ni1–N4	91.6(3)
S2–C14	1.837(11)	O1–Ni1–S2	170.8(2)
N2–C7	1.300(12)	O1–Ni1–N2	89.6(3)
N2–C8	1.419(12)	O4–Ni1–O1	85.5(3)
N4–C27	1.291(13)	O4–Ni1–N4	89.9(3)
N4–C28	1.457(13)	O4–Ni1–S2	92.2(2)
O1–C1	1.298(13)	O4–Ni1–N2	92.6(3)
O4–C21	1.279(11)		

is small as judged from the spread of the *cis*-angles 85.1(3)°–92.4(2)° and the *trans* angles 170.8(2)° and 175.0(2)°. The Ni(II)–S (thioether) distances are 2.399(3) and 2.464(3) Å and are quite typical of Ni(II)–S (thioether) distances observed in complexes of multidentate ligands with one or more thioether S donors [24]. The Ni(II)–N (imino) distances of the complex (2.028(9) and 2.026(8) Å) are well within the range when compared with those of typical Ni(II)–N (imino) distances. The Ni(II)–O bond lengths 1.998(7) and 2.010(7) Å are in the range of octahedral complexes [25]. The bond length of S2–C13, 1.772(11) Å increases compared with S2–C13 bond of the free ligand, 1.7610(11) Å. The S2–C14 bond also increases showing a weak coordination of S with Ni. Imine bond lengths of complex N2–C7, 1.300(12) Å and N4–C27, 1.291(13) Å increase compared with the free ligand, 1.2863(14) Å, due to delocalization of the imine-bonded electrons. Interestingly, the O1–C1, 1.298(13) Å and O4–C21, 1.279(11) Å bond lengths decrease compared with the corresponding bond length of free ligand O1–C1, 1.3348(13) Å, due to delocalization of the negative charge of oxygen to the ligand backbone giving partial double bond character.

Detailed crystallographic data are given in table 2. Selected bond lengths and angles of the nickel complex are given in table 3.

4. Conclusion

A new sterically and electronically tunable hydroxyl-imine-thioether tridentate ligand and three mononuclear Ni(II), Cu(II), and Co(II) complexes were prepared. These compounds were characterized by different physico-chemical analyses. Single-crystal X-ray diffraction data, magnetic susceptibility measurements, and electronic spectra revealed slightly distorted octahedral arrangement for **2**. The copper complex showed good catalytic activity towards oxidation of thioether.

Supplementary material

CCDC 980688 and 980689 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223 336 033; or E-mail: deposit@ccdc.cam.ac.uk.

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