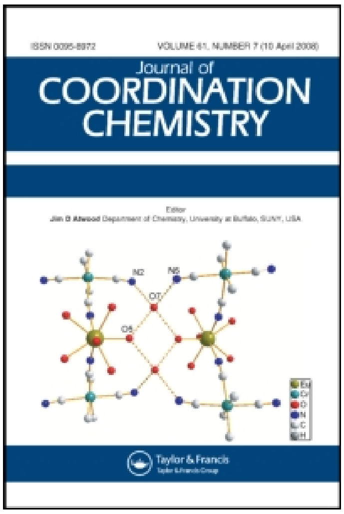

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Synthesis and spectral studies of Cu(II), Ni(II), Co(II) macrocyclic complexes by template reaction of (±)-<i>trans</i>-1,2-diaminocyclohexane, metal(II) nitrate and salicylaldehyde derivatives

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Synthesis and spectral studies of Cu(II), Ni(II), Co(II) macrocyclic complexes by template reaction of (±)-*trans*-1,2-diaminocyclohexane, metal(II) nitrate and salicylaldehyde derivatives

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Six new macrocyclic complexes were synthesized by template reactions of salicylaldehyde derivatives with (±)-*trans*-1,2-diaminocyclohexane and metal(II) nitrates. The metal to ligand ratio was 1 : 1. The Cu(II) complexes are proposed to be square planar and the Ni(II) and Co(II) complexes are proposed to be tetrahedral. The complexes are 1 : 2 electrolytes as shown by their molar conductivities (Λ_M) in DMF. The metal complexes are characterized by elemental analyses, FT-IR, UV–Vis, magnetic susceptibility measurements, molar conductivity measurements and mass spectra.

Keywords: Schiff-base complexes; Macrocyclic complexes; Salicylaldehyde derivatives

1. Introduction

Schiff bases and their metal complexes play a key role in understanding coordination chemistry of transition metal ions [1]. Schiff-base metal complexes have industrial, antifungal, and biological applications [2]. A large number of reports are available on the chemistry and the biological activities of transition metal complexes containing O,N and S,N donor atoms [3]. Schiff-base macrocycles were among the first artificial metal macrocyclic complexes to be synthesized. Metal complexes of macrocyclic ligands can be used as models for biological macrocyclic systems, metalloporphyrins (hemoglobin, myoglobin, cytochromes, chlorophyls), corrins (vitamin B12) and antibiotics (valinomycin, nonactin) [4, 5]. The stability of macrocyclic metal complexes depends upon a number of factors, including the number and type of donor atoms in the ligand and their relative positions within the macrocyclic skeleton, as well as the number and size of the chelate rings formed on complexation. For transition metal ions, features such as the nature and magnitude of crystal-field effects also play an important role [6]. Synthetic macrocycles have a wide range of different molecular topologies and sets of donor atoms [7–13]. The chemical properties of macrocyclic complexes can be tuned to force metal ions to adopt unusual coordination geometries. Synthesis of Schiff-base complexes is

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achieved through template reactions [14–17] or transmetallation reactions [18–20] which are used when the transition metal cations are ineffective as templates. In the present work, I have synthesized six macrocyclic Schiff-base complexes by template reaction of salicylaldehyde derivatives, $Cu(NO_3)_2 \cdot 3H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Co(NO_3)_2 \cdot 6H_2O$ and (±)-*trans*-1,2-diaminocyclohexane, respectively. Spectral and magnetic properties of the new compounds were studied.

2. Experimental

The salicylaldehyde derivatives used in the synthesis were prepared from salicylaldehyde, 1,2-dibromoethane or 1,3-dibromopropane and K_2CO_3 as shown in figure 1 and according to the literature method [21,22]. All the chemicals and solvents were of analytical grade and used as received. Elemental analyses were carried out on a LECO CHNS model 932 elemental analyzer. IR spectra were recorded on a PERKIN-ELMER SPECTRUM RX1 FTIR spectrophotometer as KBr pellets in the range 4000–400 cm^{-1}. Electronic spectral studies were conducted on a SHIMADZU model 160 UV Visible spectrophotometer from 200–900 nm. Molar conductivities were measured with a WTW LF model 330 conductivity meter using prepared solution of the complexes in DMF. Magnetic susceptibilities were determined on a Sherwood Scientific Magnetic Susceptibility Balance (Model MK1) at room temperature (20°C) using $Hg[Co(SCN)_4]$ as a calibrant; diamagnetic corrections were calculated from Pascal's constants [23]. Electrospray ionization mass spectrometric analysis (ESI–MS) was obtained on the AGILENT 1100 MSD spectrometer and are described in Supplementary Material.

2.1. *Synthesis of complexes*

To a stirred solution of salicylaldehyde derivatives (2 mmol) and $M(NO_3)_2 \cdot nH_2O$ (2 mmol) in methanol (60 mL) was added dropwise (±)-*trans*-1,2-diaminocyclohexane (2 mmol) in methanol (40 mL). After the addition was complete, stirring was continued for 2 h, then precipitate was filtered and washed with methanol and dried in air (M=Cu; $n = 3$ or M=Co, Ni; $n = 6$).

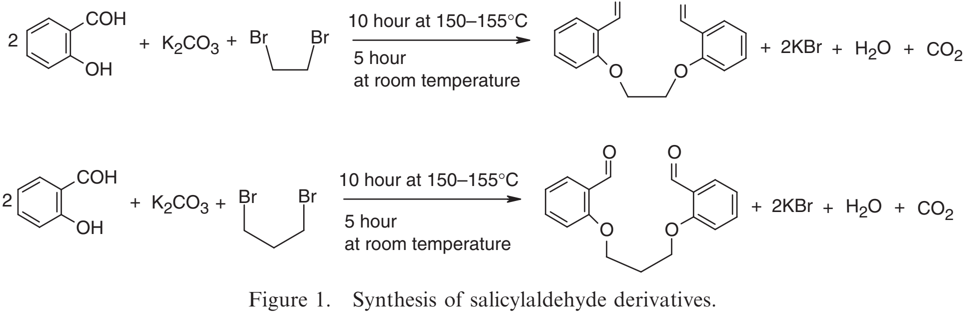


Figure 1. Synthesis of salicylaldehyde derivatives.

2.2. *Characterization of [CuL^1](NO$_3$)$_2$·4H$_2$O*

Yield: 0.40 g (32.9%). Anal. Calcd for $CuC_{22}H_{24}N_4O_8 \cdot 4H_2O$: C, 43.42; H, 5.26; N, 9.21. Found: C, 44.17; H, 5.64; N, 9.32. Selected IR data (KBr, ν cm^{-1}): 3338 ν(H$_2$O), 3131 ν(Ar–CH), 2966, 2882 ν(Alph.–CH), 1635 ν(C=N), 1385, ionic ν(NO_3^-), 1277, 1242 ν(Ar–O), 1143, 1042 ν(R–O), 754 ν(Substituted benzene), 527 ν(Cu–O), 479 ν(Cu–N). $\Lambda_M = 193\ \Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 245, 263, 323, 569. Mass spectrum (*m*/*z*): 536 [[CuL^1](NO$_3$)$_2$]$^+$, $\mu_{eff} = 1.81$ B.M.

2.3. *Characterization of [CoL^1](NO$_3$)$_2$·4H$_2$O*

Yield: 0.36 g (29.9%). Anal. Calcd for $CoC_{22}H_{24}N_4O_8 \cdot 4H_2O$: C, 43.78; H, 5.31; N, 9.29. Found: C, 44.14; H, 5.57; N, 9.31. Selected IR data (KBr, ν cm^{-1}): 3369 ν(H$_2$O), 3071 ν(Ar–CH), 2939, 2886 ν(Alph.–CH), 1636 ν(C=N), 1384, ionic ν(NO_3^-), 1292, 1243 ν(Ar–O), 1162, 1042, ν(R–O), 752 ν(Substituted benzene), 519 ν(Co–O), 506 ν(Co–N). $\Lambda = 142\ \Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 239, 270, 310, 655. Mass spectrum (*m*/*z*): 530 [[CoL^1](NO$_3$)$_2$–H]$^+$. $\mu_{eff} = 3.85$ B.M.

2.4. *Characterization of [NiL^1](NO$_3$)$_2$·5H$_2$O*

Yield: 0.34 g (27.4%). Anal. Calcd for $NiC_{22}H_{24}N_4O_8 \cdot 5H_2O$: C, 42.51; H, 5.48; N, 9.02. Found: C, 42.14; H, 5.62; N, 8.83. Selected IR data (KBr, ν cm^{-1}): 3364 ν(H$_2$O), 3072 ν(Ar–CH), 2936, 2833 ν(Alph.–CH), 1635 ν(C=N), 1384, ionic ν(NO_3^-), 1285, 1242 ν(Ar–O), 1193, 1090, ν(R–O), 755 ν(Substituted benzene), 515 ν(Ni–O), 482 ν(Ni–N). $\Lambda = 154\ \Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 234, 262, 321, 695. Mass spectrum (*m*/*z*): 532 [[NiL^1](NO$_3$)$_2$+H]$^+$. $\mu_{eff} = 2.73$ B.M.

2.5. *Characterization of [CuL^2](NO$_3$)$_2$·3H$_2$O*

Yield: 0.47 g (38.9%). Anal. Calcd for $CuC_{23}H_{26}N_4O_8 \cdot 3H_2O$: C, 45.70; H, 5.30; N, 9.27. Found: C, 45.84; H, 5.42; N, 9.17. Selected IR data (KBr, ν cm^{-1}): 3333 ν(H$_2$O), 3051 ν(Ar–CH), 2936, 2852 ν(Alph.–CH), 1635 ν(C=N), 1384, ionic ν(NO_3^-), 1286, 1244 ν(Ar–O), 1175, 1051, ν(R–O), 751 ν(Substituted benzene), 518 ν(Cu–O), 481 ν(Cu–N). $\Lambda = 161\ \Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 248, 288, 319, 662. Mass spectrum (*m*/*z*): 551 [[CuL^2](NO$_3$)$_2$+H]$^+$. $\mu_{eff} = 2.09$ B.M.

2.6. *Characterization of [CoL^2](NO$_3$)$_2$·2H$_2$O*

Yield: 0.44 g (37.9%). Anal. Calcd for $CoC_{23}H_{26}N_4O_8 \cdot 2H_2O$: C, 47.50; H, 5.16; N, 9.64. Found: C, 47.61; H, 5.27; N, 9.62. Selected IR data (KBr, ν cm^{-1}): 3348 ν(H$_2$O), 3119 ν(Ar–CH), 2926, 2863 ν(Alph.–CH), 1634 ν(C=N), 1383, ionic ν(NO_3^-), 1311, 1238 ν(Ar–O), 1091, 1039, ν(R–O), 752 ν(Substituted benzene), 531 ν(Co–O), 496 ν(Co–N). $\Lambda = 172\ \Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 239, 268, 321, 651. Mass spectrum (*m*/*z*): 547 [[CoL^2](NO$_3$)$_2$+2H]$^+$. $\mu_{eff} = 3.88$ B.M.

2.7. *Characterization of $[NiL^2](NO_3)_2 \cdot 4H_2O$*

Yield: 0.52 g (42.1%). Anal. Calcd for $NiC_{23}H_{26}N_4O_8 \cdot 4H_2O$: C, 44.73; H, 5.51; N, 9.08. Found: C, 45.14; H, 5.65; N, 9.13. Selected IR data (KBr, $\nu\,cm^{-1}$): 3351 $\nu(H_2O)$, 3066 ν(Ar–CH), 2936, 2875 ν(Alph.–CH), 1635 ν(C=N), 1384, ionic $\nu(NO_3^-)$, 1287, 1242 ν(Ar–O), 1190, 1045, ν(R–O), 754 ν(Substituted benzene), 526 ν(Ni–O), 492 ν(Cu–N). $\Lambda = 166\,\Omega^{-1}\,cm^2\,mol^{-1}$ (in DMF). UV–Vis (λ_{max}, nm) in DMF: 240, 278, 308, 665. Mass spectrum (*m*/*z*): 545 $[NiL^2](NO_3)_2]^+$. $\mu_{eff} = 2.89$ B.M.

3. Results and discussion

3.1. *Macrocyclic Schiff-base complexes*

The reaction between 1,2-*bis*(2-formylphenyl)ethane or 1,3-*bis*(2-formylphenyl)propane, metal(II) nitrate and (±)-*trans*-1,2-diaminocyclohexane in methanol, gave [1 + 1] macrocyclic Schiff-base complexes. Minor product, diamine complexes and unreacted dialdehyde, metal salt and diamine were removed with methanol. The macrocyclic complexes were characterized by elemental analysis, mass and IR spectrometry. The mass spectra of the complexes play an important role in confirming the monomeric [1 + 1] (dicarbonyl and diamine) nature (table 1). Crystals were unsuitable for single-crystal X-ray structural determination and are insoluble in most common solvents including water, methanol, ethanol, ethyl acetate and/or acetonitrile.

3.2. *FTIR spectra*

The characteristic infrared spectral data are given in the experimental section. Broad bands at ca $3350\,cm^{-1}$ in the spectra of all the complexes can be attributed to stretching vibrations of water $\upsilon(H_2O)$ [24]. A strong band in the IR spectra of the complexes at $1636–1634\,cm^{-1}$ is attributed to the υ(C=N) stretch, indicating coordination of the azomethine nitrogen to metal [25]. The absorptions of the nitrate ions, at ca 1460–1452 (ν_5), 1300 (ν_1) and 1040 (ν_2) cm^{-1}, suggest bidentate nitrate groups and an intense band at ca $1384\,cm^{-1}$ attributable to ionic nitrate, is also present [26]. The spectra of all the complexes are dominated by bands at $2965–2855\,cm^{-1}$ due to ν(Alph.–CH) groups [27]. Conclusive evidence of the bonding is also shown by the observation that new bands in the IR spectra of the complexes at $531–498\,cm^{-1}$ assigned to ν(M–O) and $506–346\,cm^{-1}$ assigned to ν(M–N) stretching vibrations [28–30] (table 2).

3.3. *Electronic spectra*

Electronic absorption spectral data of the complexes in dimethylformamide (DMF) at room temperature, presented in the experimental section, show four bands in the visible-ultraviolet region. Absorption bands below ca 300 nm are practically identical and can be attributed to $\pi \rightarrow \pi^*$ transitions in the benzene ring and azomethine (–C=N) groups. Absorption bands observed at ca 300–325 nm are due to $n \rightarrow \pi^*$ transitions of imine [31]. The general character of these spectra is very similar to that of the corresponding complexes of unsymmetrical disubstituted phenoxy groups,

Table 1. Physical characterization, analytical, molar conductance and mass data of the complexes.

Compound	(calcd) Found %C	%H	%N	Λ_M (ohm^{1}cm^{2}mol^{-1})	Formula Weight	MS/EI	Assigment	μeff (BM)
$[CuL^1][NO_3]_2 \cdot 4H_2O$	(43.42) 44.17	(5.26) 5.64	(9.21) 9.32	193	608	536	**$[[CuL^1](NO_3)_2]^+$**	1.81
$[CoL^1][NO_3]_2 \cdot 4H_2O$	(43.78) 44.14	(5.31) 5.57	(9.29) 9.31	142	603	530	**$[[CoL^1](NO_3)_2H]^+$**	3.85
$[NiL^1][NO_3]_2 \cdot 5H_2O$	(42.51) 42.14	(5.48) 5.62	(9.02) 8.83	154	621	532	**$[[NiL^1](NO_3)_2{+}H]^+$.**	2.73
$[CuL^2][NO_3]_2 \cdot 3H_2O$	(45.70) 45.84	(5.30) 5.42	(9.27) 9.17	161	604	551	**$[[CuL^2](NO_3)_2{+}H]^+$**	2.09
$[CoL^2][NO_3]_2 \cdot 2H_2O$	(47.50) 47.61	(5.16) 5.27	(9.64) 9.62	172	581	547	**$[[CoL^2](NO_3)_2{+}2H]^+$**	3.88
$[NiL^2][NO_3]_2 \cdot 4H_2O$	(44.73) 45.14	(5.51) 5.65	(9.08) 9.13	166	617	545	**$[NiL^2](NO_3)_2]^+$**	2.89

Table 2. IR (cm^{-1}) spectral data for the complexes.

Compound	$\nu(H_2O)$	$\nu(C{=}N)$	Ionic $\nu(NO_3^-)$	ν(M–O)	ν(M–N)
$[CuL^1][NO_3]_2 \cdot 4H_2O$	3338 s	1635 m	1385 m	527 w	479 w
$[CoL^1][NO_3]_2 \cdot 4H_2O$	3369 s	1636 m	1384 m	519 w	506 w
$[NiL^1][NO_3]_2 \cdot 5H_2O$	3364 s	1635 m	1384 m	515 w	482 w
$[CuL^2][NO_3]_2 \cdot 3H_2O$	3333 s	1635 m	1384 m	518 w	481 w
$[CoL^2][NO_3]_2 \cdot 2H_2O$	3348 s	1634 m	1383 m	531 w	496 w
$[NiL^2][NO_3]_2 \cdot 4H_2O$	3349 s	1635 m	1384 m	526 w	492 w

m: medium, s: strong, w: weak.

probably because metal-to-ligand charge transfer and ligand-to-metal charge transfer transitions have similar energy differences [32]. The electronic spectra of the Cu(II) complexes shows an absorption at 53–713 nm attributed to the $^2E_g \rightarrow {}^2T_{2g}$ transitions, characteristic for square-planar geometry [23]. The electronic spectra of the Ni(II) and Co(II) complexes shows an absorption at 555–680 nm attributed to the $^2E_g \rightarrow {}^2T_{2g}$ transitions, characteristic for tetrahedral geometry [32], and exhibit solvent dependence behavior. The observed red shifts in the low-energy d–d band of Ni(II) and Co(II) metal complexes in DMF can be interpreted in terms of weakening of ligand field strength [22].

3.4. *Magnetic measurements*

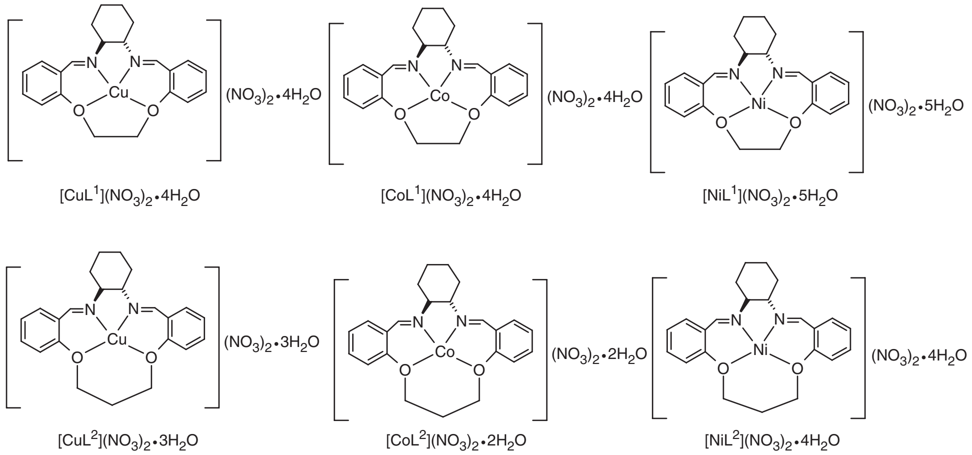
The metal-ligand mole ratio was 1 : 1 according to elemental analysis. The magnetic moments of Cu(II) complexes at room temperature are in the range 1.81–2.09 BM, typical for Cu(II) complexes having one unpaired electron. The magnetic moments of the Ni(II) complexes at room temperature are in the range 2.73–2.89 BM, typical for Ni(II) complexes having two unpaired electrons [23, 28, 32]. The room temperature magnetic moments of 3.85–3.88 BM determined for Co(II) complexes are close to the spin-only magnetic moments ($\mu = 3.87$ BM) for three unpaired electrons. These results and the absorption bands observed for electronic spectra of Ni(II) and Co(II) complexes support tetrahedral geometry for Ni(II) and Co(II) complexes.

3.5. *Molar conductivity*

The molar conductivities (Λ_M) of the complexes are 2 : 1 electrolytes in DMF (dimethyl formamide) at 10^{-3} M, in the range 140–190 $\Omega^{-1}\,cm^2\,mol^{-1}$ for 1 : 2 electrolytes [23, 32, 33].

4. Conclusion

Six Schiff-base macrocyclic complexes were prepared and characterized by elemental analyses, FTIR and UV–Vis spectra, conductivity measurements, magnetic



$[CuL^1](NO_3)_2{\cdot}4H_2O$ $[CoL^1](NO_3)_2{\cdot}4H_2O$ $[NiL^1](NO_3)_2{\cdot}5H_2O$

$[CuL^2](NO_3)_2{\cdot}3H_2O$ $[CoL^2](NO_3)_2{\cdot}2H_2O$ $[NiL^2](NO_3)_2{\cdot}4H_2O$

Figure 2. Suggested structure of the complexes.

susceptibilities and mass spectra. General structures of the complexes are shown in figure 2. The Ni(II) and Co(II) complexes are tetrahedral and Cu(II) complexes are square planar.

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