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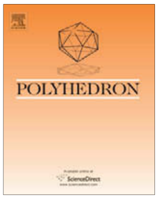

Synthesis and crystal structure characterization of iron(II), cobalt(II) and nickel(II) complexes with 1,3-bis(2-pyridylmethylthio)propane

Animesh Patra [a], Sandipan Sarkar [a], Michael G.B. Drew [b], Ennio Zangrando [c], Pabitra Chattopadhyay [a,*]

[a] Department of Chemistry, Burdwan University, Golapbag, Burdwan 713 104, India
[b] Department of Chemistry, Reading University, Reading RG6 6AD, Berks, UK
[c] Dipartimento di Scienze Chimiche, Via Licio Giorgieri 1, 34127 Trieste, Italy

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ABSTRACT

Three new mononuclear complexes of nitrogen–sulfur donor sets, formulated as $[Fe^{II}(L)Cl_2]$ (**1**), $[Co^{II}(L)Cl_2]$ (**2**) and $[Ni^{II}(L)Cl_2]$ (**3**) where L = 1,3-bis(2-pyridylmethylthio)propane, were synthesized and isolated in their pure form. All the complexes were characterized by physicochemical and spectroscopic methods. The solid state structures of complexes **1** and **3** have been established by single crystal X-ray crystallography. The structural analysis evidences isomorphous crystals with the metal ion in a distorted octahedral geometry that comprises NSSN ligand donors with *trans* located pyridine rings and chlorides in *cis* positions. In dimethylformamide solution, the complexes were found to exhibit Fe^{II}/Fe^{III}, Co^{II}/Co^{III} and Ni^{II}/Ni^{III} quasi-reversible redox couples in cyclic voltammograms with $E_{1/2}$ values (versus Ag/AgCl at 298 K) of +0.295, +0.795 and +0.745 V for **1**, **2** and **3**, respectively.

1. Introduction

Considerable attention has been paid to the coordination chemistry of nitrogen–sulfur donor ligands towards transition metal ions [1–7] because these complexes can present many characteristics of related biological systems containing metal ions in the same coordination environment. Thus it is a challenge for chemists to design appropriate ligand frames with the purpose of synthesizing metal complexes with predetermined properties. Structural [8–17] and reactivity [18–20] studies could provide useful insight into the chemistry and biochemistry of bio-active molecules. The interest in the coordination chemistry of S-ligated transition metal complexes arises in part from the fact that they mimic the ligation of certain biomolecules in proteins.

The first crystal structure of [NiFe] hydrogenase enzyme extracted from '*Desulforibrio gigas*' showed the active site containing an unexpected disulfur bridged heterobimetallic Ni–Fe core, and in addition two cystein groups were bound to nickel [15–17]. For the modelling of the active site, imidazole and thioether containing ligands are most relevant. Several N_xS_y ligands have been designed where the nitrogen belongs to aromatic systems such as pyridine, imidazole and benzimidazole and their reactivity towards transition metals have been investigated, in particular with copper(II) to mimic blue copper proteins such as plastocyanine [10].

As part of our continuing interest on nitrogen–sulfur polydentate chelators [21], herein we report an account on the iron(II), cobalt(II) and nickel(II) complexes with 1,3-bis(2-pyridylmethylthio)propane (L) having two pyridinic-N and two thioether-S donors. Detailed structures of $[Fe^{II}(L)Cl_2]$ (**1**) and $[Ni^{II}(L)Cl_2]$ (**3**), established by single crystal X-ray crystallography, are also included. The study of the electrochemical behavior of all three complexes shows M^{II}/M^{III} quasi-reversible redox couples in the cyclic voltammograms.

2. Experimental

2.1. Materials and physical measurements

All chemicals and reagents were obtained from commercial sources and were used as received, unless otherwise stated. Solvents were distilled from an appropriate drying agent. The organic ligand 1,3-bis(2-pyridylmethylthio)propane (L) was synthesized following the procedure as stated in our earlier report [21].

The C, H, N elemental analyses were performed on a Perkin–Elmer model 2400 elemental analyzer. The metal contents in the complexes were analyzed by using a Varian Atomic Absorption spectrophotometer (AAS) model-AA55B, GTA equipped with a graphite furnace. The electronic absorption spectra were recorded on a JASCO UV–Vis/NIR spectrophotometer model V-570. IR spectra were obtained by a JASCO FT-IR spectrometer model 460 *plus* using KBr disks. Room temperature magnetic susceptibilities were recorded using a vibrating sample magnetometer PAR 155 model. Molar conductances (Λ_M) were measured in a systronics conductivity meter 304 model in dimethylformamide with a complex

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[*] Corresponding author. Tel.: +91 342 2558339; fax: +91 342 2530452.
E-mail address: pabitracc@yahoo.com (P. Chattopadhyay).

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concentration of ~10^{-3} mol L^{-1}. The redox behaviors of the complexes were examined on a computer controlled EG&G PAR model 270 VERSTAT electrochemical instrument using a Ag/AgCl electrode with TBAP as the supporting electrolyte. All the measurements were made at 298 K in dimethylformamide solution for a complex concentration range of 10^{-3}–10^{-4} mol L^{-1} using dry dinitrogen for 3–4 min in order to remove dissolved oxygen.

2.2. Preparation of the complexes

The complexes were synthesized following a common procedure, as described below, using the respective metal(II) chloride salts and 1,3-bis(2-pyridylmethylthio)propane (L) in a 1:1 mole ratio. To a methanolic solution (15 mL) of L (276.0 mg, 1.0 mmol), iron(II) chloride tetrahydrate (1.0 mmol) (for **1**), cobalt(II) chloride hexahydrate (1.0 mmol) (for **2**) or nickel(II) chloride hexahydrate (for **3**) was added dropwise, stirring the mixture for an hour. The reaction mixture was refluxed for 3 h and then kept at room temperature. The pure crystallized product was obtained from this solution by allowing slow evaporation of the solvent. The product was collected by filtration followed by washing with water and methanol, and dried in *vacuo*.

*[Fe(L)Cl$_2$] (**1**):* Yield: 70–75%; *Anal.* Calc. for $C_{15}H_{18}Cl_2FeN_2S_2$: C, 43.15; H, 4.31; N, 6.71; Fe, 13.42. Found: C, 43.18; H, 4.32; N, 6.79; Fe, 13.50%. IR (cm^{-1}): $\nu_{C=N}$ 1468; ν_{C-S} 758. Magnetic moment (μ, B.M.): 5.45. Conductivity (Λ_o, Ω^{-1} cm^2 mol^{-1}) in DMF: 45.

*[Co(L)Cl$_2$] (**2**):* Yield: 71–75%; *Anal.* Calc. for $C_{15}H_{18}Cl_2CoN_2S_2$: C, 42.86; H, 4.29; N, 6.67; Co, 14.03. Found: C, 43.12; H, 4.15; N, 6.5 1; Co, 13.91%. IR (cm^{-1}): $\nu_{C=N}$ 1472; ν_{C-S} 761. Magnetic moment (μ, B.M.): 4.91. Conductivity (Λ_o, Ω^{-1} cm^2 mol^{-1}) in DMF: 52.

*[Ni(L)Cl$_2$] (**3**):* Yield: 80–85%; *Anal.* Calc. for $C_{15}H_{18}Cl_2N_2NiS_2$: C, 42.85; H, 4.29; N, 6.67; Ni, 13.99. Found: C, 42.99; H, 4.18; N, 6.79; Ni, 14.08%. IR (cm^{-1}): $\nu_{C=N}$ 1470; ν_{C-S} 760. Magnetic moment (μ, B.M.): 3.08. Conductivity (Λ_o, Ω^{-1} cm^2 mol^{-1}) in DMF: 42.

2.3. X-ray crystal structure analysis

Single crystals of complexes **1** and **3** suitable for X-ray diffraction analysis were grown by slow evaporation of methanolic solutions at ambient temperature. Crystallographic data and details of the refinements are provided in Table 1. Diffraction data for **1** were collected at 150(2) K using the Oxford Diffraction X-Calibur CCD System (321 frames, counting time of 2 s, the crystal positioned at 50 mm from the CCD). The experiment for **2** was carried out at room temperature on a Nonius DIP-1030H system (45 frames, the crystal positioned at 90 mm from the image plate). Mo Kα radiation (λ = 0.71073 Å) was used for both data collections. Cell refinement, indexing and scaling of the data sets were carried out using programs CRYSALIS [22], DENZO and SCALEPACK [23]. An absorption correction was applied to **1** by the ABSPACK program [24]. Both structures were solved using direct methods with the SHELXS97 program and refined by the full-matrix least-squares method based on F^2 with all observed reflections [25]. Hydrogen atoms were fixed at geometrical positions. As the complexes were located on a crystallographic twofold axis, the central methylene C(8) atom results was disordered over two sites (0.5 occupancy each). All the calculations were performed using the WINGX System, Version 1.70.01 [26].

3. Results and discussion

3.1. Synthesis and characterization

The organic ligand 1,3-bis(2-pyridylmethylthio)propane (L) was synthesized by the reaction of the respective alkyl dithiol compound with 2-picolyl chloride in the presence of sodium ethoxide. The complexes were obtained in good yields from the reaction of the corresponding metal salt with an equimolar amount of the respective organic moiety in methanol medium. All the complexes are monomeric in nature and are sparingly soluble in common organic solvents. The complex conductivity measurements in dimethylformamide show conductance values in range 42–52Λ_o mol^{-1} cm^{-1}, which suggest that the complexes exist in solution as non-electrolytes. The magnetic moment (μ) at room temperature of iron(II) in (**1**) is 5.45 B.M., which indicates a high-spin octahedral iron(II) complex; the values of μ for the cobalt(II) and nickel(II) complexes are 4.91 and 3.08 B.M., respectively.

3.2. Structures of complexes **1** and **3**

The X-ray structural analyses evidence that the crystals of **1** and **3** are isomorphous, and an ORTEP view of complex **1** is shown in Fig. 1, together with the atom numbering scheme. The metal resides on a crystallographic twofold axis bisecting the Cl–M–Cl and S–M–S angles. Thus the methylene C(8) atom of the organic moiety is disordered over two positions (each of occupancy 0.5), and only one orientation is displayed in Fig. 1. The tetradentate NSSN ligand is wrapped around the metal in such a way that the pyridine rings are in *trans* positions and the metal ion completes the highly distorted octahedral coordination sphere with two chlorides located in *cis* positions. The present coordination pattern of L

Table 1
Crystallographic data for **1** and **3**.

	1	**3**
Empirical formula	$C_{15}H_{18}Cl_2FeN_2S_2$	$C_{15}H_{18}Cl_2N_2NiS_2$
Formula weight	417.18	420.04
Temperature (K)	150(2)	293(2)
Crystal system	orthorhombic	
Space group	*Pbcn*	
a (Å)	14.315(2)	14.312(3)
b (Å)	8.4443(9)	8.551(2)
c (Å)	14.5516(18)	14.678(3)
Volume ($Å^3$)	1759.0(4)	1796.3(7)
Z	4	
ρ_{calc} (g/cm^3)	1.575	1.553
$F(000)$	856	864
θ range (°)	2.80–30.00	2.77–29.64
μ (Mo Kα) (mm^{-1})	1.395	1.606
Unique reflections/data $I > 2\sigma(I)$	2528/1011	2402/1600
Final R_1/wR_2 indices [$I > 2\sigma(I)$]	0.0854/0.2129	0.0348/0.0943
Goodness-of-fit	0.891	0.888
$\Delta\rho$ (e A^{-3})	1.596, −1.604	0.491, −0.476

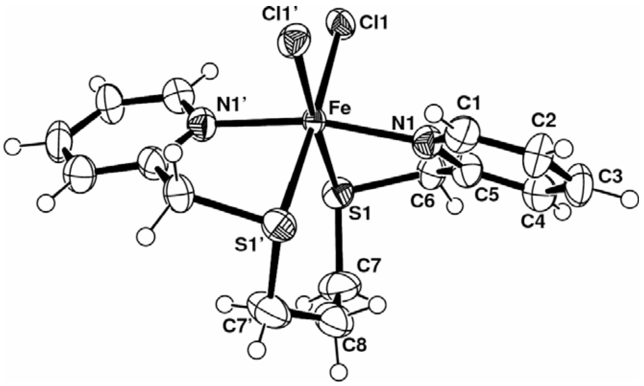

Fig. 1. ORTEP drawing (ellipsoids at 50% probability) of complex [Fe(L)Cl$_2$] (**1**). The molecule is located on a twofold axis and only one position for the disordered C(8) methylene is shown. The same atom numbering is applied to the isomorphous complex [Ni(L)Cl$_2$] (**3**), substituting Fe with Ni.

Table 2
Coordination bond distances (Å) and angles (°) for **1** and **3**.

	1, M = Fe	**3**, M = Ni
Bond distance (Å)		
M–N(1)	2.099(5)	2.104(2)
M–S(1)	2.440(2)	2.451(1)
M–Cl(1)	2.384(2)	2.392(1)
Bond angles (°)		
N(1)–M–N(1′)	167.4(3)	166.48(9)
N(1)–M–Cl(1′)	95.35(15)	95.50(5)
N(1)–M–Cl(1)	93.16(15)	93.63(5)
N(1)–M–S(1)	82.40(15)	82.22(5)
N(1)–M–S(1′)	89.23(15)	88.77(5)
Cl(1)–M–Cl(1′)	94.90(9)	94.88(4)
Cl(1)–M–S(1)	84.20(6)	84.38(3)
Cl(1)–M–S(1′)	177.51(5)	177.54(2)
S(1)–M–S(1′)	96.80(9)	96.45(3)

Primed atoms at $-x, y, -z + 1/2$.

was also detected in the neutral [Ni(L)(ONO$_2$)$_2$] derivative [27], but it is noteworthy that in the trigonal bipyramidal copper(II) complex [Cu(L)Cl]$^+$ [21] a pyridine-N and a thioether-S reside at the axial positions whereas the equatorial sites were occupied by the other N and S donors and by the chloride ligand. The coordination bond lengths and angles of **1** and **3**, listed in Table 2, show only minor differences related to the metal ionic radii. The M–N bond lengths are comparable within their e.s.d.'s (2.099(5) and 2.104(2) Å in **1** and **3**, respectively), while the values of the M–S and M–Cl distances differ by *ca.* 0.01 Å, being of 2.440(1), 2.451(1) Å and 2.384(2), 2.392(1) Å in **1** and **3**, respectively. The bond angles subtended at the metal differ by less than 1° in the two structures, but they deviate considerably from the ideal octahedral values and the N–M–N angle of 167° (mean value for the two structures) shows the highest deviation. In fact the pyridine rings of the ligand are bent with respect to the S$_2$Cl$_2$ plane, forming a dihedral angle of 80.57° (mean value), likely induced by ligand constraints upon coordination, while the dihedral angle made by the trans located pyridine planes is 55.3(2)° and 54.9(1)° in **1** and **3**, respectively. The bond distances and geometrical distortions of **3** agree with those found in [Ni(2,2′-(2,5-dithiahexamethylene) dipyridine)Cl$_2$] [28] while small differences are evidenced when compared with the disordered crystal structure of [Ni(L)(ONO$_2$)$_2$] [7].

3.3. Spectroscopic properties

The infrared spectra of all the complexes exhibit characteristic strong to medium intensity bands in the regions 1468–1472 and 758–761 cm^{-1}, which are assignable to $\nu_{C=N}$ and ν_{C-S}, respectively, in addition to the other characteristic bands of the organic moieties. This observation supports the presence of the ligand frame containing the pyridine ring and the C–S bond, and ν_{C-S} is generally observed in the free ligand in the range of 780–790 cm^{-1} [21].

Table 3
UV–Vis spectral and electrochemical data.[a]

Compound	UV–Vis data[a]	Electrochemical data[b]			
	λ (nm) (ε, dm^3 mol^{-1} cm^{-1})	E_{pc}[a] (V)	E_{pa}[a] (V)	$E_{1/2}$ (V)	Reversibility (I_{pc}/I_{pa})
1	212(9530), 261(8310), 332(3010), 475(975), 752(108)	+0.235	+0.355	+0.295	0.97
2	210(9000), 257(6300), 337(2820), 425(89), 560(186), 967(22)	+0.600	+0.990	+0.795	1.03
3	210(10230), 258(7430), 328(2915), 475(221), 685(520), 873(31)	+0.665	+0.825	+0.745	0.93

[a] In DMF.

[b] Data recorded in mV, at 298 K and scan rate 100 mV s^{-1}; $E_{1/2} = (E_{pc} + E_{pa})/2$; I_{pc}/I_{pa} is constant for scans in the range of 50–400 mV s^{-1}.

The electronic absorption spectra of complexes **1**–**3** were recorded at room temperature using dimethylformamide as the solvent and the data tabulated in Table 3. All the spectra in the high energy region (below 400 nm) exhibit bands corresponding to intramolecular $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions. In addition, characteristic bands for the complexes were detected as follows.

In the electronic spectrum of [Fe(L)Cl$_2$], a prominent peak around 475 nm with a shoulder at about 752 nm was observed, corresponding to MLCT and d–d transitions, respectively. The electronic spectrum of **2** shows bands at 425, 560 and 967 nm, assignable to the transitions $^4T_{1g} \rightarrow {}^4T_{1g}(P)$, $^4T_{1g} \rightarrow {}^4A_{2g}(F)$ and $^4T_{1g} \rightarrow {}^4T_{2g}(F)$, respectively. The position of these bands indicates an octahedral geometry about the Co^{II} ion [29]. The spectrum of **3** exhibits bands at 475, 685 and 873 nm, which may be assigned to $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ and $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ transitions, respectively. These observations suggest an octahedral geometry of the Ni^{II} ion [30].

3.4. Electrochemistry

The redox properties of the complexes were examined by cyclic voltammetry using a Pt-disk working electrode and a Pt-wire auxiliary electrode in dry dimethylformamide using [n-Bu$_4$N]ClO$_4$ as the supporting electrolyte. Voltammetric data are set out in Table 3.

The voltammetric response for [Fe(L)Cl$_2$] (**1**) exhibits an oxidative couple at the $E_{1/2}$ value of +0.295 V, assigned to the Fe^{II}/Fe^{III} pair, [29] with a peak potential separation (ΔE_p) of 120 mV and cathodic to anodic current intensity ratio of 0.97. The cyclic voltammogram of [Co(L)Cl$_2$] (**2**) shows a quasi-reversible peak for the Co^{II}/Co^{III} redox couple at $E_{1/2}$ of 0.795 V with ΔE_p of 195 mV. The cyclic voltammogram of [Ni(L)Cl$_2$] (**3**) exhibits a quasi-reversible Ni^{II}/Ni^{III} redox couple centered at 0.745 V versus Ag/AgCl. Here, the $E_{1/2}$ value for complex **3** lies at the higher side of the range generally measured, 0.3–0.8 V, for the Ni^{III}/Ni^{III} oxidation potential data for thioether containing nickel(II) complexes, [31] indicating that Ni^{II} is fairly stabilised in **3**. No cathodic response, recommended for Ni^{II} → Ni^{I} reduction, was observed for **3** up to −1.51 V. The voltammetric parameters were studied in the scan rate interval 50–400 mV s^{-1}. The ratio between the cathodic peak current and the square root of the scan rate ($I_{pc}/v^{1/2}$) is approximately constant. The peak potential shows a small dependence on the scan rate. The ratio I_{pc} to I_{pa} is close to unity. From these data, it can be deduced that the redox couple is related to a quasi-reversible one-electron transfer process controlled by diffusion.

4. Conclusion

The synthesis and characterization of three new mononuclear complexes of iron(II), cobalt(II) and nickel(II) with a N$_2$S$_2$ donor set have been performed. No inert atmosphere (i.e. dinitrogen) is required for the synthesis of the iron(II) and cobalt(II) complexes, but these are prone to aerial oxidation. Structural characterization of **1** and **3** by X-ray diffraction indicates isomorphous crystals with the metal ion in an octahedral geometry with the chloride and py ligands occupying *cis* and *trans* positions, respectively. All the complexes manifest very similar redox behavior in dimethylformamide solution, showing M^{II}/M^{III} redox couples in the cyclic voltammograms, but no cathodic response for the M^{II}/M^{I} redox process.

Supplementary data

CCDC 697636 and 697637 contain the supplementary crystallographic data for compounds **1** and **3**, respectively. 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 CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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