Synthesis, Spectroscopic Studies and Crystal Structure of a New Co (III) Complex derived from ONO donor tridentate Schiff base ligand

ABSTRACT

A new Co (III) complex prepared by the reaction of 2-((2 hydroxyethylimino)methyl)phenol (H_2L) with Co(III) ion is reported in this paper. The H_2L ligand is structurally characterized by elemental analysis, NMR and infrared spectroscopies. The mononuclear complex $[Co(HL)_2]\cdot Cl\cdot H_2O$, is characterized by infrared spectroscopy, elemental analysis, conductance and magnetic room temperature measurements and single crystal X-ray diffraction. The compound crystallizes in the monoclinic system in the space group $P2_1/c$ with the unit cell parameters a = 16.4258 (6) Å, b = 10.1398 (4) Å, c = 11.7705(4) Å, β = 94.885(3)°, Z = 4, R_1 = 0.0374 and wR_2 = 0.075. The asymmetric unit of the compound contains a discrete $[Co(HL)_2]^+$ cation, one free chloride anion and one uncoordinated water molecule. The coordination polyhedron around the Co (III) center is best described as a distorted octahedral with CoN_2O_4 chromophore. The crystal structure of the complex is stabilized by intramolecular and intermolecular hydrogen bonds.

Keywords: Schiff base, salicylaldehyde, cobalt, X-ray diffraction, complex, mononuclear.

1. INTRODUCTION

“Schiff bases derived from salicylaldehyde constitute a family of organic compounds widely studied” [1]. They result from a condensation reaction between salicyladehyde and primary aliphatic or aromatic amines. The great interest given to these compounds is mainly due to their easy preparation process, generally carried out in a one step with good yields, their great thermal and photonic stability as well as their great coordination power, in solution or in the solid state, with respect to the different transition metal ions [2-6] and lanthanide [7]. “These Schiff bases, generally possessing donor atoms N, O or/and S, play an important role in coordination chemistry. They easily form stable complexes with most transition metal ions generating original and various structures” [8-13]. “Due to their numerous physicochemical properties, these complexes are used in different fields of chemistry, such as catalysis, liquid-liquid extraction and corrosion, in which they are used as inhibitors” [14-15]. The biological activities of these ligand and those of their complexes have been widely explored in recent decades [4–6, 10-13, 16]. Their antimicrobial [17-21], antifungal [22-24], antitumor [25-26], anti-Alzeimer’s [27], anticancer [28], antibacterial [16], and antioxidant [29] properties have been successfully tested. In this paper, we report the synthesis, the spectroscopic characterization, and the crystal structure of the new complex $[Co(HL)_2]\cdot Cl\cdot H_2O$ derived from the Schiff base 2-((2-hydroxyethylimino)methyl)phenol (H_2L).

2. MATERIALS AND METHODS

2.1 Starting Materials and Instrumentations

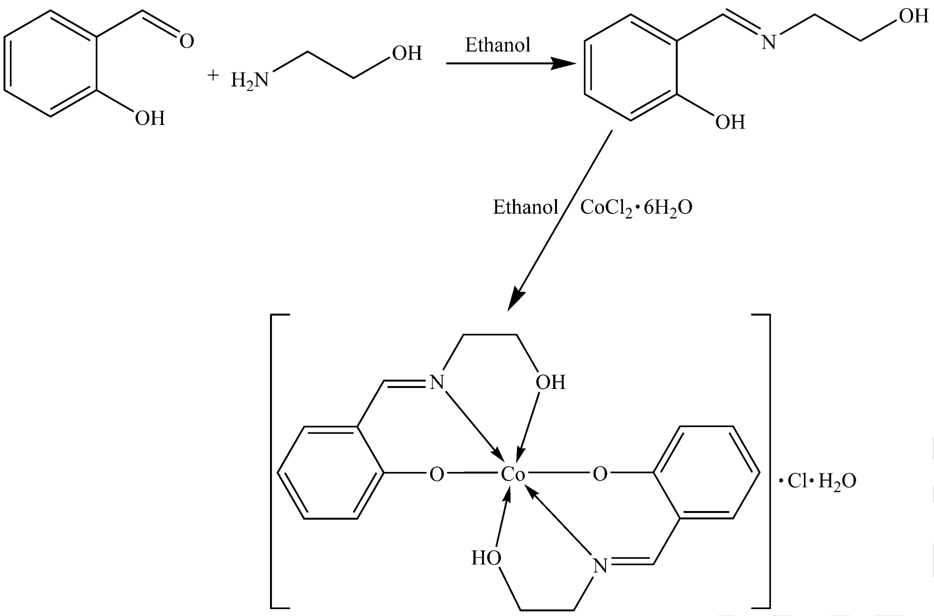
“Salicylaldehyde, 2-aminoethanol, glacial acetic acid and cobalt chloride hexahydrate were commercial products (from Aldrich) and were used without further purifications. The solvents were reagent grade and were purified by usual methods. Elemental analyses were carried out using a VxRio EL Instrument. The IR spectra were recorded on a FTIR Spectrum Two of Perkin Elmer (4000–400 cm^{-1}). The UV–Vis spectra were run on a Perkin-Elmer UV/Visible spectrophotometer Lambda 365 (1000–200 nm). The ^{1}H and ^{13}C NMR spectra of the Schiff bases were recorded in DMSO-d_6 on a BRUKER 500 MHz spectrometer at room temperature, using TMS as an internal reference. The molar conductance of 10^{-3} M solutions of the metal complex in DMF was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Magnetic measurements for the complex was performed at room temperature by using a Johnson Mattey scientific magnetic susceptibility balance (Calibrant: $Hg[Co(SCN)_4]$)” [40].

2.2 Synthesis of 2-((2-hydroxyethylimino)methyl)phenol (H_2L)

Method from literature [23] was used with slight modification. In a 250 mL flask containing 20 mL of ethanol and 1.25 g (10.235 mmol) of salicylaldehyde, 0.75 g (10.235 mmol) of 2-aminoethanol dissolved in 10 mL of ethanol was added. The resulting mixture was refluxed for two hours. The methanol was removed, and a viscous red oil was recovered. Yield 75.2 %. Anal. Calc for $C_9H_{11}NO_2$: C, 65.44; H, 6.71; N, 8.48. Found: C, 65.46; H, 6.68; N, 8.45. FTIR (ν, cm^{-1}): 3329, 2971, 2876, 1632, 1580, 1493, 1466,1273, 1196, 1120. UV-vis (Solution, DMF, λ (nm)): 285, 320. NMR ^{1}H [DMSO-d_6, 300MHz,(δ, ppm)]: 13.56 (s, Ar-OH, 1H) ; 8.51 (s, HC=N, 1H); 6.87–7.44 (m, H-Ar, 4 H); 4.77 (s, –OH, 1H); 3.54 (t, $–CH_2–N$, 2H); 3.74 (t , $–CH_2–OH$, 2H). NMR ^{13}C [DMSO-d_6, 300 MHz, (δ, ppm)] : 118.61 (C_{Ar}) ; 161.06 (C_{Ar}–OH) ; 118.05 (C_{Ar}) ; 132.12 (C_{Ar}) ; 118.24 (C_{Ar}) ; 131.99 (C_{Ar}) ; 166.60 (C=N) ; 60.24 ($–CH_2–$) ; 60.90 ($–CH_2–OH$).

2.3 Synthesis of the complex $[Co(HL)_2]\cdot Cl\cdot H_2O$

In a 100 mL flask, 10 mL of ethanol solution containing 0.1 g (1 mmol) of the ligand H_2L and a solution of the $CoCl_2\cdot 6H_2O$ 0.2379 g (1 mmol) in 10 mL of ethanol was added. The resulting solution was stirred at room temperature for one hour and then filtered. The filtrate was left to slow evaporation. Brown crystals suitable for X-ray analysis were formed after one week. Yield 59 %. Anal. Calc for $C_{18}H_{22}N_2O_5ClCo$: C, 49.05 ; H, 5.03; N, 13.37; Cl, 8.04. Found: C, 49.03; H, 5.01; N, 13.35; Cl, 8.01. IR (cm^{-1}): 3722, 3200, 1643, 1599, 1572, 1490, 1466, 1298, 1196, 1106, 835, 781. UV-vis (Solution, DMF, , λ (nm)): 302, 333, 429. Magnetic moment: diamagnetic. Conductance Λ ($\Omega^{-1}\cdot cm^2\cdot mol^{-1}$): 70.35 (fresh solution) and 71.81 (two weeks after).



Scheme 1. Synthetic procedure of the ligand H_2L and the complex $[Co(HL)_2] \cdot Cl \cdot H_2O$

2.4 Crystal structure determination of complexe $[Co(HL)_2] \cdot Cl \cdot H_2O$

"The details of the crystal structure solution and refinement are given in Table 1. Measurements were made on a Rigaku Oxford Diffraction Dual source diffractometer at the MoKα radiation (0.71073 Å). All data were corrected for Lorentz and polarization effects. Empirical absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL" [30]. "The structures were solved by intrinsic phasing, which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms" [31]. "All hydrogen atoms were located in their calculated positions and refined using a riding model. Molecular graphics were generated using ORTEP-3" [32].

Table 1. Crystallographic data and refinement parameters for the complex

Empirical formula	$C_{18}H_{22}ClCoN_2O_5$
Formula weight	440.75
Temperature (K)	293(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	16.4258(6)
b (Å)	10.1398(4)
c (Å)	11.7705(4)
α (°)	90
β (°)	94.885(3)
γ (°)	90
Volume (Å^3)	1953.31(12)
Z	4
ρ_{calc} (g/cm^3)	1.499
μ (mm^{-1})	1.047
F(000)	912.0
Crystal size (mm^3)	0.19 × 0.12 × 0.03
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.726 to 57.368
Index ranges	$-20 \le h \le 21$, $-13 \le k \le 13$, $-15 \le l \le 15$

No. of measured reflections	29197
independent reflections	4745
Observed [$I > 2\sigma(I)$] reflections	4020
R_{int}	0.0374
$R[F^2 > 2\sigma(F^2)]$,	0.028,
$wR(F^2)$	0.075
No. of reflections	4745
No. of parameters	257
No. of restraints	0
GOF	1.036
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.41

3. RESULTS AND DISCUSSION

3.1 General Study

The acyclic Schiff base ligand H_2L was prepared following a method reported in the literature [23]. The synthesis of H_2L was achieved in one step procedure by the condensation reaction between salicylaldehyde and 2-aminoethanol in quantitative yield (Scheme 1). The analytical data agree with the formulation. The infrared spectrum of the ligand shows a broad band between 3200 and 3500 cm^{-1} which is attributable to the ν_{OH} vibrations of the phenol and the alcohol groups. The bands observed between 2876 and 2971 cm^{-1} are attributed to the methylene groups. Bands characteristic of phenyl ring are pointed in the region 1490-1580 cm^{-1}. The band observed at 1632 cm^{-1} corresponds to the $\nu_{C=N}$ stretching vibration of the imine group. The band observed at 1276 cm^{-1} is attributed to the $\nu_{C_{Ar}–O}$ vibration. The ^{1}H NMR spectrum of the ligand recorded in DMSO-d$_6$ reveals singlet signal at 13.56 ppm which is attributed to the phenolic proton Ar–O**H**. The signals due to the aromatic protons are observed in the range [6.87–7.44] ppm. Two signals appearing as triplet are observed at 3.54 and 3.74 ppm and assigned, respectively, to the methylene protons ($–N–C\mathbf{H}_2–$) and ($–C\mathbf{H}_2–OH$). The Signals at 8.51 and 4.77 ppm are, respectively, assigned to the proton of the imine function (**H**C=N) and the protons of the primary alcohol $–CH_2–O\mathbf{H}$. The ^{13}C NMR shows a signal at 161.06 ppm assigned to the $\mathbf{C}_{ipso}$–OH of the phenol ring. The signals in the range [118–132] ppm are attributed to the aromatic carbon atoms. The signal at 166.60 ppm is attributed to the azomethine carbon atom (**C**=N). The signals of the methylene carbon atoms are observed at 60.24 ($–N–\mathbf{C}H_2–$) and 60.90 ppm ($–\mathbf{C}H_2–OH$). The reaction of H_2L with cobalt chloride metal in 1:1 ratio, was screened. The complexation was achieved in ethanol solution by mixing both ligand and metal salt. An air-stable compound was isolated and formulated as mononuclear $[Co(HL)_2]\cdot Cl\cdot H_2O$. Crystals suitable for X-ray analyses were isolated by slow evaporation of the solution of the compound. The complex is characterized by elemental analysis, IR and UV spectroscopies, molar conductivity measurements, room temperature magnetic moment measurement and X-ray diffraction. The analytical data agree with the formulation [$C_{18}H_{22}ClCoN_2O_5$]. Upon the complexation of H_2L with Co(III) ion, the infrared spectrum of the complex reveals a shift of the $\nu_{C=N}$ band in comparison to the corresponding band of the free ligand which appears at 1632 cm^{-1}. On the FTIR spectrum of the complex, the $\nu_{C=N}$ band shifts to high frequencies and appears at 1643 cm^{-1}. This displacement is indicative of the involvement of the azomethine nitrogen atoms in the coordination [33]. “The shifts of the alcoholic ν_{C-O} and the phenolic ν_{C-O} vibration bands of the ligand, upon complexation, indicates the coordination of the two different oxygen atoms to the metal ion” [33]. “These bands which appear, respectively, at 1120 cm^{-1} and 1273 cm^{-1} on the spectrum of the free ligand are observed at 1100 cm^{-1} and 1298 cm^{-1} on the spectrum of the complex. The electronic spectrum of the free ligand shows two main bands at 285 nm and 320 nm attributable to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ of the aromatic ring, the azomethine and the phenol groups. The electronic spectrum of the complex of Co(III) exhibits distinct absorption bands at 302 nm, 332 nm and 429 nm. The band appearing at 302 nm is attributed to $\pi\rightarrow\pi^*$ transition, while the band at 332 nm is due to $n\rightarrow\pi^*$ transition” [34]. These transitions are due to the benzene ring, the azomethine and the phenolate moieties. Comparatively to the bands on the spectrum of the free ligand, reduction in intensity is observed. The band at 429 nm is assigned to the ligand to metal charge transfer (LMTC) $PhO\rightarrow Co^{3+}$ and $C{=}N\rightarrow Co^{3+}$ [32]. “Room temperature magnetic susceptibility measurements show that the complex $[Co(HL)_2]\cdot Cl\cdot H_2O$ is diamagnetic as expected for low-spin cobalt(III) complex” [35, 36]. “The molar conductivity measurements of the complex recorded from a fresh solution of DMF and after fifteen days of storage [70.35 $\Omega^{-1}.cm^2.mol^{-1}$ and 71.81 $\Omega^{-1}.cm^2.mol^{-1}$] indicate that the complex is 1:1 electrolyte type, according to *Geary”* [37]. The small variation in the values obtained shows that the complex is stable in the DMF solution.

3.2 Description of the Crystal Structure of the Complex $[Co(HL)_2]\cdot Cl\cdot H_2O$

Suitable Single crystals for X-ray diffraction of the mononuclear cobalt (III) complex were obtained by slow evaporation of its ethanol solution. Crystallographic data, collection and refinement parameters are listed in Table 1. Selected bond lengths and angles are summarized in Table 2. Hydrogen bond data are reported in Table 3. An ORTEP view of the structure is shown in Figure 1 and the packing diagram is presented in Figure 2. The asymmetric unit contains two monodeprotonated ligand molecules, one Co (III) ion, one uncoordinated water molecule and one free chloride anion. Each ligand molecule acts in tridentate fashion through one phenolate oxygen atom, one alcoholic oxygen atom and one azomethine nitrogen atom, yielding a complex in which Co(III) is octacoordinated. The Co(III) ion is situated in a N_2O_4 inner. The coordination environment around the Co (III) ion can be described as a distorted octahedron. The O1, O2, N1 and N2 atoms occupy the equatorial plane, while O3 and O4 occupy the axial positions. The *cissoid* angles values in the basal plane deviated from the ideal value of 90° [O1–Co1–N1 = 95.22(6)°; N1–Co1–O2 = 83.96(5)° ; N2–Co1–O2= 90.59(5)° ; O1–Co1–N2= 90.05(5)°] with a sum of 359.82 °. The *transoid* angles values [O3–Co1–O4 = 178.01(5)° and N1–Co1–N2 = 173.67(6)°] deviated severely from the ideal value of 180°. The angle subtended by the atoms in apical positions [O3–Co1–O4 = 178.01(5)°] is slightly different of the ideal value of 180 °. The bond lengths in the equatorial plane are Co1–O1 = 1.8500(11) Å, Co1–O2 = 1.9614(11) Å, Co1–N1 = 1.8901(13) Å, Co1–N2 = 1.8941(13) Å. The axial positions bonds lengths are Co1–O3 = 1.8561(11) Å, CO1–O4 = 1.9565(11) Å. These values are comparable to those reported for similar octahedral cobalt complex [38, 39]. The crystal packing of the compound is stabilized by intramolecular hydrogen bonds $O_{alcoholic}$—H···O_{water} (O2—H2···O5) and $O_{alcoholic}$—H···Cl [O4—H4···Cl1], classical intermolecular hydrogen bonds: O_{water}—H···Cl [O_5—H5A···Cl1^{iv}, iv = x, y−1, z; O5—H5B···Cl1^{iii}, ii = −x, −y+1, −z+1] and unclassical intermolecular hydrogen bonds C—H···$O_{phenolate}$ [C16—H16···O3^{i}, i = x −y+1/2, −y+1/2], C—H···$O_{alcoholic}$ [C9—H9B···O4^{iii}, iii = −x, −y+1, −z+1] and C—H···Cl [C8—H8B···Cl1^{ii}, ii = x, −y+3/2, z+1/2]. These hydrogen bonds (Table 3)connect the units and stabilize the structure. A view of the packing diagram of the complex [Co(HL)$_2$]·Cl·H$_2$O in the *ab* plane is shown in Figure 2.

Table 2. Selected geometric parameters (Å, °) for the complex.

Co1–O2	1.9614(11)	Co1–N1	1.8901(13
Co1–O3	1.8561(11)	Co1–N2	1.8941(13)
Co1–O4	1.9565(11)	Co1–O1	1.8500(11)
O3–Co1–N1	88.26(5)	O3–Co1–O4	178.01(5)
O4–Co1–O2	86.72(5)	O3–Co1–O2	91.69(5)
O1–Co1–O2	176.77(5)	O3–Co1–N2	95.13(5)
O1–Co1–O3	91.40(5)	O1–Co1–O4	90.20(5)
O1–Co1–N2	90.05(5)	O1–Co1–N1	95.22(6)
N2–Co1–O2	90.59(5)	N2–Co1–O4	83.68(5)
N1–Co1–O2	83.96(5)	N1–Co1–O4	92.77(5)
N1–Co1–N2	173.67(6)		

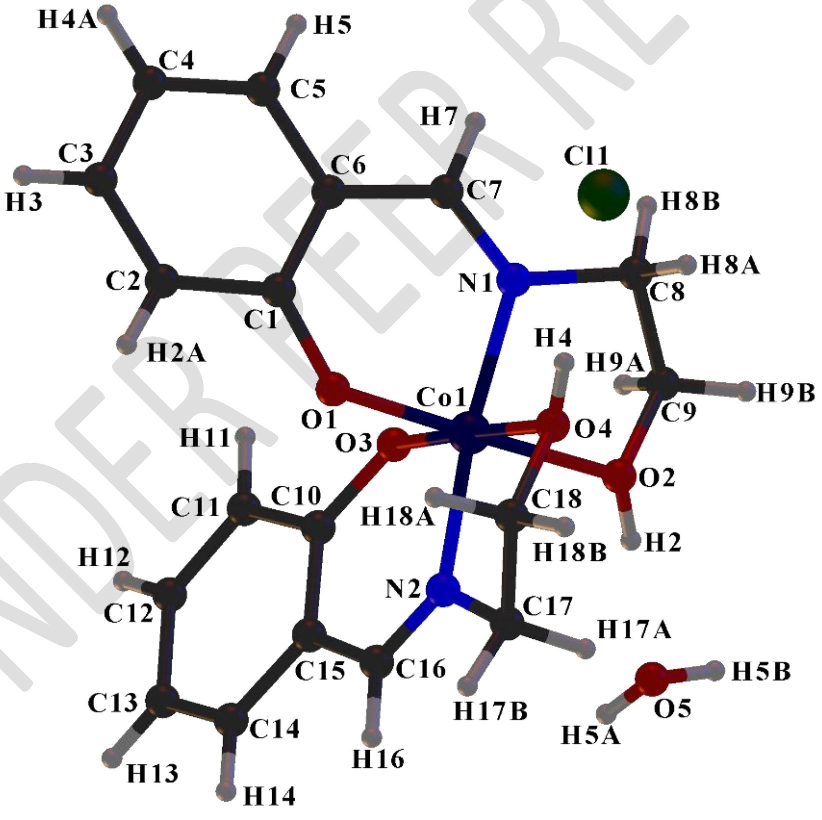


Fig. 1. Crystal structure of the complex [Co(HL)$_2$]·Cl·H$_2$O

Table 3. Hydrogen-bond geometry (Å, °) for the complex

D—H···*A*	*D*—H)	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O5	0.82	1.77	2.584(2)	169.0
C16—H16···O3^{i}	0.93	2.31	3.2130(19)	162.7
C8—H8B···Cl1^{ii}	0.97	2.76	3.7194(18)	171.4

C9—H9B···O4^{iii}	0.97	2.48	3.436(2)	169.9
O4—H4···Cl1	0.81(3)	2.17(3)	2.9805(13)	170(2)
O5—H5A···Cl1^{iv}	0.85(3)	2.33(3)	3.160(2)	165(2)
O5—H5B···Cl1^{iii}	0.80(4)	2.38(4)	3.163(2)	167(3)

Symmetry codes: (i) x, −y+1/2, z−1/2; (iii) −x, −y+1, −z+1; (iv) x, y−1, z.

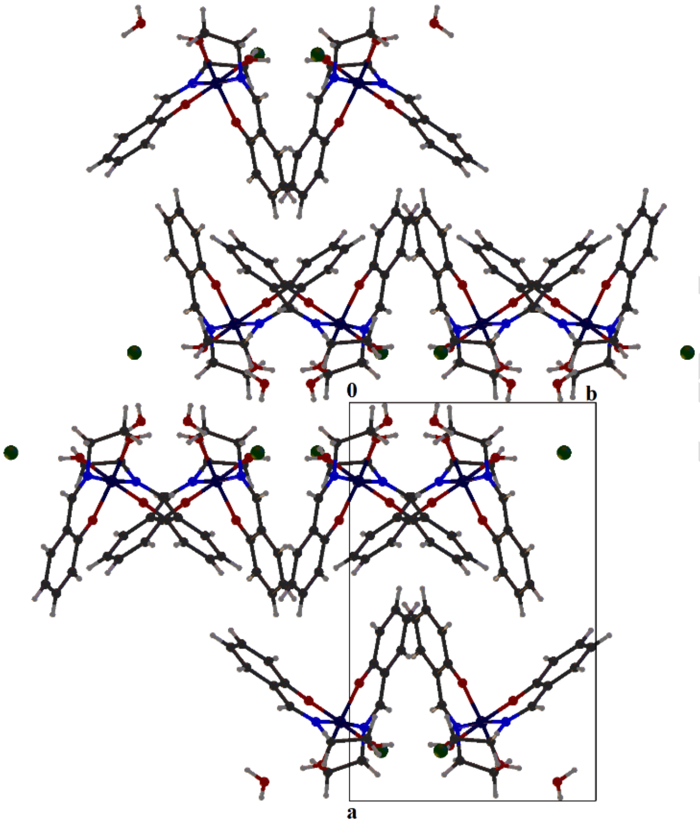


Fig. 2. View of the packing diagram of the complex [Co(HL)$_2$]·Cl·H_2O in the *ab* plane.

4. CONCLUSION

The complex [Co(H*L*)$_2$]·Cl·H_2O synthetized by the reaction of the Schiff base 2-((2-hydroxyethylimino)methyl)phenol (H_2*L*) and hexahydrate cobalt chloride have been characterized by IR and UV spectroscopies, conductivity and room temperature magnetic moment measurements. The structure of the complex was solved by X-ray diffraction. Considering the conductance values, the complex is stable in DMF solution and is 1:1 electrolyte in nature. The complex is diamagnetic in nature indicating a high-spin cobalt (III) complex. The X-ray diffraction study shows that the Co (III) complex is mononuclear, and the metal atom is situated in an octahedral environment, surrounded by two ligand molecules acting in tridentate fashion. The structure of the complex is consolidated by extensive intermolecular hydrogen bonds which produce a three-dimensional network.

5. SUPPLEMENTARY DATA

CCDC– 2409457contains the supplementary crystallographic data for the complex. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

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