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Synthesis, spectroscopic studies and X-ray structure determination of a trinuclear cobalt complex derived from the Schiff base ligand *N*,*N*-dimethyl-*N*'-((5-methyl-1*H*-imidazol-4-yl)methylene)ethane-1,2-diamine

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Abstract: *Polynuclear mixed oxidation Co^{III}—Co^{II}—Co^{III} complex formulated as{[(HL)(SCN)Co](m-L)[Co(SCN)$_4$](m-L)[Co(SCN)(HL)]}$_2$·2(C_2H_5OH)·2(H_2O) (**1**) has been prepared from a Schiff base (HL = N, N-dimethyl-N'- ((5-methyl-1H-imidazol-4-yl)methylene)ethane-1,2-diamine) containing imidazole ring. The complex (**1**) is characterized by elemental analysis, UV–Vis and IR spectroscopic studies. The complex (**1**) crystallizes in the triclinic space group P-1 with unit cell dimensions a = 11.3017 (7) Å, b = 15.1468 (11) Å, c = 18.9796 (18) Å,α = 71.849 (7)°, β = 82.048 (7)°, γ = 86.477 (6)°, V = 3057.1 (4) Å^3, Z = 1, R_1 = 0.092 and wR_2 = 0.308. Both Co^{II} andCo^{III} cationsare in a N_6 inner and are situated in a distorted octahedral environment. The Co^{II} cation is linked by two imidazole nitrogen atoms and fournitrogen atoms from thiocyanate anions. The Co^{III} cation is octacoordinated is linked by two imidazole nitrogen atoms, two imino nitrogen atoms, one secondary amino nitrogen atom and one nitrogen atom from thiocyanate anion. Variable temperature (300–2 K) magnetic susceptibility measurements have been carried out for the complex.*

Keywords: *Schiff base, crystal, complex; cobalt, octahedral, magnetism.*

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I. Introduction

Coordination chemistry based on the use of Schiff bases has been experiencing a revival in recent years owing to the contribution of organic chemistry. Increasingly functionalized organic molecules used as ligand are prepared from concepts of modern organic chemistry [1-4]. These new Schiff bases combined with transition metal yield coordination compounds with original structures and with diverse physical properties [5-8]. Cobalt is a versatile transition metal with several degrees of oxidation. Complexes with Co(II)[9,10], Co(III)[11,12], or both Co (II) and Co (III)[13,14]are widely reported. These cobalt complexes are present in fields such as electrochemical materials [15,16], catalysis[17,18], biology [19,20], and molecular magnetism [21,22], and in natural compound such as vitamin B12. Schiff bases derived from a precursor having a functionalized or non-functionalized imidazole moiety and their cobalt complexes are widely used. Several mononuclear [23-25], dinuclear [26-28], or trinuclear [26, 29-31] complexes are reported in the literature. These compounds are studied for their physical properties as well as their antimicrobial activities and the possibility that they offer in the study of metallo-enzymes. Indeed, there are often metal ions linked to the nitrogen atom of imidazole in biomolecules [32,33]. It's in this context that we prepared a Schiff base containing a substituted imidazole ring to synthesize a new bis-trinuclear complex {[(HL)(SCN)Co](μ-L)[Co(SCN)$_4$](μ-L)[Co(SCN)(HL)]}$_2$·2(C_2H_5OH)·2(H_2O) (**1**) (HL = N, *N*-dimethyl-*N*'- ((5-methyl-1*H*-imidazol-4-yl)methylene)ethane-1,2-diamine ($C_9H_{16}N_4$)). In this work, we have described the synthesis, the spectroscopic properties and the crystal structure of the complex (**1**) of neutral cobalt obtained by the complexation of the previous Schiff base with $Co(NO_3)_2·6H_2O$ with the presence of KSCN.

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Table-1. Crystallographic data and refinement parameter for the complex (**1**).

Chemical formula	$C_{42}H_{62}Co_3N_{22}S_6 \cdot C_{42}H_{62}Co_3N_{22}S_6 \cdot 2(C_2H_6O) \cdot 2(H_2O)$
M_r	2616.73
Crystal shape/color	Cut block/clear yellow
Crystal system, space group	Triclinic, *P*-1
Crystal size (mm)	0.09 × 0.08 × 0.08
a (Å)	11.3017 (7)
b (Å)	15.1468 (11)
c (Å)	18.9796 (18)
□ (°)	71.849 (7)
β (°)	82.048 (7)
γ (°)	86.477 (6)
V (Å^3)	3057.1 (4)
Z	1
D_{calc} (g.cm^{-3})	1.382
□ (MoK□)(Å)	0.71073
T (K)	293
μ (mm^{-1})	1.06
Index ranges	$-15 \leq h \leq 15$, $-19 \leq k \leq 20$, $-26 \leq l \leq 25$
F(000)	1312
□ range (°)	3.40-29.71
No. of measured reflections	58378
No. of independent reflections	15441
No. of observed [$I > 2\sigma(I)$] reflections	10360
R_{int}	0.088
$R[F^2 > 2\sigma(F^2)]$	0.092
$wR(F^2)$	0.308
Goodness-of-fitt (Gof) on F^2	1.05
No. of parameters	698
No. of restraints	25
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.77, −1.82

Table-2. Selected bond distances [Å] and angles [deg] for the complex (**1**).

Co1—N3	1.888 (5)	Co3—N5A	2.119 (5)
Co1—N5	1.896 (4)	Co3—N5A^{i}	2.119 (5)
Co1—N2A	1.902 (4)	Co3—N6A	2.130 (5)
Co1—N2	1.933 (4)	Co3—N6A^{i}	2.130 (5)
Co1—N3A	1.980 (5)	Co3—N1A	2.189 (4)
Co1—N4	2.060 (5)	Co3—N1A^{i}	2.189 (4)
Co2—N3B	1.897 (5)	Co4—N5C	2.056 (7)
Co2—N2C	1.913 (4)	Co4—N5C^{ii}	2.056 (7)

Co2—N5B	1.918 (5)	Co4—N1C^{ii}	2.151 (5)
Co2—N2B	1.932 (4)	Co4—N1C	2.151 (5)
Co2—N3C	1.975 (4)	Co4—N6C	2.265 (10)
Co2—N4B	2.048 (4)	Co4—N6C^{ii}	2.265 (10)
N3—Co1—N2A	93.46 (19)	N2—Co1—N4	167.3 (2)
N5—Co1—N2A	175.9 (2)	N3A—Co1—N4	97.8 (2)
N3—Co1—N3A	174.65 (19)	N5A—Co3—N5A^{i}	180.0
N2A—Co1—N3A	81.41 (19)	N5A—Co3—N6A	89.2 (2)
N3—Co1—N4	84.1 (2)	N1A—Co3—N1A^{i}	180.0

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

II. Materials and physical methods

2.1. Startingmaterials and Instrumentations

All chemicals and solvents were of analytical reagent grade and were used directly without further purification. The ligand *N*,*N*-dimethyl-*N*'-((5-methyl-1*H*-imidazol-4-yl)methylene)ethane-1,2-diamine (HL) was synthesized according to the literature[34]. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on a FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm^{-1} region. The UV-Visible spectra were recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The molar conductance of 1×10^{-3} M in DMSO solutions of the metal complexes was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibilities of the powdered samples were measured using a Johnson Mattey scientific magnetic susceptibility balance (Calibrant: $Hg[Co(SCN)_4]$).

2.2. Synthesis of the complex

{[(HL)(SCN)Co](m-L)[Co(SCN)$_4$](m-L)[Co(SCN)(HL)]}$_2$·2(C$_2$H$_5$OH)·2(H$_2$O) (**1**)

To a solution of 5-methyl-1*H*-imidazole-4-carbaldehyde (0.1100 g, 1 mmol) in 30 mL of ethanol was added (0.0881 g, 1 mmol) of *N*,*N*-dimethylethane-1,2-diamine. The resulting yellowish mixture was stirred for 30 min. A solution of $Co(NO_3)_2\cdot6H_2O$ (0.294 g, 1 mmol) in 5 mL of ethanol was added at room temperature. The initial yellow solution which turned immediately into a clear yellow color upon addition of metal salt was stirred for 2 hours. The mixture was filtered off and the solution was evaporated near dryness. The solid was isolated by filtration and recrystallized from a minimum of ethanol. On standing for one day crystals suitable for X-ray analysis were formed. Yied: 63 %. M.p.: 212°C. Anal. Calc for $C_{88}H_{140}N_{44}S_{12}O_4Co_6$: C, 40.39; H, 5.39; N, 23.55; S, 14.70. Found: C, 40.37; H, 5.35; N, 23.52; S, 14.67. IR (cm^{-1}): 3429 ($\nu_{O—H}$), 3420($\nu_{N—H}$), 2052 (ν_{SCN}), 1588 ($\nu_{C=N}$), 770 ($\delta_{C=S}$). UV-visible (λ_{max} (nm), DMF): 987, 497. Conductance Λ (S.cm^2.mol^{-1}): 10 (fresh solution) and 11 (two weeks after).μ_{eff} (MB) :11.758.

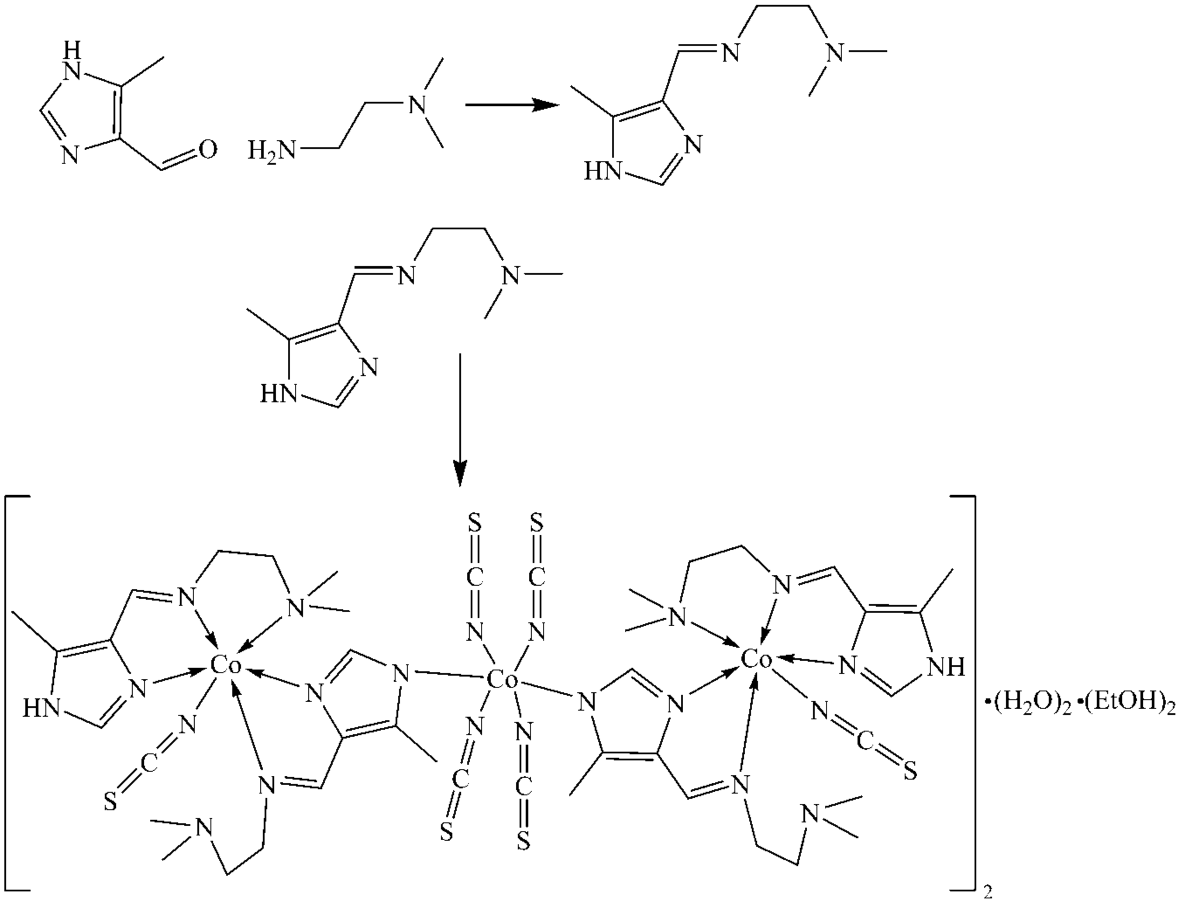
2.3. Crystal structure determination

Crystals suitable for single-crystal X-ray diffraction, of the reported compound (**1**), were grown by slow evaporation of EtOH solution of the complexes. Details of the crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized MoKα radiation (λ = 0.71073 Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL[35]. The structures were solved by direct methods 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[36]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH_3 groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP[37].

III. Results and Discussion

3.1. General studies

The ligand HL was prepared in situ by a facile condensation of 5-methyl-1*H*-imidazole-4-carbaldehyde and *N*,*N*-dimethylethane-1,2-diamine in methanol as reported in our previous work[34](Scheme 1). The polynuclear mixed Co(II)/Co(III) complex was obtained by adding to the above solution an equimolecular mixture of $Co(NO_3)_2{\cdot}6H_2O$ and KSCN in 1:2 ratio in ethanol. The versatile thiocyanate possesses a polarizable π system and two donor atoms with different character (N is a hard donor atom and S is a soft donor atom). It can act in unidentate fashion through the terminal N-atom or the terminal S-atom, in bidentate fashion as bridge through the N-atom donor and the S-atom donor or remains free as counter anion. In the synthesis reaction of the complex, the substitution of the nitrate anions of cobalt(II) salt is facilitate by the use of ethanol in which KSCN and cobalt nitrate are very soluble while the resulting KNO_3 is insoluble. The elemental analyses result are in accordance with the chemical formulae obtained from X-ray diffraction study : $\{[(HL)(SCN)Co](\mu\text{-}L)[Co(SCN)_4](m\text{-}L)[Co(SCN)(HL)]\}_2{\cdot}2(C_2H_5OH){\cdot}2(H_2O)$.



Scheme 1. Synthetic scheme for the ligand and it'sCo^{III}—Co^{II}—Co^{III}complex preparation.

The infrared spectrum of the complex exhibitstwo broad bands in the range 3440-3430 cm^{-1} which are attributed to OH of lattice water/ethanol molecules and NH stretching[38,39]. The vibration of the imine function appears at1558 cm^{-1} while the spectrum of the free Schiff base reveals a band at 1644 cm^{-1}. This shift to low frequencies is indicative of the involvement of the nitrogen atom of the azomethine in the coordination sphere of the cobalt atom. The thiocyanate moiety shows an intense and sharp band at 2052 cm^{-1} assignable to the coordinated thiocyanate group[40]. Bands due to the aromatic ring are pointed in the region 1545-1446 cm^{-1}. Three bands at 10129 cm^{-1}, 20 120 cm^{-1} and 36101 cm^{-1} appear on the UV spectrum of the polynuclear complex. The two higher energy bands are allocated to d - d transitions while the third lower energy band is allocated to an MLCT transfer. In the case of an octahedral environment with a Co(II) ion, three d - d transitions are allowed and expected: $^4T_{1g} \rightarrow {}^4T_{2g}$ (ν1), $^4T_{1g} \rightarrow {}^4A_{2g}$ (ν2) and $^4T_{1g} \rightarrow {}^4T_{1g}$ (P) (ν3). In the case of this complex, the wide band pointed at 20120 cm^{-1} corresponding to the high energy transition is attributed to the transition $^4T_{1g} \rightarrow {}^4T_{1g}$. The second expected band involving a bi-electronic process which induces an extremely low intensity is attributed to the transition $^4T_{1g} \rightarrow {}^4A_{2g}$. The lower energy band is 10129 cm^{-1} is located in the near infrared and is assigned to the $^4T_{1g} \rightarrow {}^4T_{2g}$ transition[41,42].The band pointed at 31250 cm^{-1} is due to $\pi \rightarrow \pi^*$ electronic transition in the azomethine chromophore. The thiocyanate moiety also exhibits a band at 28169cm^{-1}which is due to $n \rightarrow \pi^*$ electronic transition in the thiocyanate chromophore[34].The bands due to the transitions in Co(III) ions in octahedral geometry[43,44], expected in the range 21740—23585 cm^{-1}, are obscured by the broad band at 20120 cm^{-1} which is due to the Co(II) ion in octahedral environment.The structure of the complex present simultaneously Co(III) and Co(II) ions. The value of the magnetic moment at room temperature of the complexof11.758 B.M.which is consistent with the presence of two high-spin Co(II) ions in octahedral environment. The molar conductivity which is 10 S.cm^2.mol^{-1} is indicative of a neutral electrolyte in DMF solution[45].

3.2. Structure description of the complex

The compound **1** crystallizes in triclinic space group P-1and displays three-D architecture. The asymmetric unit contains two Co (II), four Co (III) ions, four neutral HL ligand molecules, four mononegative L$^-$, twelve thiocyanate anions, two uncoordinated ethanol molecules and two lattice water molecules. There are two crystallography independent Co ions. The central Co3 and Co4 are coordinated by four thiocyanate anions through the nitrogen atoms and two imidazole nitrogen atoms from two different monodeprotonated ligand molecule which act as bridge through their second imidazole nitrogen atom. The environment of the hexacoordinated central Co(II) is best described as a slightly distorted octahedral geometry. All values of the *transoid*N—Co—N angles are 180.0° for Co3 and Co4. The values of the *cissoid* N—Co—N angles are in the ranges [86.67(19)—93.33(19)°] for Co3 and [83.2(3)—96.8(3)°] for Co4 and are close proximity to the ideal value of 90° expected for a perfect octahedral geometry. These angles values show that the environment of Co4 is more distorted than that of Co3. For the inversion center Co3, the basal plane is occupied by the four thiocyanate nitrogen atom with the shorter distances [Co3—N5A = 2.119(5) Å and Co3—N6A = 2.130(5) Å], the apical positions being occupied by two imidazole nitrogen atoms with the longer bond length [Co3—N1A = 2.189(4) Å]. For the inversion center Co4, the basal plane is occupied by two imidazole nitrogen atoms and two thiocyanate nitrogen atom with the shorter bond lengths[Co4—N1C = 2.151(5) Å and Co4—N5C = 2.056(7) Å], the apical positions being occupied by two thiocyanate nitrogen atoms with the longer distance [Co4—N6C = 2.265(10) Å]. Each of the terminal Co(III) issix-coordinated by one thiocyanate nitrogen atom, by one neutral ligand molecule through one imidazole nitrogen atom, one secondary amino nitrogen atom and one tertiary amino atom. The second monodeprotonated ligand molecule acts in bidentate fashion through one imidazole nitrogen atom and one secondary amino nitrogen atom, the tertiary nitrogen atom remains free. The basal planes are occupied by the amino nitrogen atoms while the apical positions are occupied by the thiocyanate nitrogen atom and the imidazole nitrogen atoms. The *transoid* angle values are in the range [175.9(2)—166.14(19)°] and the *cissoid* angle run in the interval [81.41(19)—97.8(2)°]. For all the terminal Co (II) the environment is described as distorted octahedral geometries. The Co—N distances are in the range [1.888(5)—2.265(10) Å] and are consistent with the normal value for Co—N bond length[46,47].The three Co atoms, in each molecule complex in the asymmetric unit, are co-linear with equal distances between two adjacent atoms [6.123 Å (Co1…Co3) and 6.096 Å (Co2…Co4)].In the crystal structure, the trinuclear units are arranged into layers parallel to [011] direction.

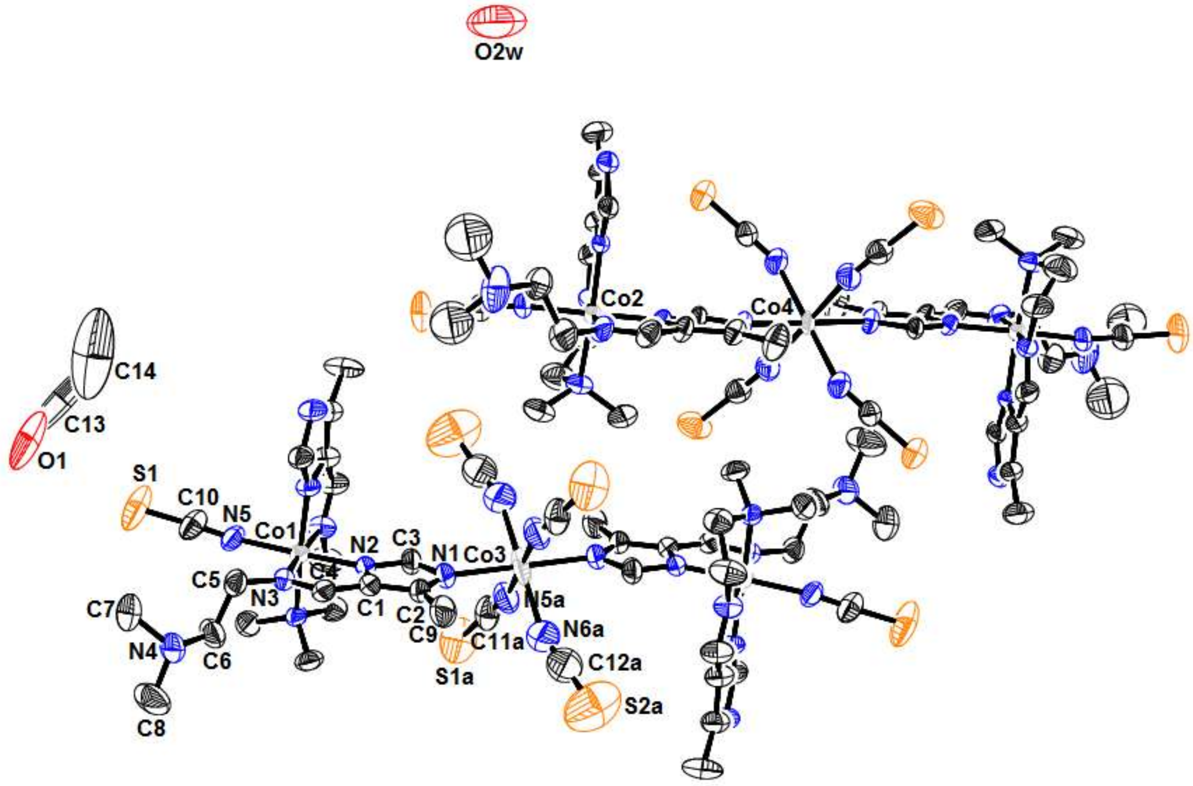


Figure 1:*ORTEP* plot (30% probability ellipsoids) showing the structure of (**1**).

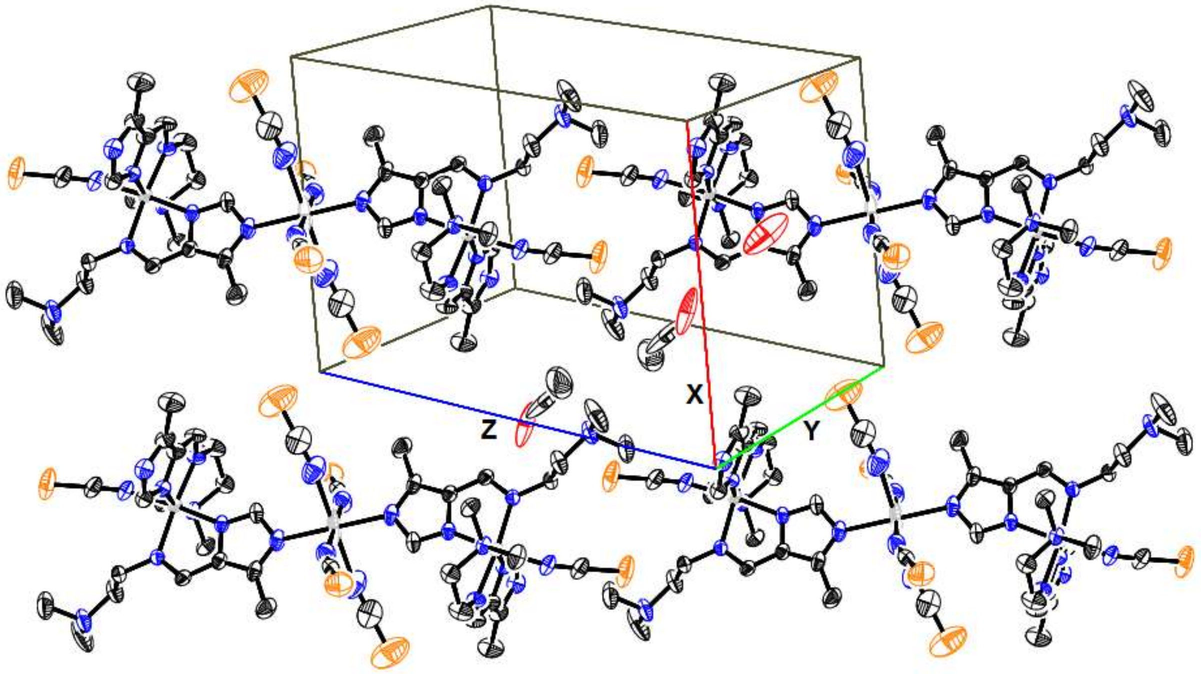


Figure 2: Layers of the trinuclear units parallel to the [011] direction.

3.3. Magnetic property

The temperature dependence of the magnetic susceptibility of the crystalline polynuclear compound wasmeasured in the temperature range 300-2 K with an applied field of 1000 Oe (Figure 3).The shape of the curve $\chi_M = f(T)$ (Figure 3), shows that the molar susceptibility increases slightly in the temperature range 300-100K and grows faster below 100 K by tending asymptotically towards the ordinate axis at low temperature.To elucidate the magnetic properties of the complex we have drawn the curve of the product of molar susceptibility by temperature as a function of the latter: $\chi_M T = f(T)$(Figure 4). The value of $\chi_M T$ at room temperature of3.29 $cm^3K\ mol^{-1}$per cobalt (II) ion is higher than the value of 1.875cm^3 K mol^{-1}expected for Co(II)ion with S = 3/2 and g = 2.00[48]. This large difference is due to the significant orbital contribution often observed for cobalt (II) ion with strong spin of the fundamental state $^4T_{1g}$. On decreasing the temperature, the $\chi_M T$value remains almost constant up to 100 K. Below 100 K, the $\chi_M T$ value decreases to reach a value of 2.05 cm^3 K mol^{-1} at 2 K. This fact may be due to an antiferromagnetic interaction, to a spin coupling and/or zero field splitting of the $^4T_{1g}$ ground state. The crystallographic structure indicates a central cobalt (II) surrounded by two terminal cobalt (III). The two Co^{3+}diamagnetic ions isolate $Co^{2+}$and prevent any intramolecular antiferromagnetic coupling. Indeed, we note that the Co-N distances are longer for the central atoms Co3/Co4 than for the terminal cobalt atoms Co1/Co2. These data confirm the low spin and high spin configurations for the Co^{3+} and Co^{2+} ions, respectively. The distance value of 10.351 Å between two cobalt (II) ions belonging to two trinuclear unitsindicates the absence of intermolecular antiferromagnetic interaction.The different trinuclear units being independent of each other. The value of $\chi_M T$ equal to 3.29 cm^3 K/mol at the temperature of 300 K would correspond to a cobalt (II) ion with spin S = 3/2 and a value of g = 2.65. Thus, the decrease in $\chi_M T$ below 100 K could be attributed to a zero-field splitting of the fundamental level $^4T_{1g}$ in $^4A_{2g}$ and ^4E_g. These gives, after spin orbit coupling, the three levelsΓ_6(J=1/2), Γ_7 (J=1/2) and Γ_7(J=3/2). The decrease of $\chi_M T$ at low temperature, can be explained by the variation of the population of these levels. At 2 K the$\chi_M T$value is 2.05 $cm^3K\ mol^{-1}$avoiding the existence of antiferromagnetic coupling.The variation of the magnetization versus the applied field at 2 K, shows the increase of the magnetization without saturation up to 70 000 Oe.The shape of the curve $M = f(H)$(Figure 5)can be explained by the low temperature populating of the low excited levels resulting from the combined effects of zero field splitting and spin orbit coupling.

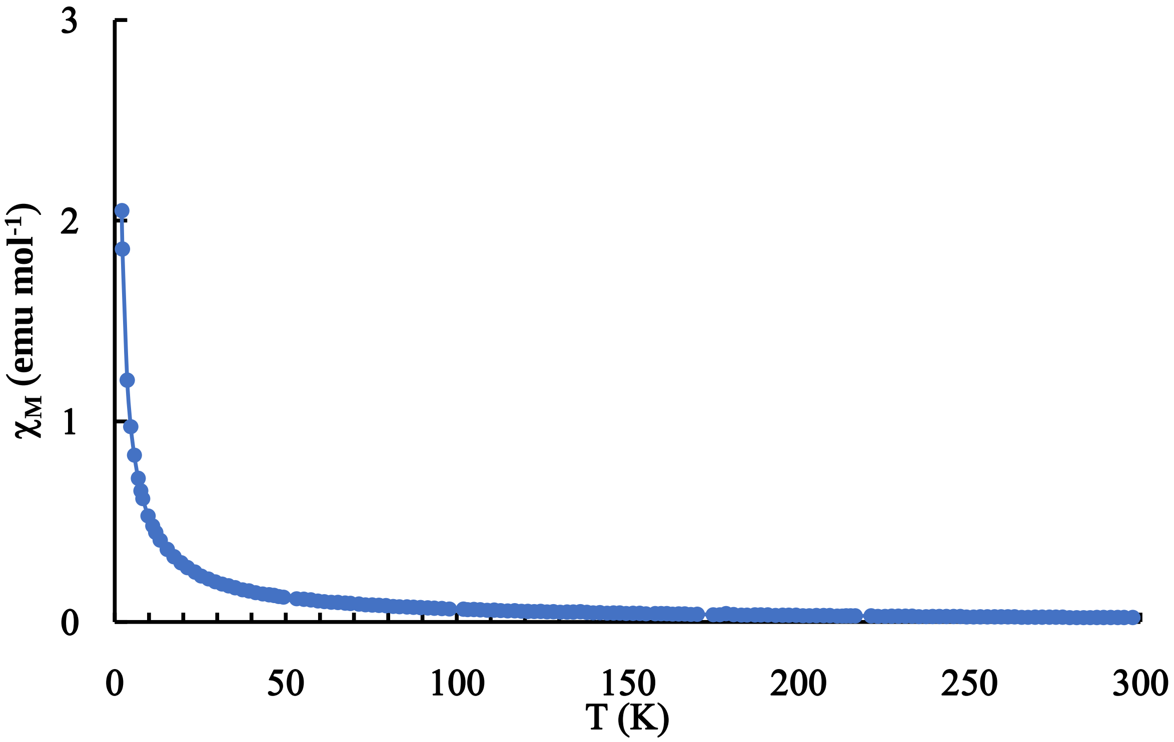


Figure 3: Experimental variations of $\chi_M$versus$T$ for complex **1**at 1000 Oe.

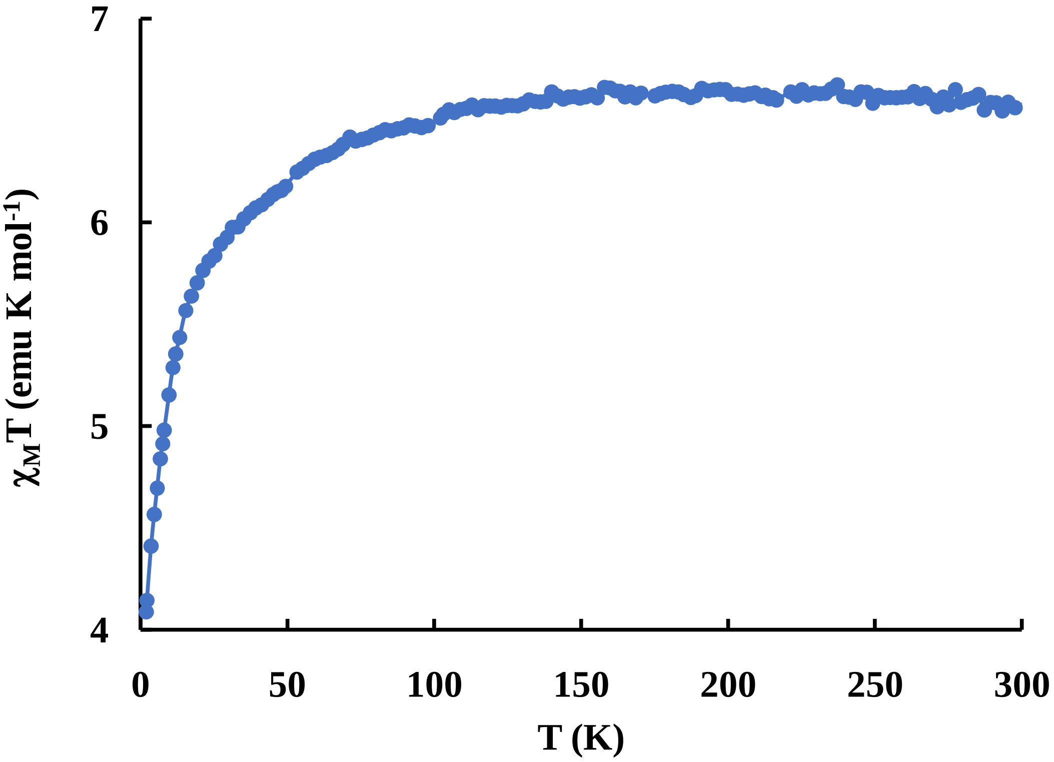


Figure 4: Experimental variations of $\chi_M T$ versus T for complex **1**at 1000 Oe.

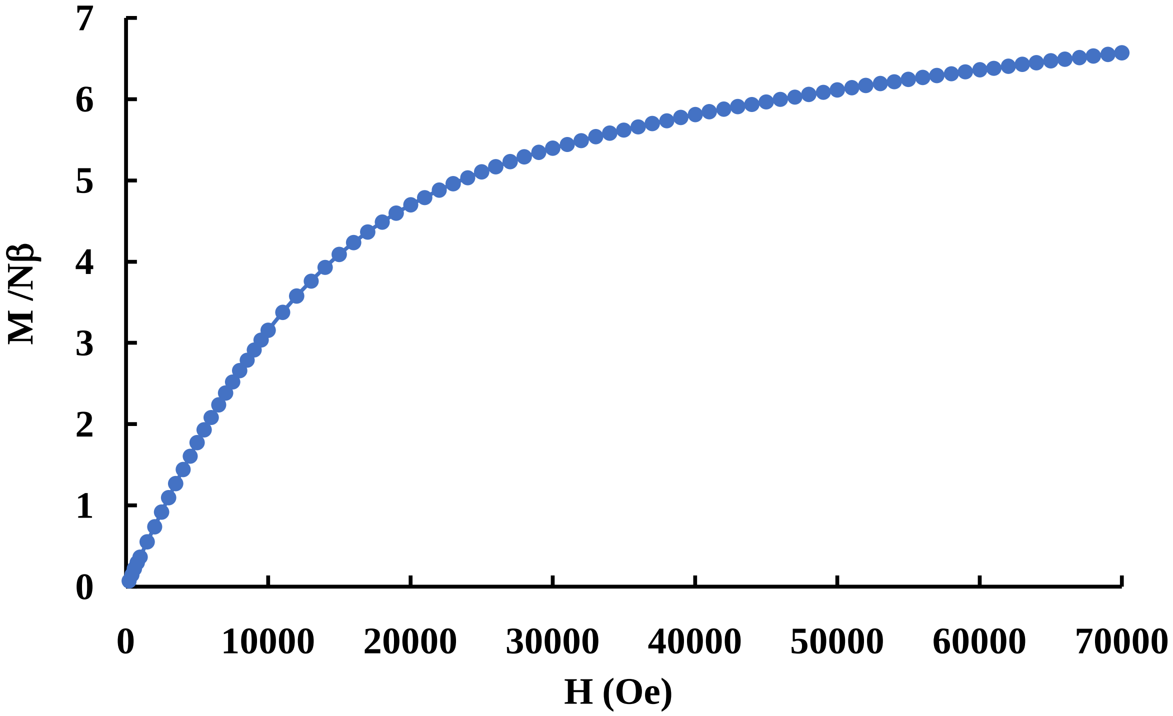


Figure 5: Isothermal magnetization of **1** at 2 K.

IV. Concluding remarks

In this present work, we synthesized a multinuclear complex with mixed valence [Co^{III}—Co^{II}—Co^{III}]using *N*,*N*-dimethyl-*N*'-((5-methyl-1*H*-imidazol-4-yl)methylene)ethane-1,2-diamine (HL) in the presence of potassium thiocyanate. In the structure each Co^{III} ion is coordinated by one neutral ligand molecule and bridged to the Co^{II} by another monodeprotonated ligand molecule.Each Co^{III} ion is coordinated to one thiocyanate group through the terminal nitrogen atomwhile the central Co^{II} is linked to four thiocyanate groups in similar fashion. The terminal Co^{III} ions as well as the central Co^{II} ion are in octahedral environment in a N_6 core. The molecules of the complex are arranged in layers parallel to [011] direction. The temperature variable magnetic measurement shows that two Co^{II} ions isolated and no significative interaction was observed.

V. Supporting information

CCDC-2010963 contains the supplementary crystallographic data forthis paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by emailingdata_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 UnionRoad, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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