ISSN 2394-9651

International Journal of Novel Research in Physics Chemistry & Mathematics
Vol. 10, Issue 1, pp: (60-66), Month: January - April 2023, Available at: www.noveltyjournals.com

Crystal structure of Y(III) Complex with the Tridentate Schiff Base Ligand *N'*– (1–(pyridin–2–yl)ethylidene)nicotinohydrazide

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DOI: https://doi.org/10.5281/zenodo.7876199

Published Date: 28-April-2023

Abstract: **The use of *N'*–(1–(pyridin–2–yl)ethylidene)nicotinohydrazide (HL) in Yttrium (III) chemistry has yielded dinuclear complex. The 1:1 $Y(CH_3COO)_3.6H_2O$/HL in methanol afforded the complex $\{[Y(L)(\eta^2–OOCCH_3)(H_2O)](\eta^1:\eta^2:\mu–OOCCH_3)_2[Y(L)(\eta^2–OOCCH_3)(H_2O)]\}$. The structure of the complex were solved by single crystal X–ray crystallography. In the complex the Y^{3+} atoms are doubly bridged by two acetates anions and each metal ion is coordinated by one tridentate monodeprotonated molecule ligand, one bidentate acetate group and one coordinated water molecule. Each Y^{3+} atom is nine–coordinated with an environment best described as a tricapped prismatic geometry. Complex crystallizes in the monoclinic space group $P2_1/n$ with the following parameters : a = 11.3775(11) Å, b = 14.2462(16) Å, c = 12.0082(12) Å, β = 100.728(10)°, V = 1912.3(3) Å^3, Z = 8, R_1 = 0.0345, wR_2 = 0.0797. The supramolecular structures are consolidated by multiple hydrogen bonds.**

Keywords: **Schiff base, dinuclear complex, Yttrium, tricapped prism.**

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1. INTRODUCTION

Since a long time, chelating agents derived from organic compounds containing N,S,O-donor as a functional group have a strong ability to form metal complexes and exhibit a variety of biological activities [1-8]. A number of studies have been done in the various Schiff bases complexes formed by the condensation of secondary amines with different aldehydes and ketones [9-12]. From the survey of existing literature, it appears that metal complexes of Schiff bases played a vital role in the development of coordination chemistry and their analytical utility in the determination of transition metal ions. Literature studies revealed that during the past decades, there has been a great deal of interest in the synthesis and structural elucidation of transition metal complexes containing N,N,O,O-donor. This article describes the tridentate Schiff base obtained by the reaction between nicotinohydrazide and acetylpyridine. The flexibility and the numerous coordination sites of the resulting acyclic ligand provide several possible structures with lanthanide ions [13–15]. Using the ligand *N'*–(1–(pyridin–2–yl)ethylidene)nicotinohydrazide (HL) in presence of Yttrium acetate salts yield complexes of Y^{3+} with different behavior of the anions. The complex is characterized by IR spectra, room temperature magnetic measurement and elemental analyses. The structure of Y(III) complex was determined by X–ray single crystal diffraction.

2. MATERIALS AND METHODS

2.1. Starting materials and instrumentations

2–acetylpyridine and nicotinohydrazide were commercial products (Aldrich) and were used without further purification. Solvents were of reagent grade and were purified by the usual methods. 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 ^1H NMR spectra were recorded at 300 MHz and ^{13}C NMR spectra at 75 MHz on a Bruker AC–300 instrument.

2.2. Synthesis of *N*'–(1–(pyridin–2–yl)ethylidene)nicotinohydrazide (HL)

In a 100 mL flask, 0.7566 g (5.5 mmol) of nicotinohydrazide and 20 mL of methanol were mixed. A methanolic solution (20 mL) of acetylpyridine 0.6627g (5.5 mmol) was added dropwise. The resulting yellow mixture was heated under stirring at reflux for 3 hours. On cooling, the yellowish solution was left on slow evaporation for a few days. The white solid which appears was recovered by filtration, washed with ether (2 x 10 mL), and dried in the air.

Yield : 55%. Anal. Calc for $C_{13}H_{12}N_4O$: C, 64.99 ; H, 5.03 ; N, 23.32. Found : C, 65.02 ; H, 5.05 ; N, 23.29.

NMR ^1H (300 MHz, DMSO–d6) (δ, ppm) : 11.13 (s, N**H**,1H); 9.05–7.43 (m, **H**–Ar, 8 H) ; 2.53 (s, –C**H3**–, 3 H). NMR ^{13}C [DMSO, 300 MHz, (δ, ppm)] : 13.32 (**CH**3–) ; 120.92 (**C**Ar); 120.96 (**C**Ar) ; 123.89 (**C**Ar) ; 123.92 (**C**Ar) ; 130.21 (**C**Ar) ; 136.37 (**C**Ar) ; 137.18 (**C**Ar) ; 149.18 (**C**Ar) ; 152.84 (**C**Ar) ; 155.48 (**C**Ar) ; 155.78 (**C**=N) ; 163.37 (**C**=O).

IR (cm^{-1}) : 3500, 1642, 1596, 1539, 1494, 1480, 1030, 794, 734, 700.

2.3. Synthesis of the complex {[Y(L)(η^2–$OOCCH_3$)(H_2O)](η^1:η^2:μ–$OOCCH_3$)$_2$[Y(L)(η^2–$OOCCH_3$)(H_2O)]}

In a 100 mL flask containing methanol (10 mL) was added (0.1 g, 0.42 mmol) of the HL ligand. The mixture was stirred until complete dissolution of the organic compound. A 10 mL methanolic solution containing $Y(OAc)_3.6H_2O$ (0.1336 g, 0.42 mmol) was added. The resulting solution was stirred and heated at reflux for 3 hours. On cooling the precipitate which appears was collected by filtration, washed with ether (2 x 10 mL) and air dried. The filtrates were left under slow evaporation. Few days after, yellow crystal of the Y(III) complex, suitable for X–ray diffraction, were collected.

Yield 48,64 %. Anal. Calc for $C_{34}H_{34}N_8O_{12}Y_2$: C, 43.75 ; H, 4.29 ; N, 12.01. Found : C, 44.01 ; H, 4.31 ; N, 12.04.

IR (cm^{-1}) : 1639, 1556, 1434, 1305, 1197, 1030, 1077, 925, 858, 776.

Conductance. ($S.cm^2.mol^{-1}$) : 17.3 and 20.4, 15 days after.

2.4. X–ray crystallography

Crystals suitable for single–crystal X–ray diffraction, of the reported compound, was grown by slow evaporation of DMF solution of the compound. Details of the crystal structure solution and refinement are given in Table. Diffraction data were collected using a Bruker APEX–II CCD diffractometer with graphite monochromatized MoKα radiation (λ= 0.71073 Å). All data were corrected for Lorentz and polarization effects. The structure was solved and refined using the Bruker SHELXTL Software Package [16]. All the structures were refined on *F*2 by a full–matrix least–squares procedure using anisotropic displacement parameters for all non–hydrogen atoms [17]. H atoms of the NH groups was located in the Fourier difference maps and refined without restraints. Other H atoms were geometrically optimized and refined as riding on their carriers with

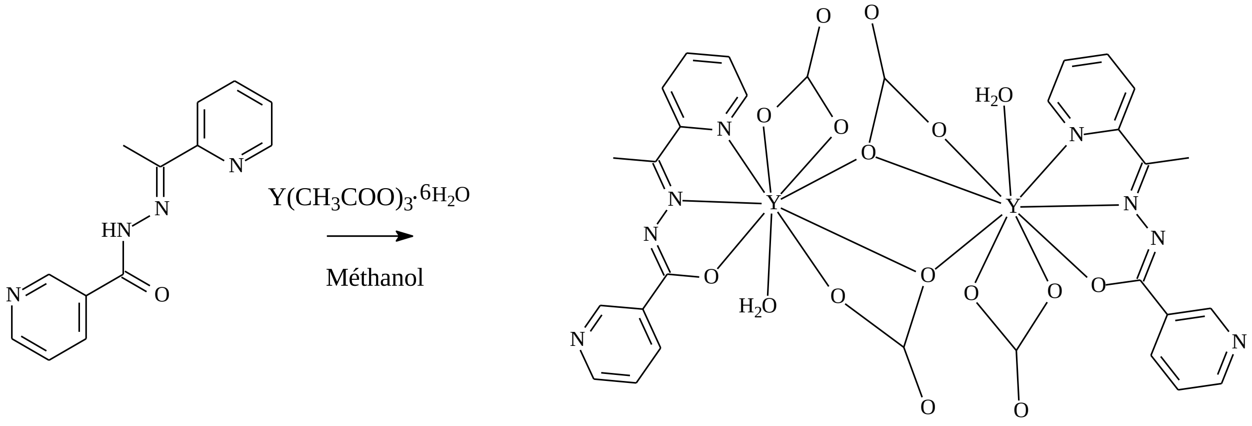
Uiso(H) = 1.2Ueq(C) (1.5 for CH_3 group). Molecular graphics were generated using ORTEP–3 [18].

3. RESULTS AND DISCUSSION

3.1. General study

HL react with $LnX_3.6H_2O$ (Ln = Y, X = CH_3COO^-) to afford dinuclear complex for Y(III) as shown in scheme. The infrared spectrum of the HL ligand shows two bands pointed at 1656 cm^{-1} attributed to the vibration of the C=O and at 1583 cm^{-1} attributed to C=N. The vibration band of the N–H bond appears at 3500 cm^{-1}, while the band of the N–N moiety is pointed at 1030 cm^{-1}. Additional bands, characteristic of the aromatic ring, are pointed in the range 1596–1471 cm^{-1}.

The ^1H NMR spectrum reveals a complex signal between 7.43 ppm and 9.05 ppm attributed to the protons of the aromatic rings. The signal which appears as a doublet at 2.53 ppm is attributed to the proton of the methyl group. The signal at 11.13 ppm is assigned to the HN of the hydrazine moiety. The assignations of these signal are correlated to the signals of the ^{13}C NMR spectrum which reveals a signal at 13.32 ppm indicative of the presence of –CH_3 group and a signal at 155.48 ppm attributed to imine carbon atoms. The signal at 163.37 ppm is attributed to the **C**=O. Additional signals in the range 120 ppm–137 ppm are attributed to the carbon atoms of the pyridine rings.



Scheme : Synthesis procedure of the complex

Upon coordination the band due to C=N shift to low frequencies for complex. For compound the bands of the C=N is pointed at 1556 cm^{-1}. This fact is indicative of the involvement of the azomethine nitrogen atom in the coordination to the Yttrium ion. Asymmetric and symmetric bands of COO group in acetic acid are respectively 1585 cm^{-1} (ν_{as}) and 1397 cm^{-1} (ν_s) [19]. Upon coordination to Yttrium ion the IR bands of the acetate groups were shifted. In the IR spectrum of complex, the ν_{as}(COO) shifted to 1455 cm^{-1}, while the ν_s(COO) appears at 1314 cm^{-1}. The $\Delta\nu = (\nu_{as}-\nu_s)$ of the carboxylate group is a criterion used to determine the coordination mode of the carboxylate moiety to the metal ion [20,21]. When $\Delta\nu$ varies between 160 and 175 cm^{-1}, the acetate group acts as counter ion. When the value of $\Delta\nu$ is larger than 175 cm^{-1}, the acetate group acts in monodentate fashion. Lower value of $\Delta\nu$ (< 160 cm^{-1}) is indicative of an acetate group acting in bidentate manner. Considering the $\Delta\nu$ value of 141 cm^{-1} for the complex, we can say that the acetate group acts in bidentate chelating fashion to the Yttrium(III) ion. The Yttrium complex has a negative paramagnetic contribution because it has no single electrons: it is therefore diamagnetic.

Molar conductivities were measured for freshly prepared solutions in DMF and after standing for two weeks. The conductivities increased very slightly with time in DMF for both complexes. The coordination moieties present cannot be replaced by the solvent molecules. Molar conductance value of 17,3 $Ohm^{-1}cm^2mol^{-1}$ and 20.4, Scm^2mol^{-1} in DMF 15 days after is indicative complex is neutral electrolyte [22]. The results of the elemental analysis, the IR spectra data, magneticmoment measurements and conductivities measurements allow to formulate the Yttrium(III) complexes as dinuclear structure$\{[Y(L)(\eta^2\text{–}OOCCH_3)(H_2O)](\eta^1:\eta^2:\mu\text{–}OOCCH_3)_2[Y(L)(\eta^2\text{–}OOCCH_3)(H_2O)]\}$. These spectroscopic observations on the geometrical features are in accordance with those obtained for X–ray crystallography analyses of the Y(III) complex.

Table 1: Crystal data and details of the structure determination for complex

Empirical Formula	$0.25(C_{34}H_{40}N_8O_{12}Y_2)$
Formula weight /g	233.139
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	11.3775(11)
b/Å	14.2462(16)
c/Å	12.0082(12)
α/°	90

β/°	100.728(10)
γ/°	90
Volume/Å^3	1912.3(3)
Z	8
ρ_{calc}g/cm^3	1.616
μ/mm^{-1}	3.092
F(000)	948.0
Radiation	Mo Kα (λ = 0.71073)
θ range for data collection /°	2.68 à 29.34
Index ranges	$-12 \leq h \leq 15$, $-19 \leq k \leq 19$, $-15 \leq l \leq 16$
Reflections collected	10586
R Indices (all data)	$R_1 = 0.0345$, $wR_2 = 0.0797$

3.2. Molecular Structure of Complex

The complex crystallizes in the monoclinic system with the space group $P2_1/n$. Partially labelled plot of the dinuclear structure of Y(III) complex is shown in Figures 1 and 2. The structure of the complex is consistence with the {[Y(L)(η^2–OOCCH$_3$)(H$_2$O)](η^1:η^2:μ–OOCCH$_3$)$_2$[Y(L)(η^2–OOCCH$_3$)(H$_2$O)]} formulation. The asymmetric unit contains two Y^{3+}, two monodeprotonated organic ligand, four acetate ions and two water molecules. The crystal structure is formed by two analogous entities of [Y(L)(η^2–OOCCH$_3$)(H$_2$O)] with are bridged by two acetate groups acting in η^1: η^2 :μ–OOCCH$_3$ mode. Each Y^{3+} is coordinated by one ligand molecule through one azomethine nitrogen atom, one pyridine nitrogen atom and one carbonyl oxygen atom resulting in two membered chelating rings YOCCN and YNCCN. The Y^{3+} ion is coordinated by one acetate group in chelating–bidentate mode and one water molecule. The Y^{3+} ion is nine–coordinated. The Y–O distances are in the range 2.314–2.440 Å and are typical of a chelating–bidentate acetate group [23]. The two metal centers are finally bridged by a pair of chelating–bridging acetato ligands which are acting in η^1:η^2:μ_2–OOCCH$_3$ mode. Two different bonds values are noted : two typical bond values of 2.324 Å and 2.324 Å (Y1–O5 and Y1–O4) and 2.378 Å (Y1–O2). These values are comparable to values noted from literature [24]. The largest Y–O (Y1–O1 = 2.440 Å) distance is observed in the chelating–bridging coordination mode η^1:η^2:μ_2–OOCH$_3$, while the shortest Y–O (Y1–O6 = 2.426 Å) bond length is observed in the chelating coordination mode η^2–OOCCH$_3$. The Y–N distances are 2.472 Å and 2.510 Å. The longest Y–N distance is due to the nitrogen atom belonging to pyridine ring, as observed for similar complexes [25]. The C–O bond in the coordinated acetate groups has an intermediate character between single C–O bond and double C=O bond. In fact, the bond lengths values observed in both coordinated acetate groups for C–O [1.245 Å–1.283 Å] are shorter than those of a single C–O bond (1.430 Å) and longer than those of a double bond (1.220 Å). The N3–C7 bond length value of 1.284 Å is consistent with double bond character. The Y···Y distance bridged by two acetate anions is 4.4774 Å. All the bond lengths are normal and fall within similar ranges to those reported for dinuclear lanthanide complexes with the same hydrazone ligand [25,26].

The chelation of the hydrazino ligand to the Y^{3+} cation results in two five–membered rings (YNCNN and YNCCN) with bite angles of 63.54(14)° (N3–Y1–N4). The bond angles of the ligands, which involve the Y(III) ion, are slightly largest than the angle subtended by the oxygen atoms of the bidentate chelating acetate groups : O2–Y1–O3 = 53.57(13)° and O5–Y1–O6 = 50.05(12)°. These angle values are comparable to the values reported for the complex [Pr($C_9H_4N_2O_4$)($C_2H_3O_2$)(H$_2$O)]$_2$ [23]. The bond angle value of 68.23(7)° of the bridged oxygen atoms of the η^1:η^2:μ_2–acetate groups [O5–Y–O5i] is comparable with the angle value observed in homologous complex [Tb$_2$(CH$_3$COO)$_6$(H$_2$O)$_4$]·4H$_2$O [27]. The value of the bridging angle Y1–O5–Y2 [111.77(7)°] is in the range expected for η^1:η^2:μ_2–acetate groups [28]. Two coordination polyhedral with nine vertices are known. The coordination sphere around the nine–coordinated Yttrium atom is best described as a distorted tricapped trigonal prism in which N4, O2 and O5 are the caps as shown in Figure 1.

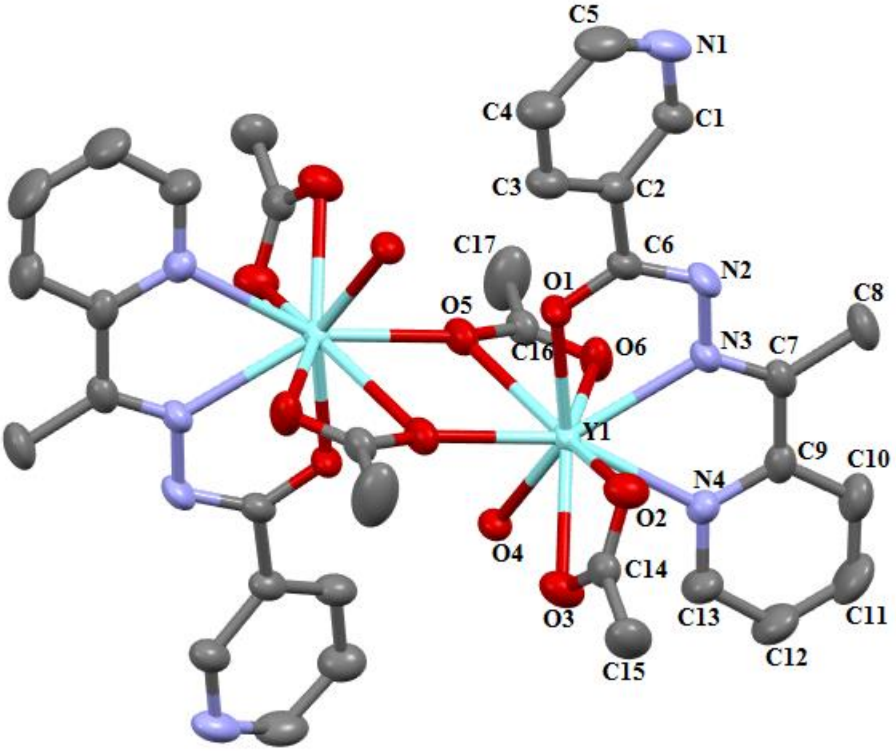


Figure 1. The structure of complex without hydrogen atoms

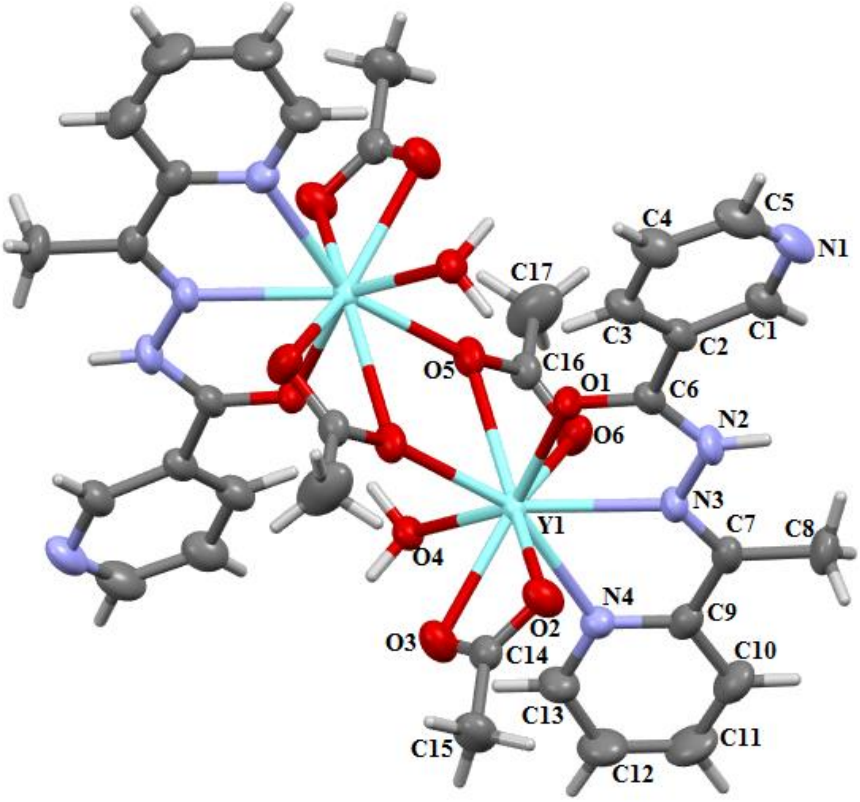


Figure 2. The structure of complex with hydrogen atoms

4. CONCLUSION

The reported work is concerned the synthesis of a Schiff base and its use to prepare a new dinuclear Y(III) complex. The reaction of the Schiff base and $Y(CH_3COO)_3{\cdot}6H_2O$ salts afford dinuclear $\{[Y(L)(\eta^2\text{–}OOCCH_3)(H_2O)](\eta^1{:}\eta^2{:}\mu\text{–}OOCCH_3)_2[Y(L)(\eta^2\text{–}OOCCH_3)(H_2O)]\}$ complex. In the yielded crystals, the Y(III) ion is nine coordinated. In the dinuclear complex the two Y(III) ions are bridged by two acetate groups acting in $\eta^1{:}\eta^2{:}\mu\text{–}OOCCH_3$ mode. The geometries around the two Y(III) ions in complex are best described as a distorted tricapped trigonal prism.

5. SUPPORTING INFORMATION

CCDC-2087203 and 2087202 contain the supplementary crystallographic data for complex. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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