

# SYNTHESIS, SPECTRAL STUDY AND ANTIBACTERIAL ACTIVITY OF ASYMMETRICAL TETRADENTATE SCHIFF BASE COMPLEXES

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## ABSTRACT

The Ligand 3,3'-((1E,1'E)-((4-methyl-1,2- phenylene)bis(azanylylidene))bis(methanylylidene))bis(naphthalene-2-ol) ( L ) have been used to synthesize four new Cr(III), Co(II), Zn(II), and Cd(II) complexes (1-4). The Ligand (L) was derived from the condensation of 3, 4- diamino Toluene and 2- hydroxy naphthaldehyde in 1:2 molar ratio. The ligand and four new metal complexes were characterized by Spectroscopic studies. Physical methods such as Melting points, Elemental Analysis, and Magnetic moment properties. The FT-IR spectral data indicate that the synthesized ligand was coordinated with the metal ion in a tetradentate manner. The UV/Visible spectra and magnetic moment data showed octahedral geometry for Cr(III) and Co(II) complexes whereas Zn(II) and Cd(II) showed distorted square pyramidal structure. The magnetic moment of metal complexes was low as expected. The synthesized metal complexes were insoluble in many organic solvents thus supports the proposed structure. The tetradentate Schiff base ligand and complexes (1-4) were tested against bacteria *E. coli*, *B. megaterium* and *P. aeruginosa* for their antibacterial activity for the determination of inhibition potential.

**Keywords:** Antibacterial Activity, Octahedral Geometry, Square Pyramidal Geometry, Schiff Base Complexes, Spectral Data.

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## INTRODUCTION

Many organic compounds containing naphthaldehyde moieties have been studied in coordination chemistry due to their facile synthesis, electronic properties and therapeutic treatments for diseases.<sup>1,2</sup> The study of d block elements with aromatic Schiff bases have been focused as a result of their various biological activities, structural varieties, potential application,<sup>3</sup> photochromic properties<sup>4-7</sup> and synthetic applicabilities.<sup>8</sup> Schiff bases are synthesized by the condensation of aldehyde/ ketone and primary amines in the presence of base or acid catalyst, resulting in the formation of azomethine(-CH=N-) group. The heavy metal Schiff base complexes have been received increasing attention owing to their remarkable characteristics, biochemical, pharmacological properties<sup>9</sup>, biological activities<sup>10,11</sup>, catalytic activity<sup>12,13</sup>, antibacterial<sup>14-17</sup>, anticancer<sup>18-20</sup>, antitumoral activities.<sup>21, 22</sup> Schiff base derived from aromatic aldehyde or ketones were capable of forming bidentate, tridentate, or tetradentate complexes with certain transition metal ions like Cr(III), Co(II), Fe(II), Mn(II), Zn(II), Cd(II), etc having many biological properties due to their electronic configuration, size, varied denticity, and steric reasons.<sup>3,4,23</sup> During the past decades, Schiff base metal complexes of Cr(III), Co(II), Cd(II), Zn(II) obtained from 2- hydroxynaphthaldehyde showed a wide range of geometries which are octahedral, tetrahedral and distorted square pyramidal.<sup>24,25</sup> Among the transition metal, Zinc, cadmium and cobalt have wide importance in biological process.<sup>2,5,26</sup> Cd(II) and Zinc(II) complexes of Schiff base have potential applications in fluorescence study and luminescence properties, due to electrons delocalization around the conjugated system of aromatic rings.<sup>9,23,27</sup> Also, naphthaldehyde based Schiff base derivatives of Co(II) and Cu(II) complexes used as urease inhibitor and anticancer drugs.<sup>18,28</sup> Recent reports have shown that cobalt(II) complexes with Schiff

base having aromatic N- substituent used as coordination network, polymers and metal-organic frameworks.<sup>29</sup>

In this paper, we synthesized a new asymmetrical mononuclear Schiff base metal complexes of Cr(III), Co(II), Zn(II), and Cd(II) derived from ligand 3,3'-((1E,1'E)-((4-methyl 1,2-phenylene)bis(azanylylidene))bis(methanylylidene))bis(naphthalene-2-ol), which were prepared by the condensation of 2- hydroxy 1-naphthaldehyde and 3,4-diamino toluene (Scheme-1). All the complexes (**1-4**) and tetradentate Schiff base ligand were identified and characterized by spectral techniques using NMR, IR, ESR, UV-Visible, Mass spectra, Elemental analysis in continuation with magnetic moment methods. Furthermore, the antibacterial properties of the ligand and mononuclear complexes (**1-4**) were tested against bacteria *E. coli*, *B. megaterium* and *P.aeruginosa* for the determination of inhibition potential.

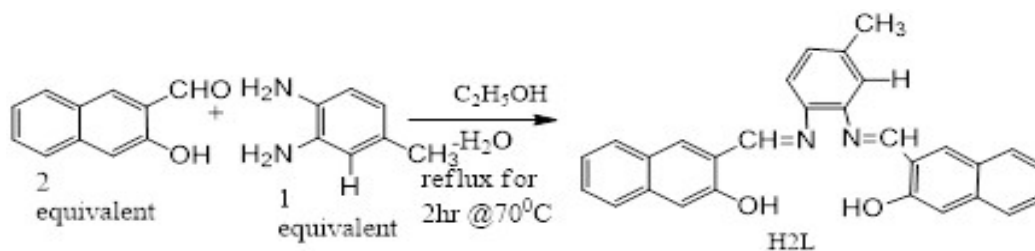
## EXPERIMENTAL

### Material and Methods

All A.R. grades chemicals and solvents were used. Elemental analysis data were recorded using a Perkin-Elmer CHN 2400 elemental analyzer. The UV/Visible spectra were recorded by Perkin-Elmer LAMBDA 25 spectrophotometer. Infra-Red spectra were recorded on a Thermo-fisher Nicolet iS5 FTIR spectrometer. <sup>1</sup>HNMR and <sup>13</sup>CNMR spectra were recorded using a Bruker AVANCE III 500 MHz NMR (AV 500) spectrometer in DMSO-d<sub>6</sub>. ESR spectra of the complexes were recorded at an EMX micro A200-9.5/12/S/W, BRUKNER BIOSPIN. Mass spectra of the ligand and metal complexes were recorded on a AB SCIEX Triple TOF 5600+ LCMS-MS spectrometer. Gouy method was used to determine the magnetic moment of metal complexes using Hg[Co(SCN)<sub>4</sub>], as calibrant at room temperature.

### Synthesis of Ligand (L) 3,3'-((1E,1'E)-((4-methyl-1,2-phenylene)bis(azanylylidene))bis(methanylylidene))bis(naphthalene-2-ol)

An ethanolic solution of 2-hydroxy naphthaldehyde (25 ml) (0.02398mol, 4.130g) was added dropwise, over a stirred solution of 3,4- diamino toluene (0.01195 mol, 1.460g) in warm ethanol (45 ml). The reaction mixture was refluxed with stirring on oil bath at 70-80°C for 2-3 hrs. A yellow solid was filtered off, washed with ethanol, diethyl ether and dried. The obtained product was purified by recrystallization from DMF. Yield: 75% (4.20gm), M.p. 220°C. Anal. Calcd. For C<sub>29</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>: C, 80.91; H, 5.15; N, 6.51; O, 7.43. Found: C, 80.78; H, 5.08; N, 6.45; O, 7.34. Selected FT-IR (KBr, cm<sup>-1</sup>): 3328(O-H), 2918(C-H aromatic), 1619(C=N), 1291(C-O phenolic). <sup>1</sup>HNMR (500MHz, DMSO-d<sub>6</sub>, ppm): δ 2.34 (d, CH<sub>3</sub>), 7.17-7.57(m, CH aromatic), 8.22-8.41(dd, HC=N), 15.14-15.36(s, OH). <sup>13</sup>CNMR (100.622 MHz, DMSO-d<sub>6</sub>): δ 21.23, 118.86, 119.29, 122.23, 127.46, 133.23, 137.60, 139.93, 168.63, 169.48. Mass spectrum(LC/MS,ESI): m/z 430.17, Found: 430.50[M]<sup>+</sup>.



Scheme-1: Synthesis of Ligand

### General Procedure For the Synthesis of Metal Complexes

Metal complexes (**1** to **4**) were prepared with ligand (L), 0.230g (0.0010mol) of Cr(CH<sub>3</sub>COO)<sub>3</sub>, 0.250g (0.0010mol) of Co(CH<sub>3</sub>COO)<sub>2</sub>·4H<sub>2</sub>O, 0.220g (0.00095mol) of Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O, 0.270g (0.0010mol) of Cd(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O, was dissolved in 20 ml DMF separately, then added dropwise to a magnetically stirred solution of ligand (L) 0.430g (0.0010mol) in DMF (20ml) in 1:1 molar ratio (M:L). The resultant mixture was refluxed and stirred on an oil bath for 3 hrs and then poured on to ice-cold water.

Finally the brown, cherry red, yellow, and dark yellow colored solid for complexes (1-4) precipitated respectively. The solid colored compounds were filtered and washed with diethyl ether. The solid products were recrystallized in DMF/ DMSO and dried over  $\text{CaCl}_2$  in a vacuum.

### Complex 1

Yield: 0.610g (92.42%), M.p.  $310^\circ\text{C}$ , Anal. Calcd. for  $\text{C}_{31}\text{H}_{24}\text{N}_2\text{O}_5\text{Cr}$ : C, 66.90; H, 4.35; N, 5.03; Cr, 9.34; O, 14.37. Found: C, 66.27; H, 4.10; N, 4.85; Cr, 9.13; O, 14.14. Selected FTIR spectrum (KBr,  $\text{cm}^{-1}$ ): 3423 (free  $-\text{OH}$ ), 2920 (C-H aromatic), 1616 (C=N), 1281 (C-O, phenolic), 563 (Cr-O), 500 (Cr-N). Mass spectrum (LC/MS, ESI):  $m/z$  556.11, Found: 558 $[\text{M}+2]^+$ .

### Complex 2

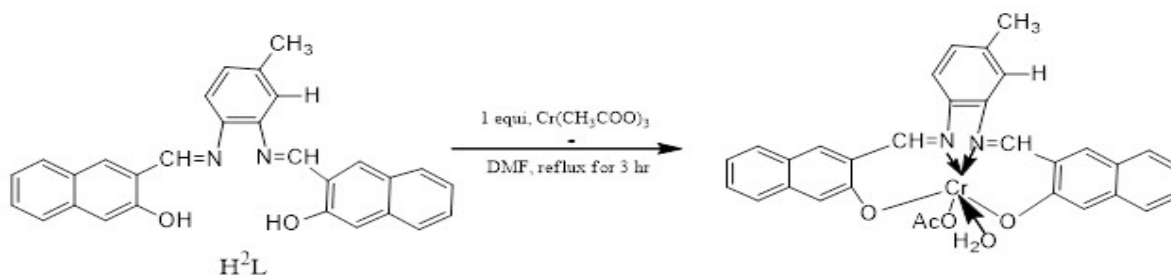
Yield: 0.510g (75%), M.p.  $290^\circ\text{C}$ , Anal. calcd. for  $\text{C}_{31}\text{H}_{24}\text{N}_2\text{O}_5\text{Co}$ : C, 66.08; H, 4.29; N, 4.97; Co, 10.46. Found, C, 65.76; H, 4.10; N, 4.56; Co, 10.23. Selected FTIR spectrum (KBr,  $\text{cm}^{-1}$ ): 3441 (free-OH), 2921 (C-H aromatic), 1614 (C=N), 1280 (C-O, phenolic), 571 (Co-O), 488 (Co-N). Mass spectrum (LC/MS, ESI):  $m/z$  563.10, Found: 566 $[\text{M}+3]^+$ .

### Complex 3

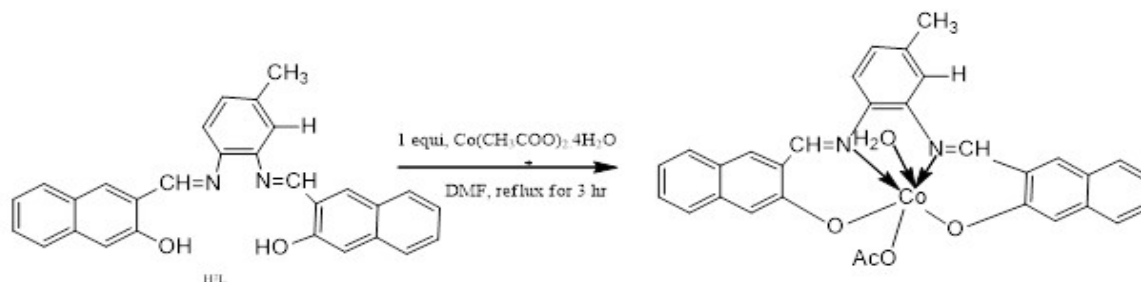
Yield: 0.510g (78.46%), M.p.  $340^\circ\text{C}$ , Anal. calcd. for  $\text{C}_{29}\text{H}_{21}\text{N}_2\text{O}_3\text{Zn}$ : C, 68.18; H, 4.14; N, 5.48; Zn, 12.80. Found C, 68.10; H, 4.05; N, 5.23; Zn, 12.20. Selected FTIR spectrum (KBr,  $\text{cm}^{-1}$ ): 3422 (free OH), 2919 (C-H, aromatic), 1616 (C=N), 1244 (C-O, phenolic), 583 (Zn-O), 546 (Zn-N).  $^1\text{H}$ NMR spectrum (500MHz, DMSO- $d_6$ , ppm):  $\delta$  2.49 (d,  $\text{CH}_3$ ), 7.22-7.77 (m, C-H aromatic), 8.45-9.80 (dd, HC=N).  $^{13}\text{C}$ NMR spectrum (DMSO- $d_6$ , 100.622MHz):  $\delta_c$  21.48, 117.34, 119.94, 122.11, 127.91, 136.16, 138.26, 156.30, 174.00. Mass spectrum (LC/MS, ESI):  $m/z$  510.08, Found: 512.49 $[\text{M}+2]^+$ .

### Complex 4

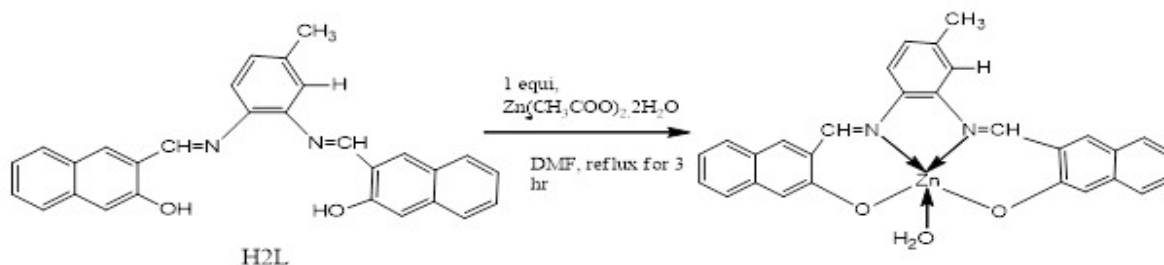
Yield: 0.520g (74.28%), M.p.  $180^\circ\text{C}$  Anal. calcd. for  $\text{C}_{29}\text{H}_{21}\text{N}_2\text{O}_3\text{Cd}$ : C, 64.40; H, 3.73; N, 5.18; Cd, 20.78. Found, C, 64.23; H, 3.56; N, 5.09; Cd, 20.38. Selected FTIR spectrum (KBr,  $\text{cm}^{-1}$ ): 3423 (free OH), 2920 (C-H aromatic), 1618 (C=N), 1246 (C-O, phenolic), 582 (Cd-O), 532 (Cd-N).  $^1\text{H}$ NMR spectrum (500MHz, DMSO- $d_6$ , ppm):  $\delta$  2.50 (s,  $\text{CH}_3$ ), 7.35 (m, C-H aromatic), 8.25-8.94 (m, HC=N), 15.12-15.35 (s, free  $-\text{OH}$ ).  $^{13}\text{C}$ NMR (DMSO- $d_6$ , 100.622MHz):  $\delta_c$  21.24, 119.03, 122.10, 123.48, 127.47, 127.95, 129.34, 136.66. Mass spectrum (LC/MS, ESI):  $m/z$  559.06.



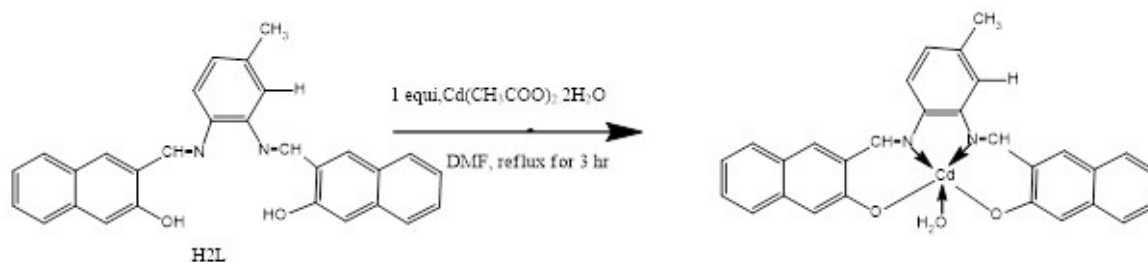
Scheme-2: Synthesis of Cr(III) Complex



Scheme-3: Synthesis of Co(II) Complex



Scheme-4: Synthesis of Zn(II) Complex



Scheme-5: Synthesis of Cd(II) Complex

## RESULTS AND DISCUSSION

The reactions between metal acetates (Cr, Co, Zn, Cd) and ligand (L) (Schemes-2 to 5) were carried out in molar ratio (1:1) and coloured compounds were obtained (1-4). The spectroscopic techniques such as FTIR,  $^1\text{H}$ NMR,  $^{13}\text{C}$  NMR, Mass spectra, EPR spectra and Elemental analysis were used, to verify the coordination of ligand with the metal ions. All compounds were stable at room temp. and were soluble in DMSO/ DMF.

### IR Spectra

The FTIR spectra of ligand (L), exhibited medium intensity stretching band at  $3328\text{ cm}^{-1}$  which was characteristic of phenolic OH group<sup>25</sup>. The  $\nu(\text{CH}=\text{N})$  stretching band of the azomethine group appears at  $1619\text{ cm}^{-1}$  for ligand, which were shifted to a lower frequency, assigned at  $1616\text{ cm}^{-1}$ ,  $1614\text{ cm}^{-1}$ ,  $1616\text{ cm}^{-1}$  and  $1618\text{ cm}^{-1}$  for complexes 1, 2, 3 and 4 respectively. The shifting of these vibrational bands was due to the coordination of azomethine nitrogen atom with that of the metal ion. The phenolic C-O stretching band persists at  $1291\text{ cm}^{-1}$  in Schiff base ligand whereas these bands were showed red shift at  $1281\text{ cm}^{-1}$ ,  $1280\text{ cm}^{-1}$ ,  $1244\text{ cm}^{-1}$  and  $1246\text{ cm}^{-1}$  in metal complexes 1, 2, 3 and 4 respectively. These results revealed that the metal ions were coordinated with oxygen atoms.<sup>31</sup> Some new additional vibrational bands appeared in the range  $583\text{--}563\text{ cm}^{-1}$  and  $546\text{--}488\text{ cm}^{-1}$  was assigned to  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  respectively.<sup>32</sup> The spectra of complexes (1 to 4) exhibited stretching vibrational bands at  $3423$ ,  $3441$ ,  $3442$  and  $3423\text{ cm}^{-1}$ , assigned to coordinated water molecules associated with the complexes<sup>25</sup>.

### UV/Visible Spectra

The electronic spectra of Land metal complexes (1 to 4) were recorded in DMSO solution. All the complexes showed a blue shift in the spectra on comparing with ligand and some new bands also appeared in the spectra of metal complexes. The UV/visible spectra of ligand exhibited three sharp peak at  $273\text{ nm}$ ,  $322\text{ nm}$  and  $375\text{ nm}$ , attributed to  $\pi - \pi^*$ ,  $n - \pi^*$  and charge transfer transition respectively. These transitions were assigned to the aromatic ring and azomethine ( $\text{CH}=\text{N}$ ) group respectively<sup>33</sup>. The UV/Visible spectra of Cr(III) complex (1) exhibit three absorption peaks at  $267\text{ nm}$ ,  $311\text{ nm}$  and  $475\text{ nm}$ , which attributed to  $^4\text{A}_{2g} \rightarrow ^4\text{T}_{1g}(\text{P})$ ,  $^4\text{A}_{2g} \rightarrow ^4\text{T}_{1g}(\text{F})$ , and  $^4\text{A}_{2g} \rightarrow ^4\text{T}_{2g}(\text{F})$ , transitions respectively. These bands are similar to the complex of octahedral geometry. The magnetic moment was found to be  $4.25\text{ B.M.}$  due to 3 unpaired

electrons. The absorption spectra of Co(II) complex(**2**) showed five peaks at 274nm, 333nm, 372nm, 420nm and 472nm, assigned to  $\pi-\pi^*$ ,  $n-\pi^*$ , MLCT and  ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$  and  ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$  transition respectively. These bands are also similar to six coordinated complexes which are compatible with octahedral geometry. The magnetic moment was found to be 5.34 B.M. at room temperature. The Zn(II) complex(**3**) exhibits four absorption peaks at 267nm, 332nm, 415nm and 466 nm respectively, which are assigned to  $\pi-\pi^*$ ,  $n-\pi^*$ , MLCT transition respectively and suggesting a distorted square pyramidal geometry. The Cd(II)complex(**4**) exhibit four absorption peaks at 267 nm, 319 nm, 369 nm and 447nm respectively, which is assigned to  $\pi-\pi^*$ ,  $n-\pi^*$ ,  $n-\pi^*$ , and MLCT transitions respectively, which are similar with distorted square pyramidal geometry. Complexes**3** and **4** are diamagnetic.<sup>2,4,33</sup>

### NMR Spectra

The  ${}^1H$  NMR spectra of the ligand(L) and complexes **3** and **4** were recorded in DMSO- $d_6$  solvent using TMS as standard. The NMR spectra also supported the formation of complexes. The signals at  $\delta$  8.22 – 8.41(dd) were consistent with azomethine protons for ligand(L). The medium intensity peak at  $\delta$  2.34(d) was observed for ligand, assigned to methyl protons. The NMR spectra of ligand showed two sharp signals at  $\delta$ 15.14 (s) and  $\delta$ 15.36 (s) were assigned to the phenolic -OH protons, these peaks were absent in complex (**3**) which represents the deprotonation and complexation with Zn(II) metal ion.<sup>26, 27</sup> Complex**3** and **4** exhibits signals at  $\delta$ 8.45(dd),  $\delta$ 8.25(m) respectively, which is assigned to HC=N protons. Signals in the region  $\delta$ 7.17-7.57 (m) were due to aromatic protons for ligand, which were shifted to  $\delta$ 7.22-7.77 and  $\delta$ 7.35 in the complexes**3** and **4** respectively. The proton decoupled  ${}^{13}C$ NMR spectra of the ligand showed a signal at 21.23 ppm, 133.23-139.93 ppm, 168.63 – 169.48 ppm due to  $-CH_3$  carbon, aromatic ring of Schiff base and characteristic azomethine carbon atom respectively. The metal complexes **3** and **4** exhibit signals at 21.48ppm and 21.24ppm respectively due to  $-CH_3$  carbon atoms. The azomethine carbon peak observed at 156.30 ppm for complex **3**.

### Mass Spectra

The molecular ion peak of ligand and its complexes (**1-4**), was observed at  $m/z$  430.50 $[M]^+$ , 558 $[M+2]^+$ , 566 $[M+3]^+$ , 512.49 $[M+2]^+$ , 559.06 $[M]^+$  respectively, which corresponds to their molecular formula and formula weight. Also of molecular ion peak, the ligand and its complexes (**1** to **4**) exhibited some additional peaks due to fragmentation, which arise from the thermal cleavage of the complexes. All these results suggest the stability of fragments and are good agreement with the proposed formulae of the ligand and its metal complexes.

### Antibacterial Activity

The bacterial strains were obtained from Lala Lajpat Rai Memorial Medical College, Meerut. The tetradentate ligand and its metal complexes(**1**to **4**) were studied for their *in-vitro* antibacterial activity against *E. coli*, *B. megaterium* and *P. aeruginosa* bacterial strains by agar diffusion method. In this method, a solution of ligand and metal complexes were spread over agar plates seeded with tested bacterial strains. The agar plates were used for incubation at 35°C for 2-3 days. The antibiotic gentamicin was used as a standard drug against bacterial strains. The bacteriacidal activity was resolved by calculating the diameter of the inhibition zone.<sup>19, 33</sup> These results showed that complexes (**1-4**) showed significant activity as compared to the ligand.

### CONCLUSION

The ligand (L) was synthesized and further used for the preparation of Cr(III), Co(II), Zn(II), and Cd(II) complexes. The spectral data indicate that the metal complexes (**1-4**) were hexa and pentacoordinated with tetradentate ligand and hydroxyl group. Thus exhibit octahedral and distorted square pyramidal geometry respectively. All the complexes(**1-4**) were tested against the bacteria *E. coli*, *B. megaterium* and *P. aeruginosa* were investigated. The Co (II) complex showed higher activity against these bacteria.

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