

BIOLOGICAL STUDIES OF SOME NOVEL TRANSITION METAL COMPLEXES WITH SCHIFF BASE

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ABSTRACT

Schiff bases and their coordination complexes have acquired great significance in the field of inorganic research mainly because of their biological activity. Antibacterial, antifungal and antitumour activities are reported (Xinde Zhu, 2002) for these compounds. In the present study, the Schiff base derived from 2-hydroxybenzaldehyde and 2-aminophenol are used for preparing complexes of selected transition metal salts. The complexes were prepared by mixing equimolar quantities of the Schiff base and the transition metal salts, $MX_n \cdot nH_2O$ where $M = Co, Ni, Cu$ and Zn , $X = -NO_3, -ClO_4$ and $-Cl$. Characterizations of the complexes were done by metal analysis, elemental analysis, conductivity measurements in non aqueous solvents, IR and Electronic spectral analysis. Biological activity was checked against the bacteria *Escherichia Coli* and *Bacillus subtilis* by disc diffusion method. The Schiff base and the complexes were found to be active against both the bacteria. But some complexes showed more activity than the Schiff base indicating the effect of metal coordination in inhibiting bacterial growth.

Key words: Schiff base, Ligand, 2-hydroxybenzilidene-2-aminophenol, Antibacterial activity studies.

1. INTRODUCTION

Schiff bases have been extensively used in the study of structure and bonding in coordination complexes. Schiff base of 2-hydroxybenzaldehyde with different aromatic amines have been studied in particular for the past few years (Hankare and Chawan, 2003), due to the exclusive biological activity shown by their complexes. The present study concentrates on the Schiff base derived from 2-hydroxybenzaldehyde and 2-aminophenol and a few complexes with transition metals. All the compounds synthesized were characterized and tested for their antibacterial activities against *E. Coli* and *B. Subtilis*.

2. MATERIALS AND METHODS

All the chemicals used were of A.R. grade. 2-hydroxybenzaldehyde is used after purification by distillation. The transition metal salts were prepared from the respective carbonates by reacting with 60% corresponding mineral acid and then crystallizing by evaporation over a steam bath.

Synthesis of Schiff base: 2-hydroxybenzilidene 2-aminophenol

2-aminophenol (0.109g, 1mmol) in 20ml ethanol is mixed with 2-hydroxybenzaldehyde (0.122g, 1mmol) in 20ml ethanol. The mixture was refluxed for 4 hours on a boiling water bath, cooled and kept

overnight. Orange red needles were formed, which was filtered, washed with 50% ethanol and dried in a desiccator over anhydrous $CaCl_2$ and recrystallised from ethanol.

Synthesis of complexes

To the refluxing solution of the Schiff base, 2-hydroxybenzilidene 2-aminophenol (0.11g, 0.5 mm) in 30ml ethanol, 0.5mM solution of the transition metal salt $MX_n \cdot nH_2O$ where $M = Co(II), Ni(II), Cu(II), Zn(II)$ and $X = -ClO_4, -NO_3, -Cl$, added drop by drop and then refluxed the mixture for 2 hours, concentrated and kept overnight. The precipitate formed is filtered, washed with ethanol and ether. Dried in a vacuum desiccator over anhydrous $CaCl_2$ and recrystallised from ethanol.

Analytical methods

The melting points were determined on a Thoshniwal melting point apparatus. The Schiff base and the complexes were analyzed for carbon, hydrogen and nitrogen content on a Heracus CHN rapid analyzer. The metals were determined gravimetrically as oxides. Molar conductance in DMF, ethanol and nitrobenzene in $10^{-3}M$ solutions were measured at room temperature (Singh, 2003) using an ELICO CM-180 conductivity meter with a dip type cell of platinum electrodes (Cell constant = 0.986). The chloride content was determined by Volhard method and perchlorate analysis done by first reducing to the chloride and then by Volhard method (Indrasenan and Jayasankar, 1990). The IR spectrum

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of the complexes were recorded in the range 400- 4000 cm^{-1} on a Shimadzu IR 470 spectrophotometer in KBr discs. The electronic spectra in ethanol solution (10^{-3}M) were recorded in the range 200-900 nm on a Shimadzu UV- 160 A spectrometer.

Antibacterial activity

Antibacterial studies were conducted against *E. Coli* and *B. Subtills* by disc diffusion method (Powar, 1986 ; Mukherjee, 1983). Nutrient Agar was used as the cultural medium. Agar medium was prepared in distilled water and poured into sterilized Petri dishes. Inoculation was done with the help of a platinum wire loop. The ligand /complex (0.003g) was dissolved in 1ml DMSO. Blotting paper discs (3mm) diameter were dipped in the solution. Dried in an incubator and then applied on the bacteria grown in agar plates. The plates were inverted and kept in an incubator set at 37°C for 24 hours. The zone of inhibition around the disc was measured in mm. The percentage of inhibition was calculated (Raman, 2006 ; Izmail, 2002).

3.RESULTS AND DISCUSSION

The elemental analysis data and some physical properties of the ligand and its complexes are given in Table:1. The ligand is soluble in most common organic solvents such as ethanol, acetone, dichlorobenzene, acetonitrile, Hexane, DMF and DMSO. The complexes were insoluble in water, but soluble in some organic solvents like ethanol, DMF and DMSO. The molar conductance data of the complexes is given in Table:2.

IR Spectra

The important IR frequencies of the ligand and its complexes along with their assignments are reported in Table:3. The following observations are concluded from the comparison of the IR spectra of ligand and metal complexes.

The IR spectrum of the ligand is almost identical to those of the metal complexes in the region $670 - 1400\text{cm}^{-1}$. (El shazely, 1990). All the ligands showed the absence of bands at about 1735cm^{-1} and 3420cm^{-1} corresponding to the free carbonyl and amine stretching. Instead a sharp band obtained at 1631cm^{-1} corresponding to $\text{CH}=\text{N}$ stretching. The absorption band at about 3000cm^{-1} in the spectra of the free ligand is due to the stretching vibration of strong intramolecularly H- bonded O – OH which is lost in some cases due to complexation. (Semanthi, 2005). Similarly, the sharp band near 1300cm^{-1} is attributed to

the bending vibrations of –OH group. The down shift of azomethine stretching frequency in all the complexes provided evidence in support of the involvement of azomethine nitrogen in coordination to the metal atom (Muraleedharan Nair and Radhakrishnan, 1995). Similarly, a weak band in the far IR region near 600cm^{-1} attributed to $\nu(M-N)$ was observed for all the complexes (Rao, 2002), which was not found in the spectra of ligands. The broad band corresponding to –OH stretching shifted downfield in complexes suggesting coordination through oxygen. In zinc perchlorate and copper perchlorate complexes, the OH band disappeared suggesting coordination through oxygen after deprotonation and the absence of coordinated water molecules, thus ML_2 structure of the complexes (Min Zao, 2002). In all other cases, the IR data support ML structure of complexes (Manimekalai and Mahendran, 2003). In cobalt perchlorate complex, two distinguishable peaks around 1100cm^{-1} is obtained characteristic of the coordinated perchlorate stretching vibrations (Muraleedharan Nair and Radhakrishnan, 1995). The IR spectrum of $[\text{CuL}_2]$ is given in Figure 1.2.

Electronic spectra

The electronic spectra of the complexes were recorded in the solution state. The spectrum of all Co(II) complexes exhibited three bands (Figure 1.1) which may be assigned as ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$ ($\gamma=9500\text{cm}^{-1}$), ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$ ($\gamma=18000\text{cm}^{-1}$) and ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$ ($\gamma=19000\text{cm}^{-1}$) (Maurya, 2002). The Ni(II) complexes exhibit crystal field transitions at 10550cm^{-1} , 17391cm^{-1} and 25000cm^{-1} , which can be the allowed transitions ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$ (γ_1), ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$ (γ_2) and ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$ (γ_3) (Kazunori Sakata., 1990; Huheey., 2000). The third spin allowed transition is observed as a shoulder. The Cu(II) complexes displayed a broad band centred at 15000cm^{-1} which is an envelope of the transitions. Because in Cu(II) complex, the Jahn Teller effect removes the degeneracy of the d – orbitals resulting tetragonal distortion and splitting of E_g and T_{2g} levels into B_{1g} , A_{1g} and E_{2g} levels (Maurya, 2003). Thus the observed spectral properties of Cu(II) complexes are due to the weakly distorted octahedral geometry of the complex.

Antibacterial studies

The results of biological studies were reproduced in Table 4. The ligand and the metal complexes showed variable activity against the bacterial stains of *E. Coli* and *B. Subtilis* (Patel, 2003). The Schiff base 2-hydroxybenzilidene-2-aminophenol showed moderate activity against the bacteria. But comparing to the ligand, almost all metal complexes exhibited enhanced activity (Thangadurai, 2002). Especially the complexes of the type $[M(H_2L)(H_2O)_3]X_2$ showed the maximum inhibition and among the complexes formed from different metals, copper(II) complexes were found to be most effective in inhibiting the bacterial growth.

4. Conclusion

The analytical results showed that the ligand 2-hydroxy benzilidine-2-aminophenol formed both ML and ML_2 type complexes and the anions from the metal salts played a prominent role in directing the complex formation towards ML or ML_2 type. Ni(II), Cu(II) and Zn(II) perchlorate salts directed the formation of ML_2 complexes, while the chloride salts preferred ML type. All the nitrate salts and Co(II) salts stabilized MLX_2 type complexes where $X = -NO_3, -ClO_4, -Cl$. The structure of the Schiff base and the complexes are given in Figure 1.3 to Figure 1.7.

The molar conductance data provided valuable support for the above inference. The cobalt perchlorate complex exhibited anomaly forming MLX_2 type complex with the anion coordinating to the metal inside the coordination sphere which is firmly supported by the IR data. The biological studies provided valuable information regarding the variation in antimicrobial activity of a Schiff base when coordinated to different transition metals.

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Table 1: ANALYTICAL DATA OF THE LIGAND AND THE COMPLEXES

Empirical formula/Molecular formula	Mol. Wt	Colour	MP °C	Yield	% found / calculated			Metal %
					C	H	N	
$C_{11}H_{11}NO_2$ H_2L	213	Orange red	170°C	80%	60.23 (61.97)	5.98 (5.16)	6.13 (6.57)	--
$C_{11}H_{13}NO_{11}CoCl_2$ $[Co(H_2L)(H_2O)(ClO_4)_2]$	489	Reddish brown	220°C	76%	25.12 (25.53)	3.35 (2.7)	2.65 (2.7)	12.35 (12.26)
$C_{11}H_{17}NO_5CoCl_2$ $[Co(H_2L)(H_2O)_3]Cl_2$	395.9	Reddish brown	>300°C	76%	33.75 (33.34)	4.92 (4.29)	3.65 (3.53)	14.7 (14.65)
$C_{11}H_{17}N_3O_{11}Co$ $[Co(H_2L)(H_2O)_3](NO_3)_2$	449.9	Brown	>300	62%	29.51 (29.4)	3.79 (3.4)	10.1 (9.3)	11.16 (12.31)
$C_{11}H_{15}NO_5Ni$ $[NiL(H_2O)_3]$	323.7	Yellow	251°C	71%	41.32 (40.77)	5.14 (4.63)	4.89 (4.832)	17.12 (17.91)
$C_{22}H_{20}N_2O_4Ni$ $[NiL_2]$	481.2	Yellowish brown	275°C	73%	55.25 (54.86)	4.72 (4.15)	5.25 (5.82)	12.89 (12.06)
$C_{11}H_{17}N_3O_{11}Ni$ $[Ni(H_2L)(H_2O)_3](NO_3)_2$	449.7	Yellowish Brown	280°C	65%	30.12 (29.4)	3.1 (3.9)	9.82 (9.1)	15.35 (14.12)
$C_{11}H_{15}NO_5Cu$ $[CuL(H_2O)_3]$	328.54	Dark brown	210°C	85%	40.85 (40.17)	4.98 (4.56)	4.26 (4.26)	20.17 (19.33)
$C_{22}H_{20}N_2O_4Cu$ $[CuL_2]$	487.54	Dark brown	>300°C	85%	54.46 (54.15)	4.82 (4.1)	5.93 (5.74)	13.75 (13.02)
$C_{11}H_{17}N_3O_{11}Cu$ $[Cu(H_2L)(H_2O)_3](NO_3)_2$	454.5	Black	>300°C	67%	29.11 (28.65)	5.03 (4.1)	9.82 (9.31)	16.22 (15.71)
$C_{22}H_{20}NO_5Zn$ $[ZnL(H_2O)_3]$	330.4	Yellow	220°C	80%	40.26 (39.95)	4.87 (4.54)	4.75 (4.23)	20.18 (19.67)
$C_{22}H_{20}N_3O_{11}Zn$ $[ZnL_2]$	489.4	Lemon Yellow	240°C	80%	54.37 (53.94)	4.65 (4.09)	5.89 (5.7)	13.26 (12.97)
$C_{11}H_{17}N_3O_{11}Zn$ $[Zn(H_2L)(H_2O)_3](NO_3)_2$	456.4	Yellow	>300°C	65%	30.12 (29.27)	4.89 (4.3)	9.71 (9.22)	17.45 (16.92)

Table : 2 CONDUCTANCE DATA

Empirical formula/ Molecular formula	λ_m Scm^2mol^{-1}	
	DMF	Acetonitrile
$C_{11}H_{11}NO_2$ H_2L	Insoluble	Insoluble
$C_{11}H_{13}NO_{11}CoCl_2$ $[Co(H_2L)(H_2O)(ClO_4)_2]$	Insoluble	Insoluble
$C_{11}H_{17}NO_5CoCl_2$ $[Co(H_2L)(H_2O)_3]Cl_2$	173.4	286.1
$C_{11}H_{17}N_3O_{11}Co$ $[Co(H_2L)(H_2O)_3](NO_3)_2$	146.5	232.9
$C_{11}H_{15}NO_5Ni$ $[NiL(H_2O)_3]$	Insoluble	Insoluble
$C_{22}H_{20}N_2O_4Ni$ $[NiL_2]$	Insoluble	Insoluble
$C_{11}H_{17}N_3O_{11}Ni$ $[Ni(H_2L)(H_2O)_3](NO_3)_2$	119.2	215.2
$C_{11}H_{15}NO_5Cu$ $[CuL(H_2O)_3]$	Insoluble	Insoluble
$C_{22}H_{20}N_2O_4Cu$ $[CuL_2]$	Insoluble	Insoluble
$C_{11}H_{17}N_3O_{11}Cu$ $[Cu(H_2L)(H_2O)_3](NO_3)_2$	163.9	225.3
$C_{22}H_{20}NO_5Zn$ $[ZnL(H_2O)_3]$	Insoluble	Insoluble
$C_{22}H_{20}N_3O_{11}Zn$ $[ZnL_2]$	Insoluble	Insoluble
$C_{11}H_{17}N_3O_{11}Zn$ $[Zn(H_2L)(H_2O)_3](NO_3)_2$	175.2	308.2

Table : 3 IR SPECTRAL DATA

γ (i NO ₃)	γ (ClO ₄)	γ (M-O)	γ (M-N)	γ (C-O)	γ (C=C)	γ (CH=N)	γ (Ar C-H)	γ (O-H)	
				1307m	1485s 1461s	1631s	3047m	3452m, 1527s	H ₂ L
		462w	624w	1292 (ds15)	1473s (ds12) 1442s	1612s (ds19)	3058w us11	3448m, (ds4), 1542w, us15	[Co(H ₂ L) (H ₂ O) ₃] Cl ₂
	1153m 1107s	467w	624w	1288s (ds19)	1473s, (ds12) 1442s, (ds19)	1612s (ds19)	3062w (us15)	3448m, (ds 4) 1542w, (us15)	[Co(H ₂ L) (H ₂ O) (ClO ₄) ₂]
1382.,8s		460w	538w	1263s (ds44)	1467s, (ds18) 1438s, (ds23)	1602s (ds29)	3020w (ds27)	3352m, (ds100) 1583w, (us56)	[Co(H ₂ L) (H ₂ O) ₃] (NO ₃) ₂
		420w	617m	1296s (ds11)	1465s (ds20)	1620s (ds11)	3058w (us11)	3402m, (ds50) 1546m, (us 19)	[NiL (H ₂ O) ₃]
		412w	617m	1296s (ds11)	1460s (ds20)	1620s (ds11)	3058w (us11)	1531m (us4)	[NiL ₂]
1382.8s		486w	560w	1296s (ds11)	1467s (ds18)	1618s (ds13)	3055w (us8)	3452m	[Ni(H ₂ L) (H ₂ O) ₃] (NO ₃) ₂
		480w	600w	1296s (ds11)	1469s, (ds116) 1438s, (ds14)	1612s (ds19)	3055w (us8)	3402m, (ds50) 1531m, (us4)	[CuL (H ₂ O) ₃]
		486w	605w	1296s (ds11)	1465s, (ds20) 1433s, (ds28)	1612s (ds19)	3055w (us8)	- 1531m, (us4)	[CuL ₂]
1382.8s		460w	538w	1298s (ds9)	1469s, (ds16) 1440s, (ds21)	1612s (ds190)	3055w (us8)	3336m, (ds116) 1583m, (us56)	[Cu(H ₂ L) (H ₂ O) ₃] (NO ₃) ₂
		443w	609m	1296s (ds11)	1469s, (ds16) 1442s, (ds19)	1612s (ds19)	3058w (us11)	3402m, (ds50) 1542w, (us15)	[ZnL (H ₂ O) ₃]
		443w	609m	1299s (ds8)	1460s, (ds25) 1442s, (ds19)	1612s (ds19)	3055w (us8)	- 1531m, (us4)	[ZnL ₂]
		450w	609m	1298s (ds7)	1469s, (ds16) 1442s, (ds19)	1612s (ds19)	3055w (us8)	3479m, (us27) 1585s, (us58)	[Zn(H ₂ L) (H ₂ O) ₃] (NO ₃) ₂

Table: 4 ANTIBACTERIAL ACTIVITIES

Compound	E.Coli		B.Subtills	
	Activity	Percentage of inhibition %	Activity	Percentage of inhibition %
DMSO	-	0	-	0
H ₂ L	++	12	+	9
[Co(H ₂ L)(H ₂ O) ₃](ClO ₄) ₂	+++	24	++	14
[Co(H ₂ L)(H ₂ O) ₃]Cl ₂	+++	21	++	17
[Co(H ₂ L)(H ₂ O) ₃](NO ₃) ₂	+++	24	+++	23
[NiL(H ₂ O) ₃]	+++	16	+	8
[NiL ₂]	++	13	+	12
[Ni(H ₂ L)(H ₂ O) ₃](NO ₃) ₂	+++	19	+++	18
[CuL(H ₂ O) ₃]	+	10	-	0
[CuL ₂]	++	12	+	10
[Cu(H ₂ L)(H ₂ O) ₃](NO ₃) ₂	+++	24	+++	25
[ZnL (H ₂ O) ₃]	-	0	+	8
[Zn L ₂]	++	14	+++	21
[Zn(H ₂ L)(H ₂ O) ₃](NO ₃) ₂	+++	22	+++	18

Percentage of Inhibition: Below 5mm = (-)
 5mm-10mm = (+)
 10mm-15mm = (++)
 5mm-20mm = (+++)

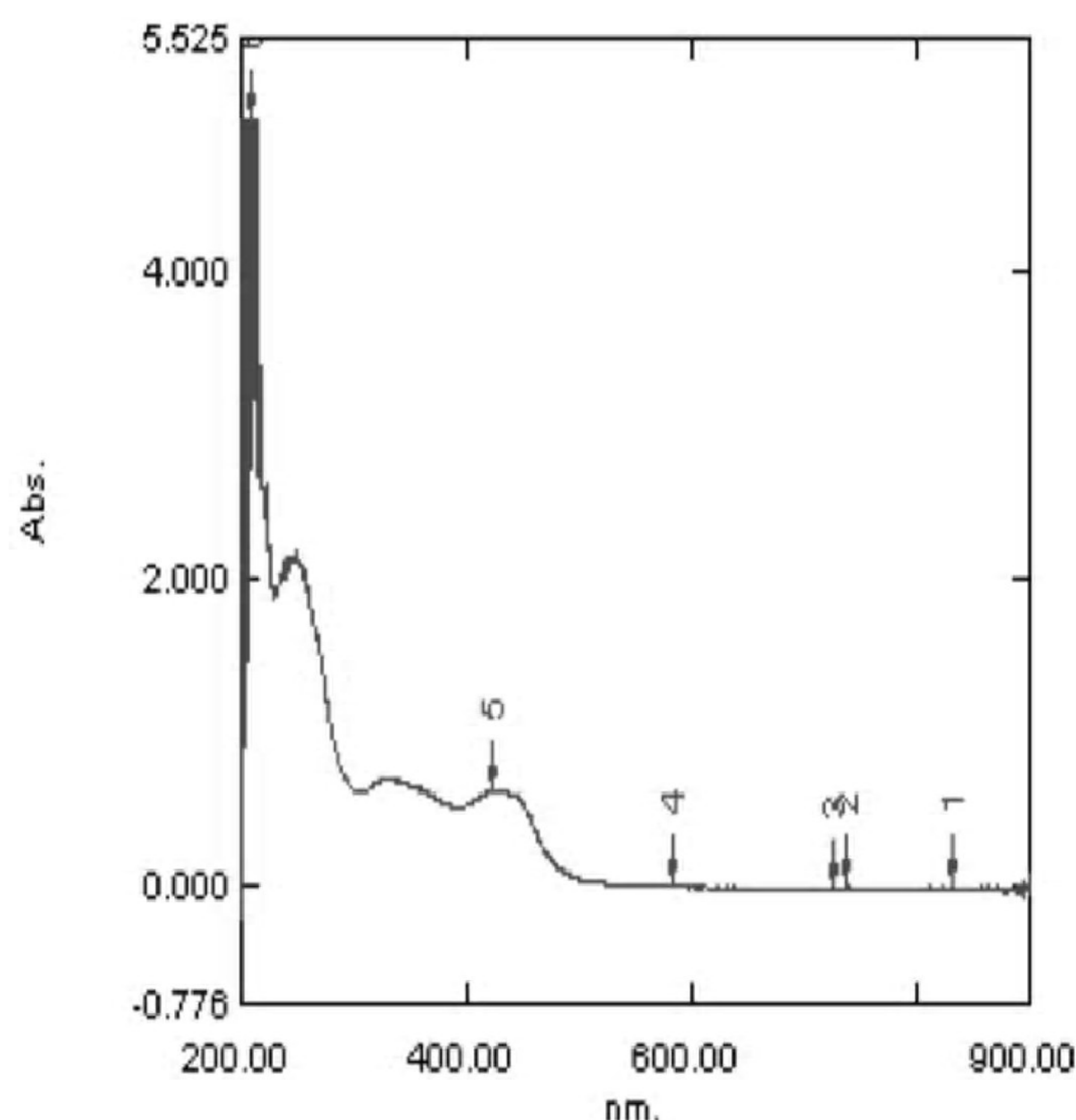
Figure 1.1 Spectrum of [Co(H₂L)(H₂O)₃]Cl₂

Figure 1.2 IR Spectrum of $[\text{CuL}_2]$

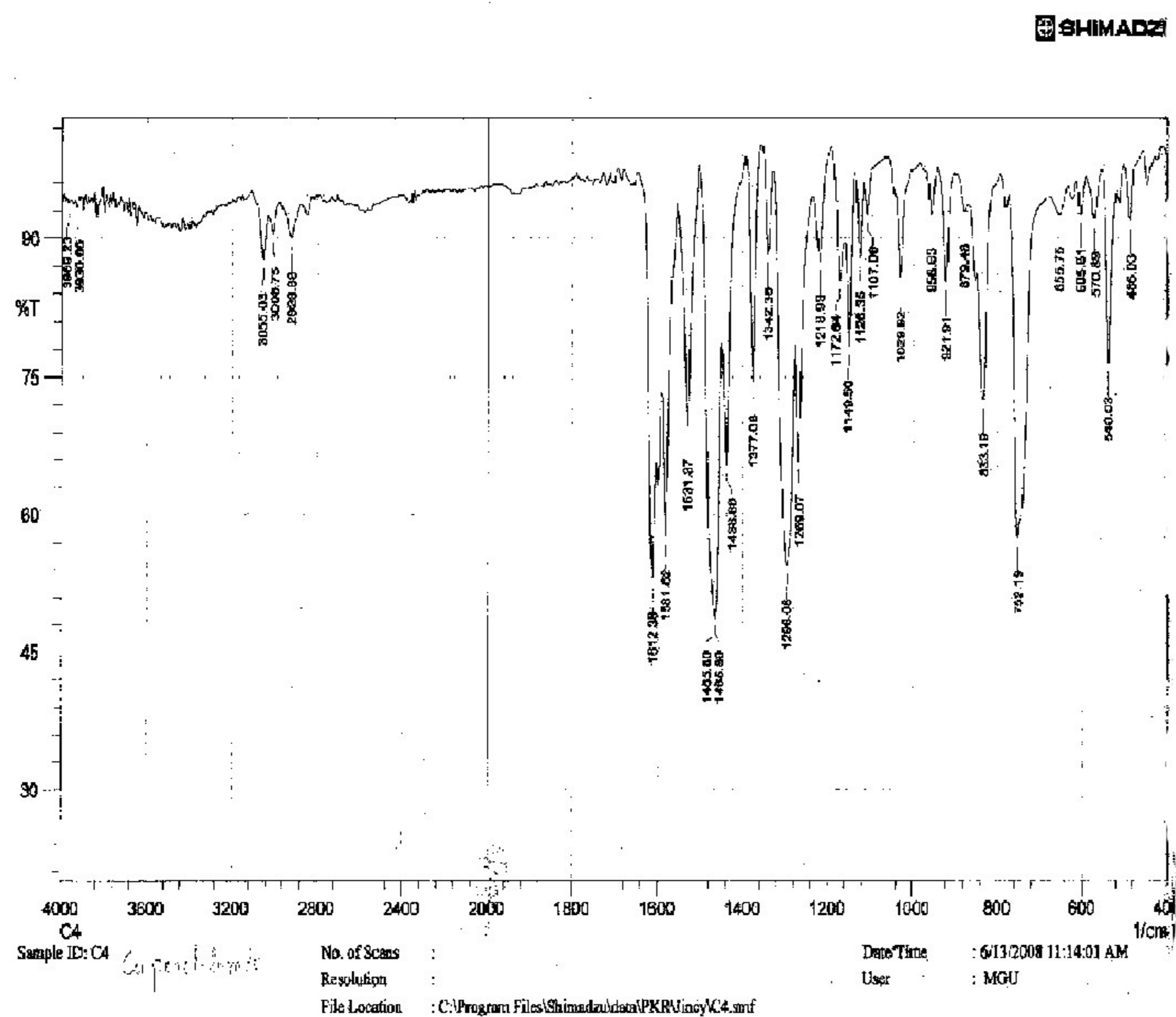


Figure 1.3 Structure of 2-hydroxybenzylidene-2-aminophenol

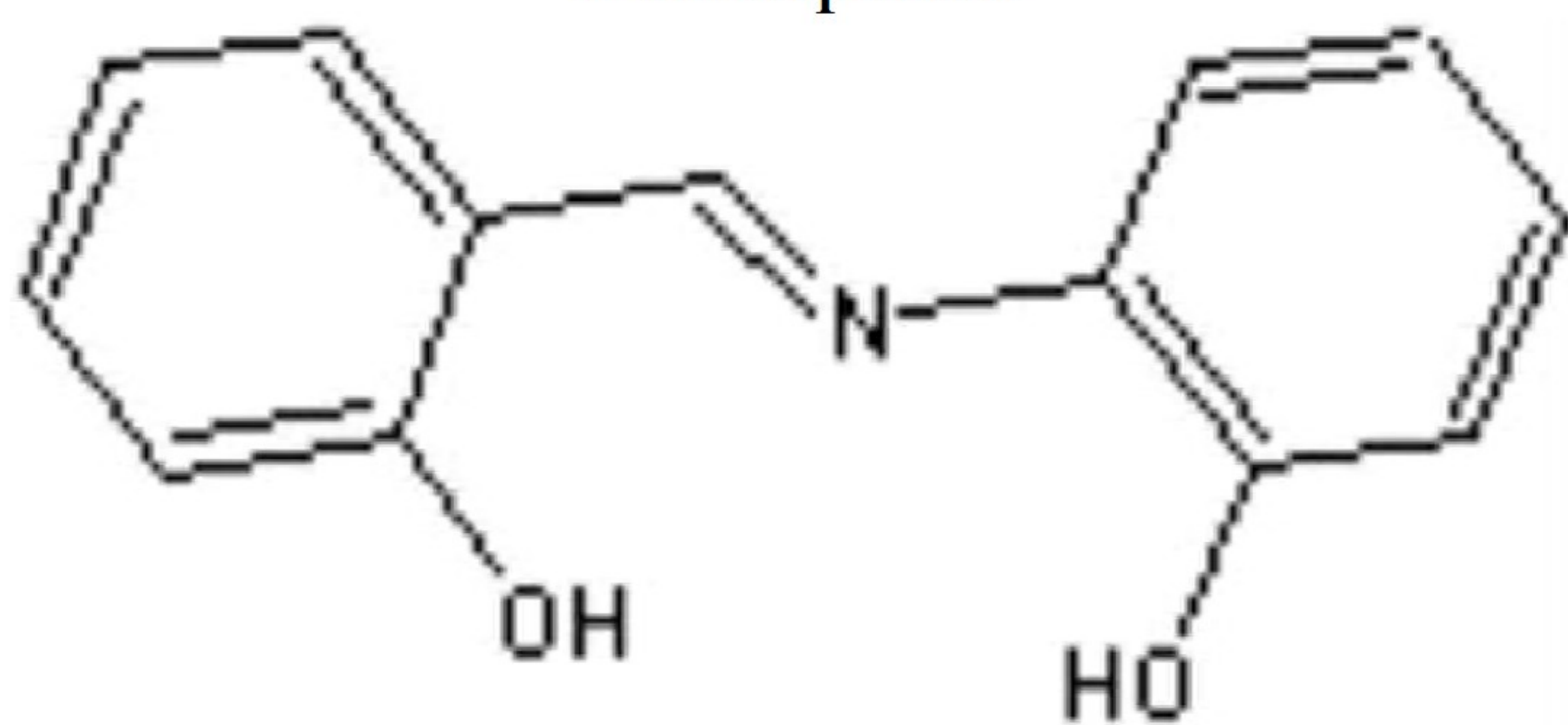


Figure 1.4 Structure of $[\text{ML}(\text{H}_2\text{O})_3]$ where M=Ni, Cu and Zn

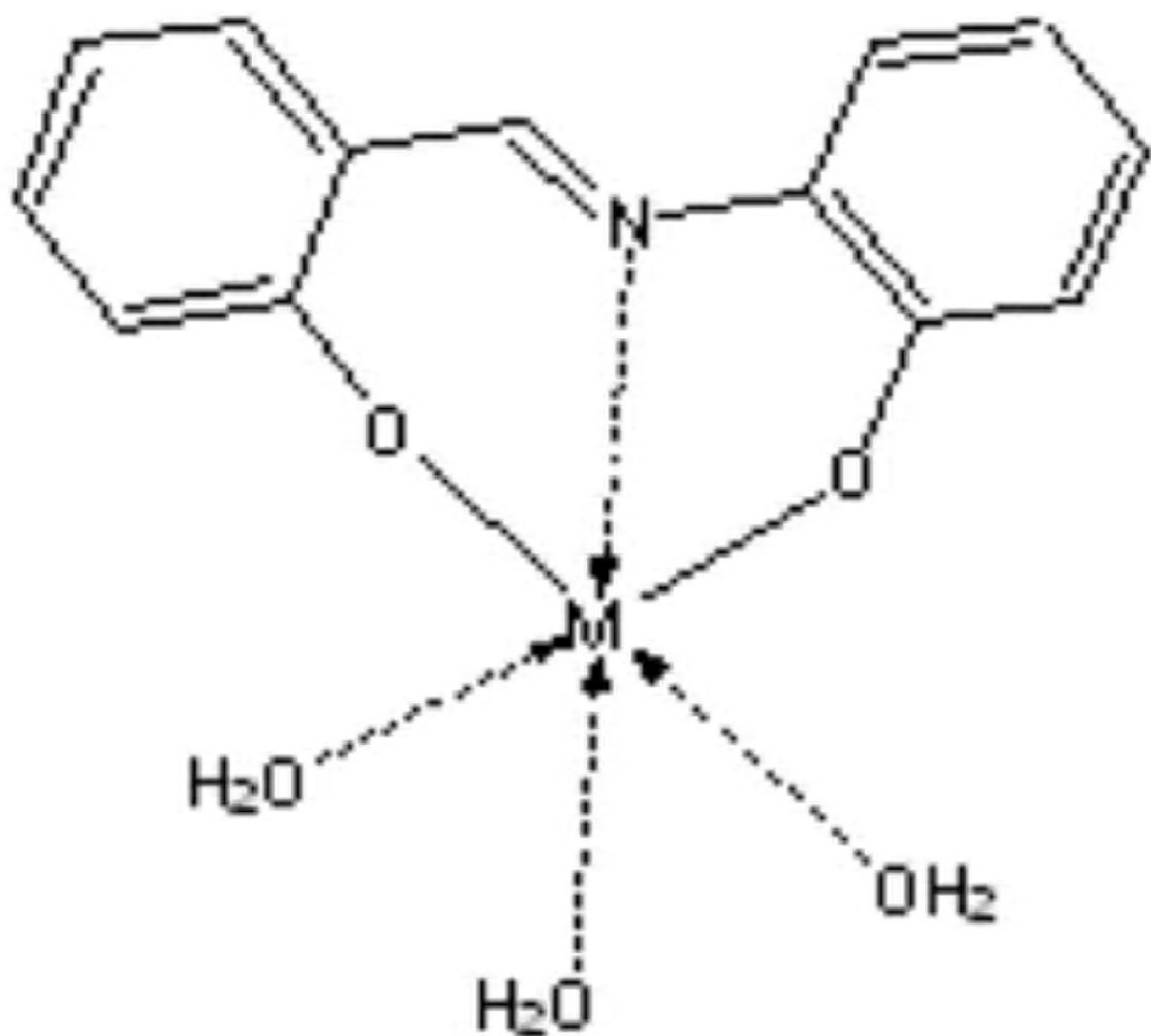


Figure 1.5 Structure of $[\text{ML}_2]$ where M= Ni, Cu and Zn

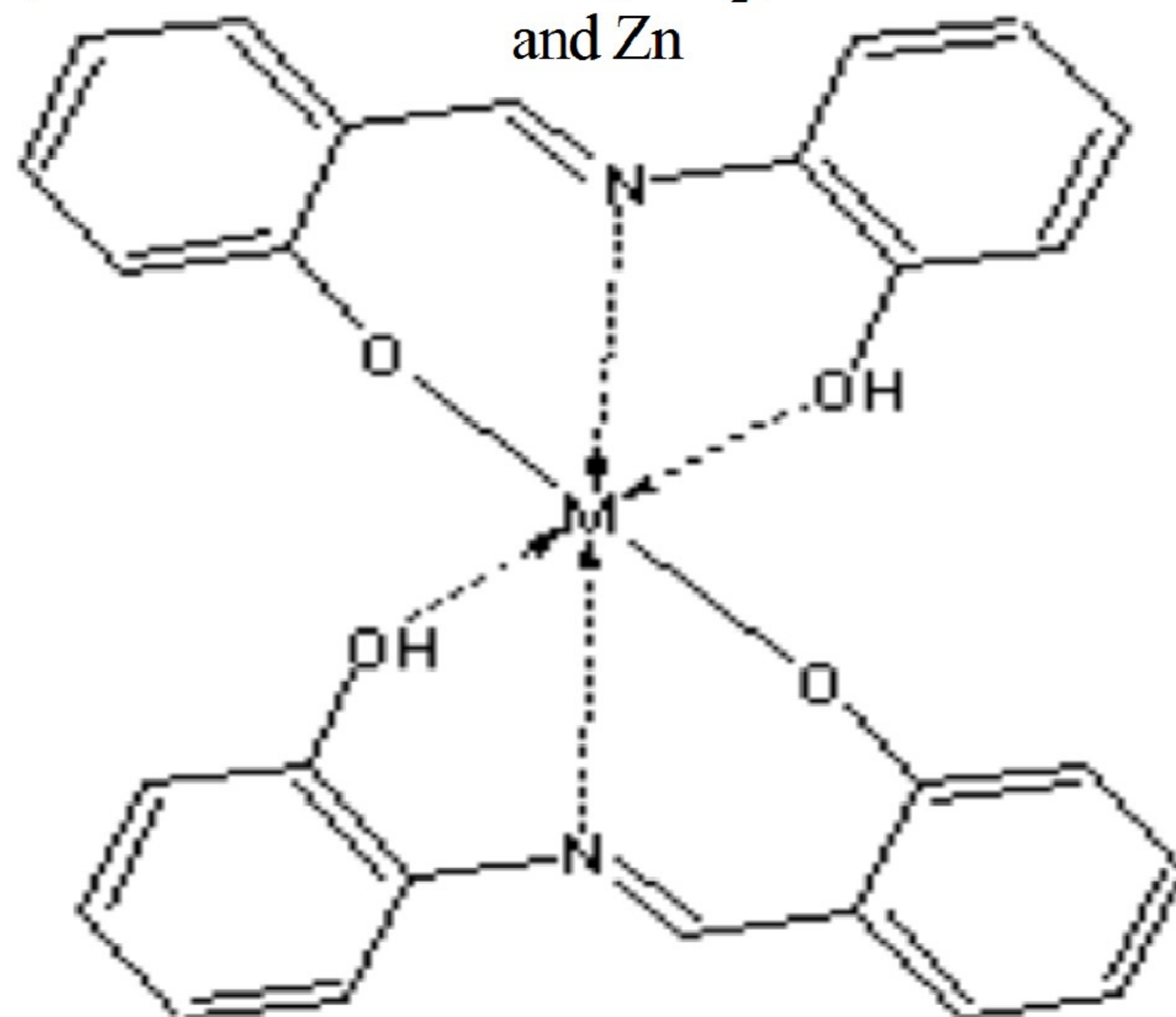


Figure 1.6 Structure of $[\text{M}(\text{H}_2\text{L})(\text{H}_2\text{O})_3]\text{X}_2$ where M= Co, Ni, Cu and Zn ; X= $-\text{NO}_3$ and $-\text{Cl}$

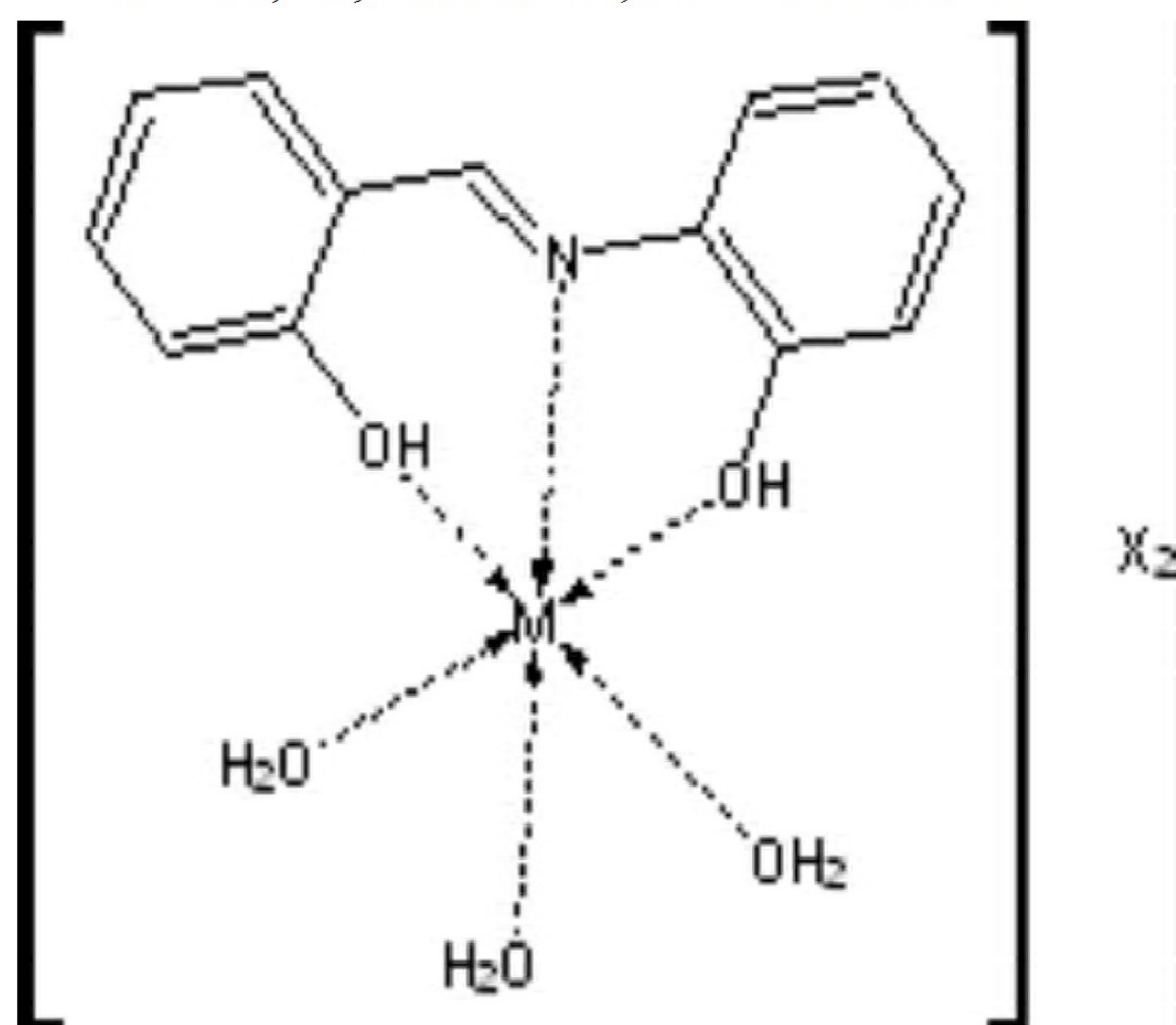
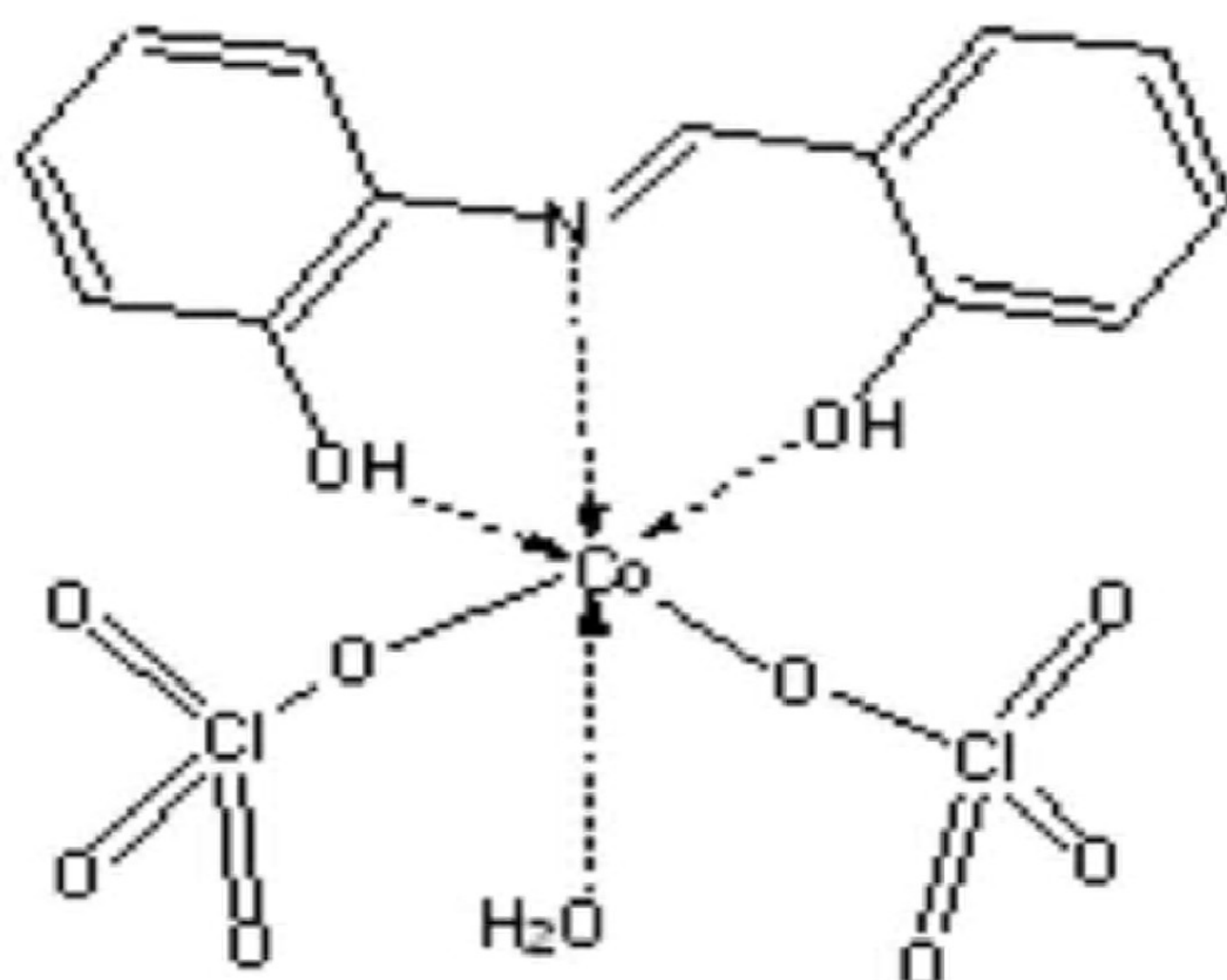


Figure 1.7 Structure of $[\text{Co}(\text{H}_2\text{L})(\text{H}_2\text{O})(\text{ClO}_4)_2]$



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