



# AMERICAN JOURNAL OF PHARMTECH RESEARCH

Journal home page: <http://www.ajptr.com/>

## Synthesis of Nickel (II) Schiff Base Molecular Adducts: Spectroscopic Characterization & Antioxidant activity

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### ABSTRACT

Transition metal complexes of Ni (II) with schiff base ligand (HL) derived from condensation of 3-Bromo benzaldehyde and hydrazine monohydrate were successfully synthesized, characterized and isolated. The schiff base ligand and its Ni(II) complexes prepared were characterized by melting point /decomposition temperature, solubility, conductivity/ Molar conductance, UV-visible, FT-IR, XRD, TGA and elemental analysis results. In the UV-Vis study, a bathochromic shift of approximately 60 nm indicating the formation of coordinated Ni(II) complexes by more than one coordinating sites. IR spectra of the free ligand showed a band at  $1660\text{cm}^{-1}$  which is assigned to the (-C=N-) stretching vibration of the azomethine functional group. These bands were observed at lower frequencies in the spectra of Ni(II) complexes which indicates complexation process between schiff base ligand and  $\text{Ni}^{2+}$  ion. Low conductance ( $7\text{-}9\text{ ohm}^{-1}\text{ cm}^2\text{ Mol}^{-1}$ ) measurement indicated that the Ni (II) complexes are non electrolytic in nature whereas elemental analysis result revealed 1:1 Nickel-schiff base ratio. TGA analysis showed the presence of two coordinated water molecules on the basis of physico chemical measurements the following empirical formulas have been assigned to coordinated Ni(II) complex:  $[\text{Ni}(\text{HL})_2] \cdot 2\text{Cl} \cdot 2\text{H}_2\text{O}$ ,  $[\text{Ni}(\text{HL})_2] \cdot \text{SO}_4 \cdot 2\text{H}_2\text{O}$ . The antioxidant activities of schiff base ligand and Ni(II) complexes were evaluated by using DPPH reagent i.e., 2, 2'-diphenyl-1-picrylhydrazyl free radical assay which showed that coordinated Ni(II) complexes have a higher antioxidant activities than that of Schiff base ligand. All compounds have been evaluated for invitro anti microbial activities against isolated bacterial strains of E. coli (MTCC-1687), E. faecalis (MTCC-439), S. aureus (MTCC-737) and MR S. aureus (Indigenous). All compounds showed mild to moderate antibacterial activities. The minimum inhibitory concentration values ranged from  $50\text{ }\mu\text{g/ml}$  to  $3.125\text{ }\mu\text{g/ml}$ . All compounds displayed invitro anti bacterial activity against both gram positive and gram negative bacterial strains. It may be proved that the antibacterial activity of compounds is related to the cell wall structure of the tested bacterial stain. The synthesis of Ni(II) complexes via template method is a good method for obtaining very efficient bioactive agents as a good drug candidates for various biological applications in future for humans. As well as schiff base molecular adducts of Ni(II) complexes are also promising material in different cutting edge research areas like applications in preparation of solar cells, super capacitors, catalysts and electrode materials.

**Keywords:** Schiff base ligands, Ni(II) complexes, Spectroscopic analysis, antioxidant activities, Antimicrobial activities, DPPH, 3-Bromobenzaldehyde.

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Received 10 August 2022, Accepted 9 October 2022

Please cite this article as: Kothari R., Synthesis of Nickel (II) Schiff Base Molecular Adducts: Spectroscopic Characterization & Antioxidant activity. American Journal of PharmTech Research 2022.

## INTRODUCTION

Transition metal complexes facilitates synthesis of drugs is of extraordinary importance in medicinal chemistry 1-5. Proper selection of the central metal ions and well designed schiff base ligands are the most important criteria for high efficiency of compound. Schiff base compounds often contain many donor atoms (N, O, S) which makes them biologically active and excellent chelators of transition metal ions. Schiff bases and their metal complexes have a variety of applications in synthetic organic chemistry as an intermediates and as catalysts <sup>1-3</sup>. These compounds also plays a very important contributions to pharmaceuticals <sup>4,5</sup> and their other industrial applications include foods, dyes and polymers. Now-a-days the coordination behavior of first row transition metal ions has been continuously reported in medicine and diagnostics <sup>6</sup>. The substituted heterocyclic ring in coordination with transition metal ions create biologically active molecules which possess excellent physicochemical and pharmacological properties <sup>7-10</sup>. Due to severe cytotoxicity of platinum based drugs, researcher have paid more attention to first row transition metal ions like Cu, Co, Ni, Zn, etc. Nickel is recognized as an essential trace element because several enzymes like hydrogenases and carbon monoxide dehydrogenases <sup>11</sup> contain nickel(II) ions in the active site. Literature reveals that the nickel (II) complexes showed various pharmacological activations like antibacterial <sup>12-14</sup>, anti fungal <sup>15</sup> antiproliferative and anticancer properties <sup>16-18</sup>.

In this paper, Schiff base ligand has been prepared by condensation of 3- Bromoben zaldehyde with hydrazine monohydrate respectively. The corresponding Ni(II) complexes with the ligand have been synthesized and characterized by various spectroscopic techniques like UV-Vis, FT-IR, XRD & TGA analysis. The antioxidant properties of the ligand and its Ni (II) complexes have been determined in-vitro using DPPH Method. The antimicrobial activity of the ligand and its Ni(II) complexes have been determined against both gram positive, gram negative bacterial as well as fungal isolates ( *C. albicans*, *A. niger* and *A. fumigatus* )using disc diffusion method. We hope our work will lead to further evaluation of these compounds as chemotherapeutic agents for human.

## MATERIALS AND METHOD

All reagents and solvents were all of analytical grade and were utilized without any further purification. DPPH, Nickel salts and thiosemicarbazide hydrochloride were obtained from commercially available sources and used as received. Doubly distilled water was used in the whole process. All complexes including ligand and complexes were dissolved in DMSO as the stock solution (  $1 \times 10^{-3}$  mol . L<sup>-1</sup>), and diluted properly as the working solution.

UV-Vis absorption spectra were recorded on a Perkin Elmer UV/Vis Lambda 25 using a 1 cm path length cell with dichloromethane solvent. . The Fourier- transform infrared spectroscopy (FT-IR) spectra (KBr pellets) were recorded using a Perkin Elmer FT-IR spectrometer with the wavelength range from 400 to 4000  $\text{cm}^{-1}$ . Melting points of compounds were obtained by an electro thermal melting point apparatus and were not corrected. The elemental analysis was conducted using CHN Analyser, Thermo –Flash EA-1112 Series at the temperature upto 900o C and vanadium penta oxide ( $\text{V}_2\text{O}_5$ ) was used as an oxidizer to prevent inhibition caused by sulphur element. Thin Layer Chromatography (TLC) was performed using n-hexane/EtOAc (1:3) as an eluent. X-ray measurements for the ligands and their Cobalt(II) complexes were performed at room temperature using a Bruker axis D8 using  $\text{CuK } \alpha$  radiation.

The conductivity measurement were carried out in DMSO (10-3m) using Digisum electronic digital conductivity meter .0.01M KCl solution is used for calibration of conductivity meter.

### **Synthesis of ligand**

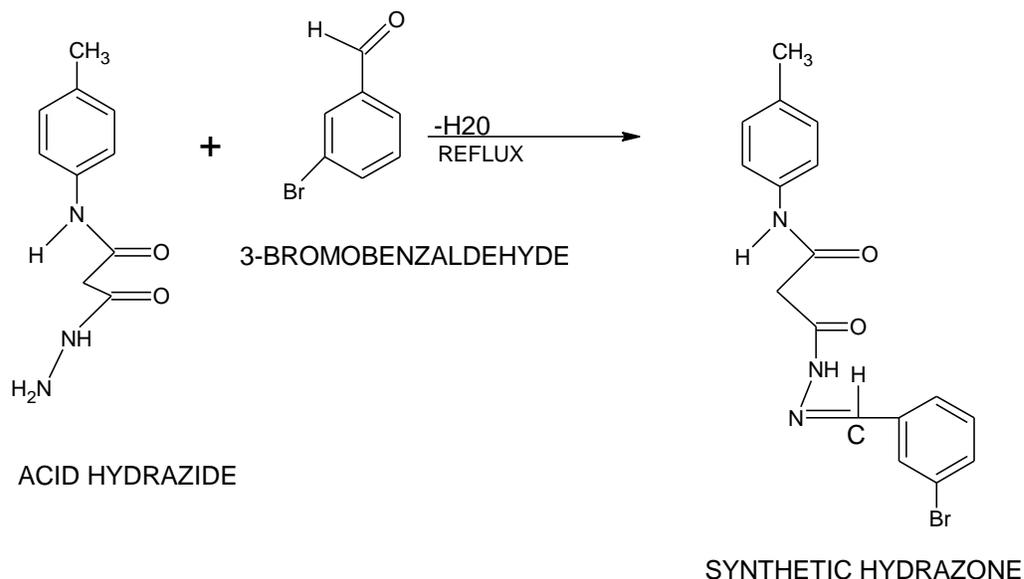
3-bromobenzaldehyde (4mmol)was added to a two necked round bottomed flask containing 20 ml ethanol, followed by addition of acid hydrazide. The mixture was stirred under reflux for 5-6 hours at 70 $^{\circ}$ c. Cool the contents at room temperature. A white precipitate was obtained from which excess solvent was removed in vacuo, then the ligand was isolated by column (silica gel) chromatography and by recrystallization from various solvents like diethyl ether, ethanol, chloroform, method etc.

Ligand: White crytals , yield 80%, m.pt. 130-133 $^{\circ}$ c; Analysis calculated for  $\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5$  : C, 68.99, H : 8.89, N : 13.40% .Found C :69.20, H : 8.96, N: 13.35%.

IR(KBr,  $\text{cm}^{-1}$ ) – 3063, 1802, 1675, 1650, 1525, 1434, 1341, 1120, 840, 393,

UV-Visible ( $\lambda_{\text{nm}}$  – 250nm) Absorbance 3.212, MS, m/z Calc. for  $\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5$  :766 gm/mol. Found 763 gm/mol.

Solubility: Ethanol, DMF, DMSO, Cyclohexane etc.



**Scheme 1: Synthesis of Ligand**

### Synthesis of Nickel (II) Complexes

Nickel (II) salts (1.52 g, 1 mmol) was dissolved in 10 ml absolute ethanol. A two fold ratio of the schiff base ligand (1.86 g ,2 mmol) dissolved in 25 ml ethanol. Stir the solution continuously until the colour change. The resulting mixture was refluxed for 6 hrs, then excess solvent was removed in vacuum. The final coloured solid precipitate was filtered off, washed with cold solvents like ethanol diethyl ether). After washing the precipitate was allowed to dry and recrystallized using various solvent mixtures like ethanol, methanol, diethyl ether and chloroform.

#### [Ni(C<sub>34</sub>H<sub>34</sub>Br<sub>2</sub>N<sub>6</sub>O<sub>5</sub>)<sub>2</sub>]Cl<sub>2</sub> · H<sub>2</sub>O

Reddish brown, yield : 70%, m.pt. 180-190<sup>o</sup>c .Analysis calculated for C<sup>1</sup><sub>34</sub> H<sub>34</sub> Br<sub>2</sub> N<sub>6</sub>O<sub>5</sub> NiCl<sub>2</sub> ; C: 69.90, H : 8.90, N: 13.45 %. Found C : 69.90, H : 8.96, N :13.40% .

IR (KBr, cm<sup>-1</sup>) – 3390, 3161, 1643, 1602, 1109, UV-Visible (λ nm) 318 nm (absorbance 3.647), molar conductance (DMSO, 25<sup>o</sup>c λm : 59 S cm<sup>2</sup> mol<sup>-1</sup>, solubility : ethanol, DMSO, DMF.

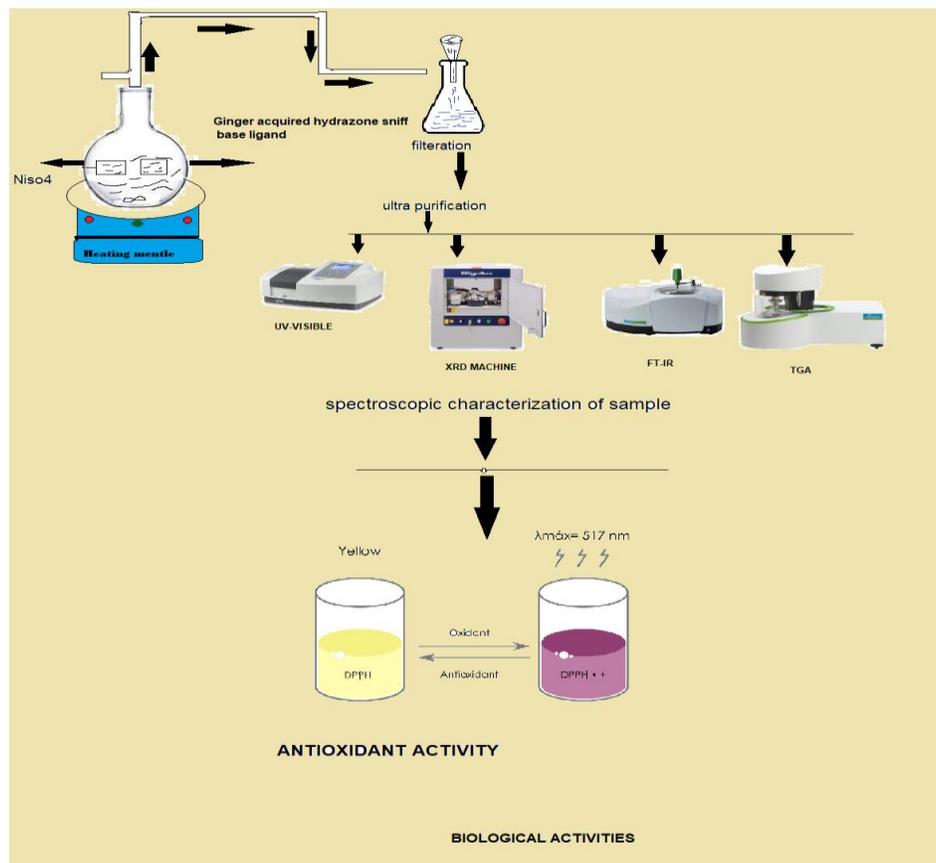
#### [Ni (C<sub>34</sub> H<sub>34</sub> Br<sub>2</sub> N<sub>6</sub> O<sub>5</sub>)<sub>2</sub>] S0<sub>4</sub>. 2H<sub>2</sub>O

Dark brown, Yield : 65%, m.pt 190-200<sup>o</sup>c, Analysis calculated for C<sub>34</sub> H<sub>34</sub> Br<sub>2</sub> N<sub>6</sub> O<sub>5</sub> NiSO<sub>4</sub> , C: 68.90, H: 8.85 , N : 13.85%, Found C : 69.0, H :8.90, N : 13.90%, IR(KBr, cm<sup>-1</sup>)- 3764, 3195, 1652, 1608, 1124 cm<sup>-1</sup>, UV-Vis (λ nm : 247 nm) absorbance 3.878, molar conductance (DMSO, 25<sup>o</sup>C, λm : 57 S cm<sup>2</sup> mol<sup>-1</sup>. ; Solubility: ethanol, menthol, DMSO, DMF.

#### [Ni (C<sub>34</sub> H<sub>34</sub> Br<sub>2</sub> N<sub>6</sub> O<sub>5</sub>)<sub>2</sub>] (CH<sub>3</sub>COO)<sub>2</sub> .6H<sub>2</sub>O

Light brown ; Yield – 70%, m.pt. 280-290<sup>o</sup> C ,Analysis calculated for C<sub>34</sub> H<sub>34</sub> Br<sub>2</sub> N<sub>6</sub> O<sub>5</sub> Ni (CH<sub>3</sub>COO)<sub>2</sub> ; C: 68.88, H: 8.36 : N: 13.95% ,Found : C: 69.01, H: 8.84 ; N:13.97%.IR(KBr, Cm<sup>-1</sup>) -3768, 3293, 1666, 1610, 1126 cm<sup>-1</sup>.





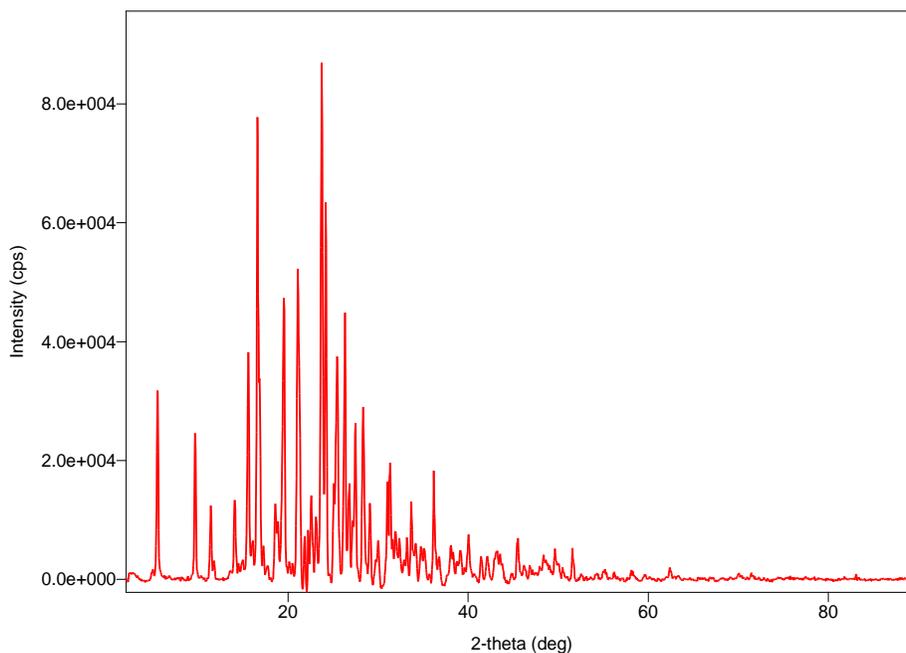
**Figure 1: Experimental set up for synthesis of ligand and its Ni (II) Complexes**

The template reactions were carried out in the presence of  $\text{NiX}_2 \cdot 6\text{H}_2\text{O}$ , an distorted octahedral  $[\text{Ni}(\text{L})_2\text{X}] \cdot 6\text{H}_2\text{O}$  (where  $x = \text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{CH}_3\text{COO}^-$ ) complexes resulted initially. In case of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  salt the axial  $\text{Cl}^-$  ligands in the Ni(II) complex are labile in aqueous solution, and the complex turns to square planar Ni (II) species  $[\text{Ni}(\text{L})_2]^{2+}$ , which is precipitated by the addition of appropriate anions such as Lithium Chlorate or  $\text{NaBF}_4$ . In the solid state, Ni (II) complexes with ligand are reddish brown, dark brown and light brown in colour. All compound are stable at room temperature and all are soluble in DMSO and DMF solvents in all proportion. The elemental analysis of the compounds was consistent with the Proposed structure of the compounds. The molar conductance values of Ni (II) complexes were 59, 57 and  $54 \text{ } \Lambda^{-2} \text{ cm}^{-2} \text{ mol}^{-1}$  in DMF solution at room temperature confirms the formation of 2:1 electrolytic nature and indicates the electrolytic nature of synthesized nickel complexes.

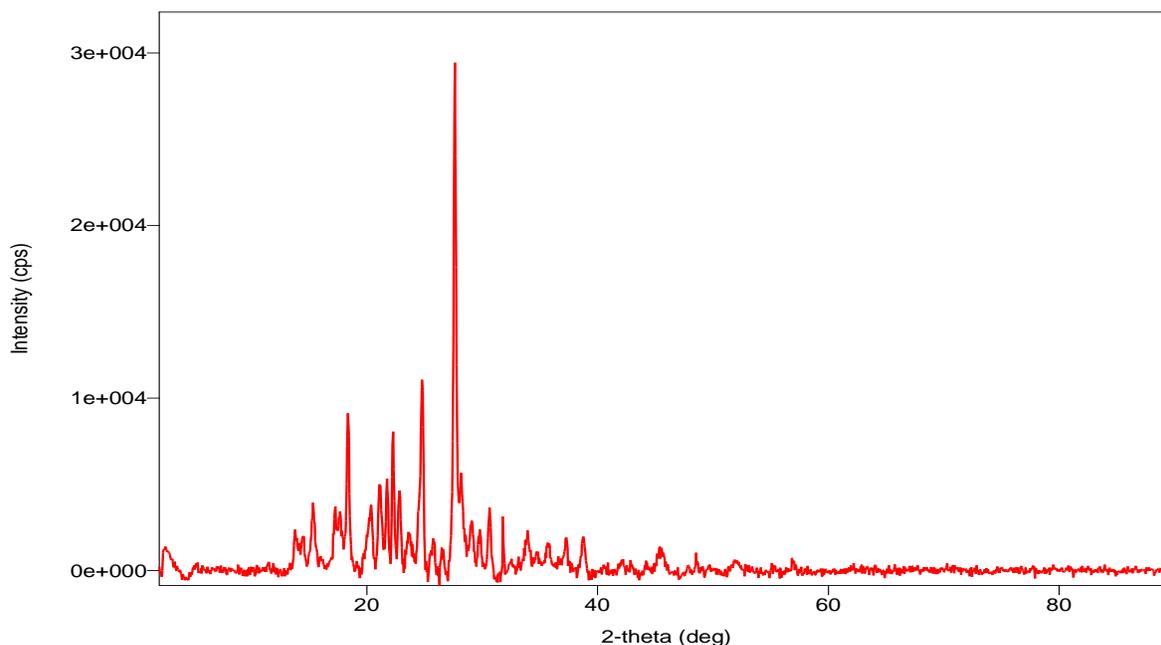
### Structural Studies

X- ray diffractogram for Ni(II) complexes obtained by Schiff base ligand is shown in fig 2. The complexes  $[\text{Ni}(\text{L})_2]\text{X} \cdot 6\text{H}_2\text{O}$  (where  $\text{X} = \text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{CH}_3\text{COO}^-$  anions) were crystallized in monoclinic crystal system. The ligand  $[\text{C}_{34}\text{H}_{34} \text{Br}_2 \text{N}_6 \text{O}_5]$  coordinated to  $\text{Ni}^{2+}$  ion in a monobasic tridentate manner through its azomethine nitrogen, and thione sulfur atoms, thus forming a six

membered rings. A distorted square planar geometry was found around the nickel ion because of the deviation of the trans angle from the ideal  $180^\circ$  angle. The anions like a  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{CH}_3\text{COO}^-$  ions were present outside the coordination sphere to compensate the oxidation state of nickel (II) ion. X-ray Patterns are shown in figure 2 & 3 for Ni (ii) complexes. All the samples were characterized at room temperature by X-ray diffraction using Cu K  $\alpha$  radiation. The diffraction pattern of complexes were recorded between  $2\theta$  ranging from  $10^\circ$  to  $80^\circ$ . The particle size of the samples are estimated using the Scherer's formula. According to Scherer's equation, the particle size is given by  $t = \frac{0.9\lambda}{B\cos\theta}$ , where  $t$  is the crystal thickness (in nm),  $B$  is half width (in radians),  $\theta$  is the Bragg's angle and  $\lambda$  is the wave length of X-rays ( $1.5413\text{\AA}$ ). The particle size corresponding to each diffraction maxima are determined from the measurement of the half width of the diffraction peak. The Particle size corresponding to each diffraction maxima are determined from the measurement of the half width of the diffraction peaks. Lattice parameters for simple cubic crystal structure is determined by  $a^2 = \frac{\lambda^2(h^2+k^2+l^2)}{4\sin^2\theta}$ . The value of lattice Parameter and particle size are shown in table 1 for all the complexes. The particle size were found to be within the range 4.23 to 9.76 nm.



**Figure 2: XRD spectra of Schiff base ligand**



**Figure 3: XRD Spectra of Ni (II) complex derived from Schiff base ligand**

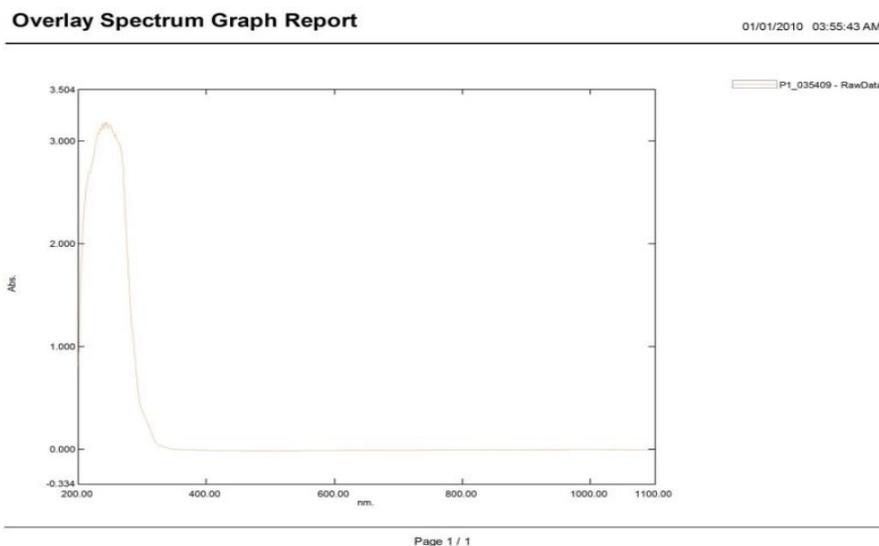
**Table 1 Lattice parameter and particle size of Ni (II) complexes.**

Sr. No.	Complexes	Lattice Parameter(A <sup>o</sup> )	Particle size (nm)
1	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ]Cl.H <sub>2</sub> O	7.56	4.20
2	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ]SO <sub>4</sub> .H <sub>2</sub> O	7.83	6.68
3	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ]CH <sub>3</sub> COO.H <sub>2</sub> O	8.08	9.70

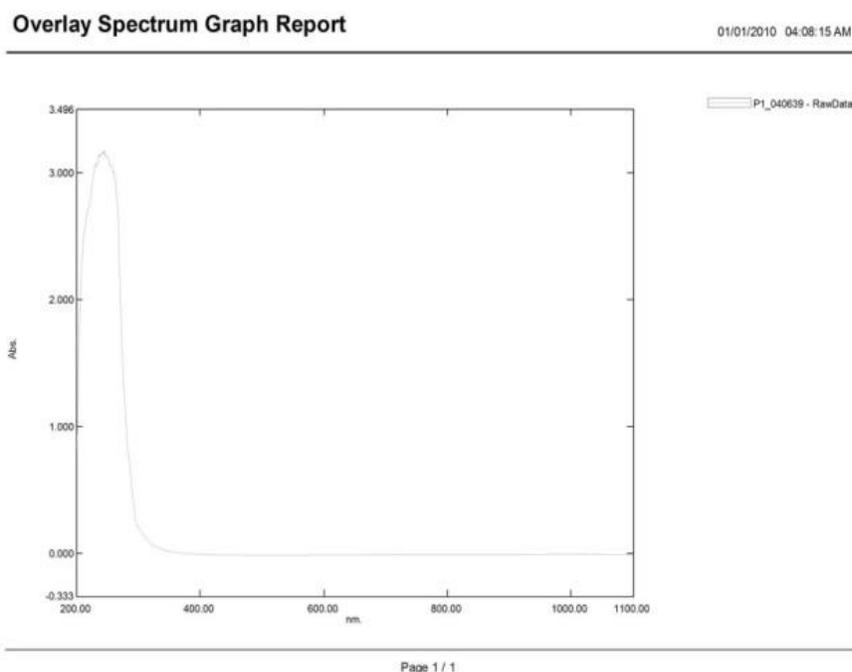
## Optical Properties

### UV-Visible Spectroscopy Study

The scanning of the spectra for the ligand and its coordinated Nickel (II) complexes were done from wavelength range of 200-700 nm. The maximum absorbance wavelength was observed from the data for the ligand and its coordination compounds with Ni<sup>2+</sup> ions as shown in figure 4 & 5. The absorption of these solutions was measured at 318 nm, 314 nm, 245 nm, 247 nm, 245 nm and 246 nm. Ligand showed strong absorption band at higher frequency (314nm) which may be due to  $\pi-\pi^*$  transition. In the case of coordination complexes of Nickel (II) absorption band occur at 314, 318, and 247nm which may be due to  $n-\pi$  and  $\pi-\pi$  transitions. Additional absorption bands also occurs at 310, 322, and 240 nm which may be due to  $n-\pi^*$  and  $\pi-\pi^*$  transitions.



**Figure 4: UV-Visible Spectra of Schiff base Ligand**

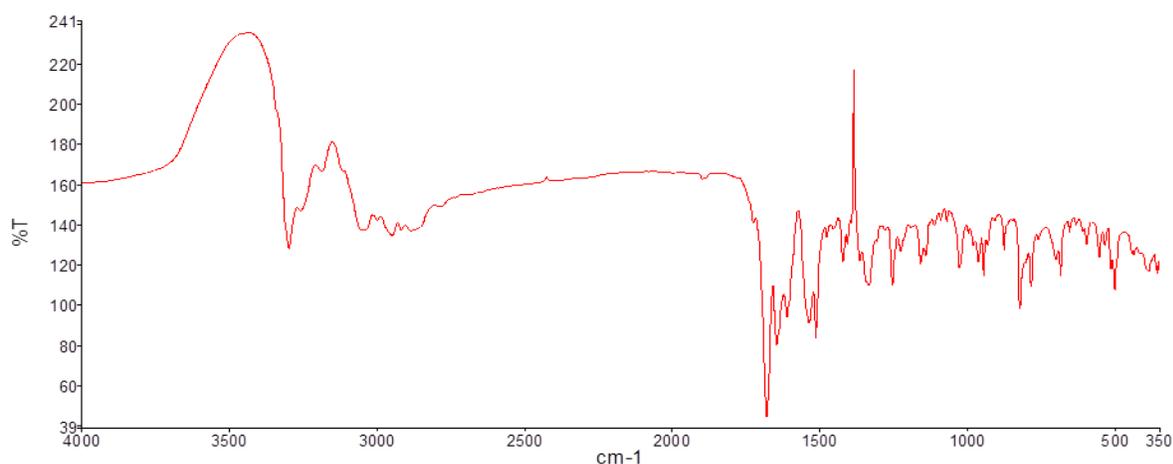


**Figure 5: UV-Visible Spectra of Ni(II) derived from Schiff base ligand**

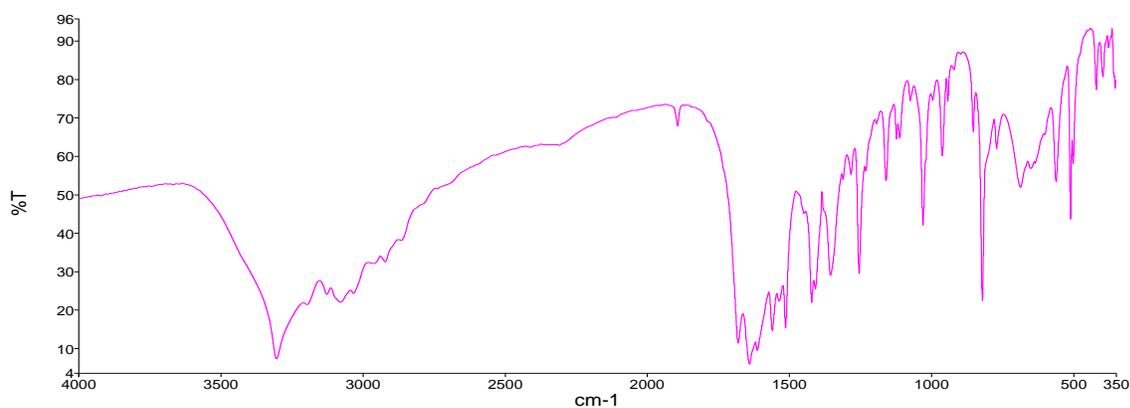
### FT-IR Spectroscopy Analysis

All synthesized compound produced IR spectra bands consistent with those in literature spectra of similar schiff base ligand and its derived Ni(II) complexes. Disappearance of bands from amine (-NH<sub>2</sub>) and aldehyde (-CHO) groups confirmed reaction of the starting compounds. Appearance of a peak in the region 1640-1660 cm<sup>-1</sup> coming from an azomethine (C=N) bond, confirmed schiff

base ligand production and this was shifted to 1620-1635  $\text{cm}^{-1}$  on production of Ni(II) complexes. This lowering in frequency indicates electron pair donation by the nitrogen atom of the Ni (II) ions. For the Ni(II) complexes there was an appearance of bands for Ni-N 480  $\text{cm}^{-1}$  in far IR region, which were not presented in spectra of the original schiff base ligand. Synthesis of the schiff base ligand was further confirmed by mass spectrometry. All experimental evidence suggests that the general formula for the synthesized Ni (II) complexes are  $\text{NiL}_2\text{X}_2$  in which the ligand acts as tridentate.



**Figure 6: FT-IR Spectra of Schiff base ligand**



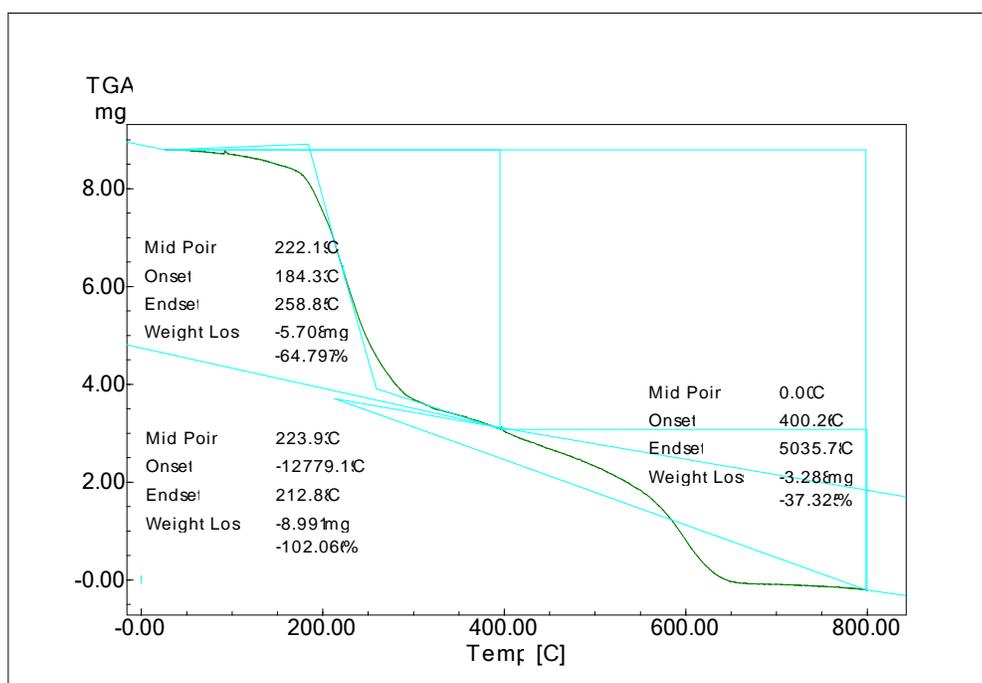
**Figure 7: FT-IR Spectra of Ni complex derived from Schiff base ligand**

Compound	V[O-H]	V[C-H]	V[C=O]	V[C=N]	V[C-O]
1. $[\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5]$	3063 $\text{cm}^{-1}$	2920 $\text{cm}^{-1}$	1796 $\text{cm}^{-1}$	1675 $\text{cm}^{-1}$	1126 $\text{cm}^{-1}$
2. $[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2] \text{Cl}_2 ; \text{H}_2\text{O}$	3390 $\text{cm}^{-1}$	3161 $\text{cm}^{-1}$	1643 $\text{cm}^{-1}$	1602 $\text{cm}^{-1}$	1109 $\text{cm}^{-1}$
3. $[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2] \text{SO}_4 \cdot 2\text{H}_2\text{O}$	3345 $\text{cm}^{-1}$	3241 $\text{cm}^{-1}$	1660 $\text{cm}^{-1}$	1600 $\text{cm}^{-1}$	1115 $\text{cm}^{-1}$
4. $[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2](\text{CH}_3\text{COO})_2 \cdot 6\text{H}_2\text{O}$	3380 $\text{cm}^{-1}$	3167 $\text{cm}^{-1}$	1670 $\text{cm}^{-1}$	1620 $\text{cm}^{-1}$	1200 $\text{cm}^{-1}$

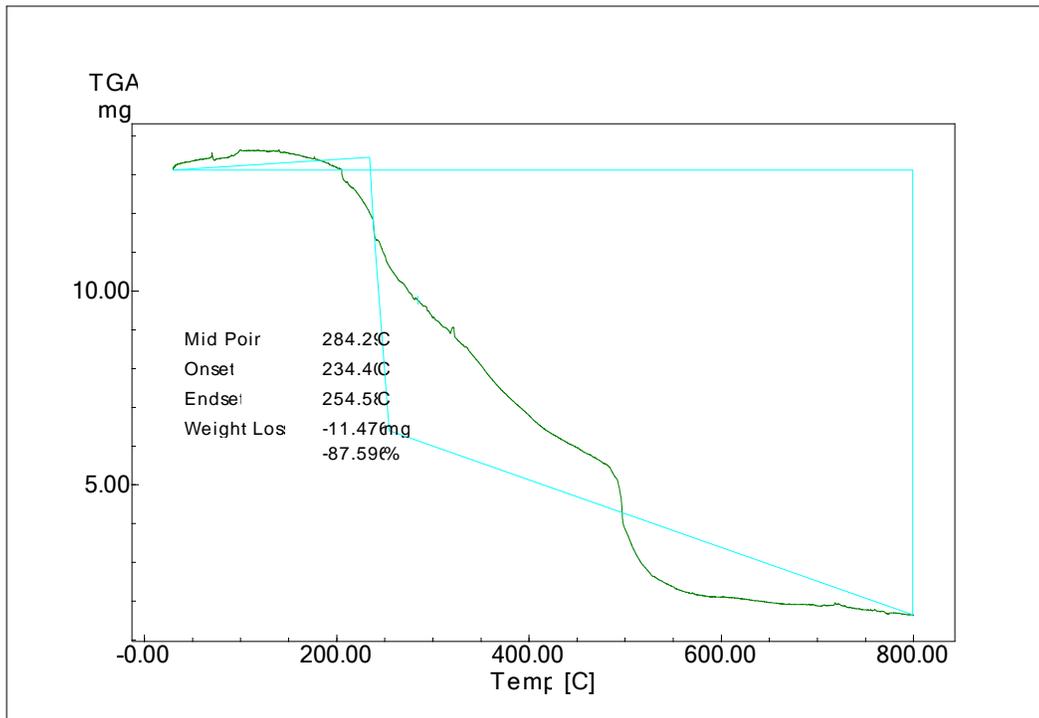
### TGA [Thermogravimetric analysis]

TGA can be used to evaluate the thermal stability of a material. In a desired temperature range, if a species is thermally stable, there will be no observed mass change. Negligible mass loss corresponds to little or no slope in the TGA trace. TGA also gives the upper use temperature of a

material. Beyond this temperature the material will begin to degrade. Thermal Analysis of Compounds can be done by Thermogravimetry analysis TGA method. The results of the thermal analysis for ligand and their coordinated nickel (II) complexes are given in the Table 2 and the thermo grams are shown figure 8 & 9 . Coordinated nickel (II) complexes are stable at room temperature and did not change the colour in safekeeping in a dry place. The thermal decomposition of the complexes takes place in four steps. The first decomposition step, an endothermic one, in the temperature range of 80<sup>0</sup>C-130<sup>0</sup>C is associated for all complexes to the loss of crystalline water (90-94<sup>0</sup>C). The second step of the thermal decomposition of the complexes corresponds to the elimination of coordinated water and takes place over a temperature range of 150<sup>0</sup>C-230 <sup>0</sup>C, associated by an endothermic peak .For the complexes, second step can be correlated with the ligand side group releases, phenolic groups of ligand for complex,weight loss in accordance with the elimination of coordinated water molecules. In the next step of decomposition is a complex reaction step, being an overlap of process. The third stage of thermal decomposition process corresponding to the loss of chloride, sulphate and acetate ions. The last step of thermal decomposition was strongly exothermic corresponding to the oxidative degradation of the organic ligand residue. It starts from 380 to 490<sup>0</sup> C and finishes around 740<sup>0</sup> C for all the complexes. The final residue of decomposition is Ni O and the nickel percentage determine from this is in accordance with the theoretical content.



**Figure 8: TGA Spectra of Schiff base ligand**



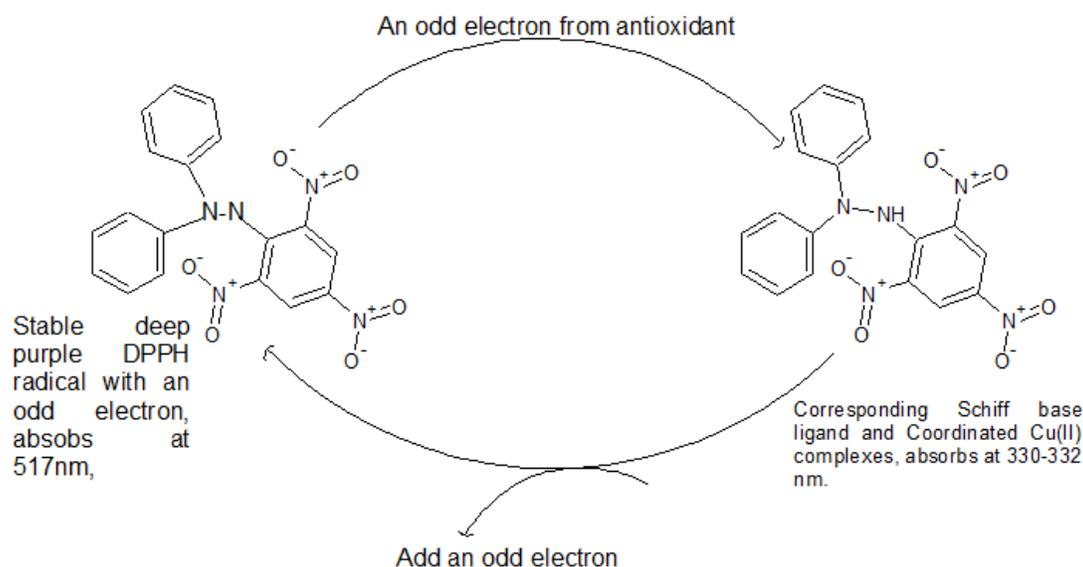
**Figure 9: TGA Spectra of Ni (II) Complex derived from Schiff base ligand**

Table 2: Thermal Decomposition data for the Schiff base ligands and their coordinated Ni(II) Complexes

Complex	Step	Thermal effect	Temperature range ( $^{\circ}\text{C}$ )	% $\Delta\text{m}_{\text{exp}}$	% $\Delta\text{m}_{\text{calc}}$	Chemical Process
$[(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2]$	I	Endothermic	80-130 $^{\circ}\text{C}$	1.82	2.10	H <sub>2</sub> O Loss
	II	Endothermic	110-210 $^{\circ}\text{C}$	3.4	4.32	Coordinated water molecule Loss
	III	Exothermic	210-360 $^{\circ}\text{C}$	19.1	19.25	Loss of moisture
	IV	Exothermic	360-780 $^{\circ}\text{C}$	65.00	65.66	Oxidative degradation of organic residue
$[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2]\text{Cl} \cdot \text{H}_2\text{O}$	I	Endothermic	60-80 $^{\circ}\text{C}$	1.94	2.20	H <sub>2</sub> O Loss
	II	Endothermic	160-220 $^{\circ}\text{C}$	3.9	4.85	Loss of Coordinate water molecules
	III	Exothermic	230-490 $^{\circ}\text{C}$	22.4	24.2	Loss of chloride ions
	IV	Exothermic	490-750 $^{\circ}\text{C}$	54.00	56.00	Oxidative degradation of organic residue
$[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2]\text{SO}_4 \cdot \text{H}_2\text{O}$	I	Endothermic	70-90 $^{\circ}\text{C}$	2.12	2.90	Loss of H <sub>2</sub> O molecules
	II	Endothermic	120-190 $^{\circ}\text{C}$	5.20	5.68	5H <sub>2</sub> O Loss
	III	Exothermic	200-400 $^{\circ}\text{C}$	24.4	25.2	Loss of sulphate ions
	IV	Exothermic	400-790 $^{\circ}\text{C}$	49.2	52.4	Oxidative degradation of organic residue
$[\text{Ni}(\text{C}_{34}\text{H}_{34}\text{Br}_2\text{N}_6\text{O}_5)_2]\text{CH}_3\text{COO} \cdot \text{H}_2\text{O}$	I	Endothermic	80-100 $^{\circ}\text{C}$	4.2	2.60	H <sub>2</sub> O Loss
	II	Endothermic	160 -180 $^{\circ}\text{C}$	6.20	6.24	Loss of Water of crystallization
	III	Exothermic	450-800 $^{\circ}\text{C}$	25.6	5.34	Loss of acetate ions
	IV	Exothermic	390 -410 $^{\circ}\text{C}$	48.8	56.8	Oxidative degradation of organic residue

## Antioxidant activity

All the synthesized compounds were screened for antioxidant activity using DPPH assay. DPPH is a stable free radical compound and has been widely used to test the radical scavenging activity of numerous chemicals, includes herbal products and synthesized compounds. It is very good to note that DPPH activity of all the synthesized compounds is greater than schiff base ligand. The results showed that Ni(II) complexes showed greater antioxidant activity than ascorbic acid (positive control), and the compound 3 showed greater antioxidant activity than ascorbic acid. The results of antioxidant activity of all the synthesized compounds data are in accordance with the theoretical aspects, because the position and the number of the –OH groups as well as the degree of conjugation of the whole molecule are important. The antioxidant efficacy of schiff base ligand and its nickel (II) complexes have similar conjugation level in roughly proportional to the presence of total number of hydroxyl groups in the benzene ring <sup>19</sup>.



Scheme-1 Mechanism of addition and elimination of electron in DPPH molecule

## Mechanism of antioxidant activity of Synthesized Compounds

An unpaired electron present on the nitrogen atom of DPPH radical is responsible for the absorbance at 5-17nm and also for a visible deep purple colour. When DPPH radical accepts an unpaired electron donated by an antioxidant compound, the colour of the DPPH solution decolorizes, which can be quantitatively measured from the changes in absorbance. The reverse reaction is evaluated by adding DPPH-H at the end of the reaction. If there is an increase in the percentage of remaining DPPH free radical at the plateau, the reaction is reversible; otherwise it is a complete reaction. <sup>20,21,22</sup>. The DPPH assay evaluates the ability of the sample to donate H to the DPPH radical, resulting in decolourization of DPPH solution. The greater the decolourization

action of solution, the higher the antioxidant activity, and this was reflected in a lower IC<sub>50</sub> value of samples.

**Table 4 : The trolox<sup>a</sup> equivalent antioxidant activity**

S.N.	Compound	DPPH activity (IC <sub>50</sub> µg/ml)
1	Schiff base ligand [(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ]	1.64
2	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ].Cl. H <sub>2</sub> O	2.46
3	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ].SO <sub>4</sub> .H <sub>2</sub> O	3.22
4	[Ni(C <sub>34</sub> H <sub>34</sub> Br <sub>2</sub> N <sub>6</sub> O <sub>5</sub> ) <sub>2</sub> ].CH <sub>3</sub> COO. H <sub>2</sub> O	3.60
5	Ascorbic Acid	3.76

TEAC- Trolox equivalent antioxidant capacity has been calculated from molar absorptivity by dividing  $1.64 \times 10^4$ .

## CONCLUSION

In conclusion, the resulting synthesized compound of schiff base ligand and its coordinated nickel (II) complexes were evaluated for their antioxidant activity and compared with standard compound Ascorbic acid. Synthesized schiff base ligand and its coordinated Ni (II) complexes showed more antioxidant activity than ligand. The complexes prepared from nickel acetate complex showed maximum antioxidant activity, while antioxidant activity decreases in nickel chloride dehydrate and nickel sulphate complexes.

## DECLARATION OF COMPETING INTEREST

The authors do not declare any conflict of interest. The authors declare no conflict of interest associated with this article and also no significant financial support has been received by us for preparing this manuscript.

## Credit authorship contribution statement

Richa Kothari: Conceptualization, Designing, Formal analysis, Funding acquisition, methodology, writing original draft, writing review & editing.

## ACKNOWLEDGMENT

The authors are grateful to the research facilities available at the Sophisticated Instrumentation Research laboratory, ITM University Gwalior. All the authors sincerely acknowledge CIF, Jiwaji University Gwalior for elemental analysis.

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