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Synthesis and characterization of Nickel (II) complexes of some 2-hydroxy-4,5-dimethylacetophenone substituted Hydrazones

Dr. Dinkar Gagare, Dr. Raju Patil, Dr. Rama Lokhande and Dr. Ajay Deshmukh

Abstract

The present paper deals with synthesis and characterization of metal complex of some novel 2-hydroxy-4,5-dimethylacetophenone substituted hydrazones. The substituted hydrazones were prepared by reacting 2-hydroxy-4,5-dimethyl acetophenone with corresponding hydrazine, Phenyl hydrazine and (2,4-dinitrophenyl) hydrazine. The Nickel (II) metal complexes of general formula ML_2 with newly prepared hydrazone synthesized and characterized by several physicochemical techniques like UV, Infrared, ¹HNMR, decomposition temperature/ melting point, elemental analysis, molar conductance and magnetic susceptibility studies. The analytical data confirmed 1:2 stoichiometry of M:L and based on above analytical technique data suggest that each one Ni (II) complexes have Octahedral geometry. The conductivity data show that each one these complexes are non-electrolytes.

Keywords: Transition metal complexes, tautomerism, hydrazone, mutagenicity, non-electrolytes, phenyl hydrazine and (2,4-dinitrophenyl) hydrazine

1. Introduction

The coordination chemistry of nickel spans a distribute of geometries, coordination numbers, and oxidation states. Nickel complexes are identified with oxidation states ranging from -1 to +4. However, the foremost common oxidation number is Ni (II) ($[Ar]3d^8$). The nickel ion in nickel (II) complexes survives within the coordination number of 4, 5 and 6. Its octahedral, quadratic-pyramidal, trigonal bipyramidal and tetrahedral complexes are paramagnetic and have in the popular of cases a green or blue colour. The quadratic-planar nickel complexes are diamagnetic and frequently have a yellow, red or brown in colour. With reference to Lewis acidity, Ni (II) is taken into account to be a uncertain metal ion. This is because it binds to both soft and hard ligands and sometimes, albeit infrequently, to both in the identical complex ^[1].

Nickel is redox active in carbon monoxide gas dehydrogenase, acetyl Coenzyme A synthase, iron-nickel hydrogenase, superoxide dismutase and methyl coenzyme M reductase. For the opposite three enzymes, the oxidation number of the nickel center is +2 and doesn't change during catalysis. Instead, the nickel center (s) in these enzymes is/are proposed to be a Lewis acid that coordinates and facilitates deprotonation of a substrate or inhibitor, or lowers the pKa of water to yield a Ni (II)-OH specie. Due to its nickel content, urease may be a exclusive example among the hydrolytic enzymes, which usually contain zinc because of the essential cofactor. In efforts to explain the inhibition process in the urease system, a number of Ni (II) complexes of acetohydroxamate derivatives were described ^[2-4].

The coordination of Ni (II) is comparatively common with tetrahedral and square planar geometries signifying the two extreme possibilities. A significant feature of square planar Ni complexes is their ability to coordinate extra ligands in solution to set up equilibrium between four, five and six coordinate complexes. Thus, diamagnetic square planar complexes can be transformed into paramagnetic octahedral Ni (II) species in coordinating solvents or in the occurrence of extra ligands.

Nickel complexes are of boundless significance and an outstanding review available on the use of them in heterogeneous catalysis, ceramics, pigments, electroplating and in hydrogen storage ^[5]. Nickel complex of benzoic acid derivative acts as a stabilizer against oxidation of polybutadiene ^[6].

Investigation on coordination chemistry has began as an active and exciting field for chemists and biologists because of the rationale that coordination compounds are mostly operated within the assembly of semiconductors, pharmaceuticals, fragrances, ceramic precursors etc. [7].

The coordination chemistry of hydrazones as ligand is a rigorous area of study and several transition metal complexes of them are investigated. Earlier, inquiries on the hydrazone complexes were focused mainly on the synthesis and structural characterization, but now these metal complexes were noted to serve some applications in various fields like sensors, non-linear optics, medicine etc.

Hydrazones are an significant class of ligand in co-ordination chemistry. Hydrazones form stable metal complexes with transition metal ions due to complexing capability of ligand through keto-enol tautomerism and readiness of other donor sites in the ligand i.e. due to occurrence of functional group, generally hydroxyl, azomethine, sufficiently near the location of co-ordination within the Hydrazone ligands [8-11]. The coordination chemistry of nickel especially in high oxidation states is of substantial attention due to its significance not only in inorganic chemistry but also in other areas of chemistry and biology [12].

Coordination chemistry of metal complexes of hydrazones, has gained a special attraction due to their biological activity and their ability to act as potential inhibitors for many enzymes. The presence of heterocyclic rings in the synthesized hydrazones plays a major role in deciding the extent of their pharmacological properties [13].

Corey *et al.* synthesized chiral complexes, tert-butyl glycinatebenzophenone nickel (II), which have improved enantioselectivity in alkylation of enolates. The biological experiments displayed that the nickel complexes have several positive applications as antiepileptic [14], anticonvulsant [15] agents or vitamins and indicated significant antibacterial [3,16], anticancer/antiproliferative [17-18], antifungal [19] and antimicrobial [20] activities.

The developments in the field of bioinorganic chemistry have increased the interest in hydrazone complexes, because it has been known that many of these complexes may act as lead for biologically important species [21-25].

2. Experimental

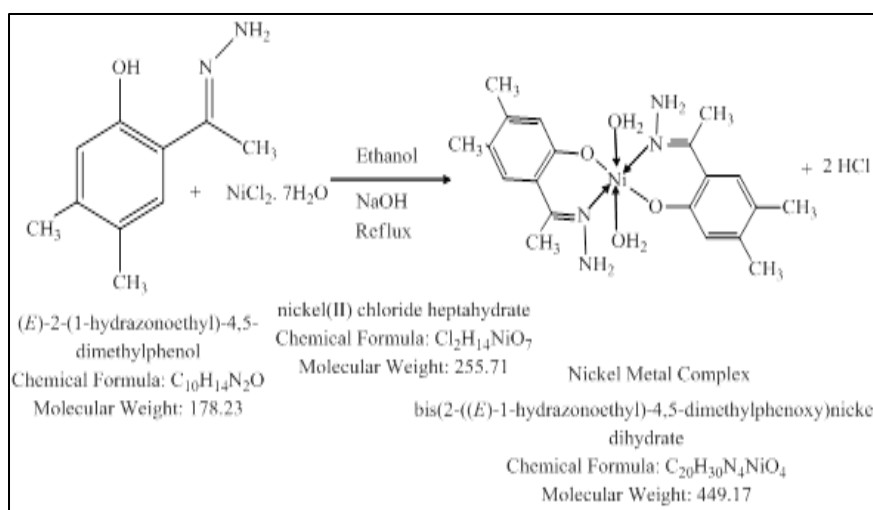
2.1 Materials

The chemicals 2-hydroxy-4,5-dimethylacetophenone, (E)-2-(1-hydrazonoethyl)-4,5-dimethyl phenol, (E)-4,5-dimethyl-2-(1-(2-phenylhydrazono)ethyl)phenol, (E)-2-(1-(2-(2,4-dinitrophenyl) hydrazono)ethyl)-4,5-dimethylphenol, nickel chloride heptahydrate, sodium hydroxide, ethanol, methanol, chloroform, DMSO, DMF etc., used in this work were of AR Grade, commercially available and used without further purification.

2.2 Preparation of Ni complex with (E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol metal Complex or bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) nickel dihydrate

(E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol were synthesized following published procedure [26]. The transition metal complex bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate were prepared by dissolution of (E)-2-(1-hydrazonoethyl)-4,5-dimethyl phenol ligand (0.01M) in ethanol at hot condition, an ethanolic solution (0.005M) of the metal salt nickel chloride heptahydrate was added drop wise with constant stirring and refluxed for 3 to 4 hrs. The progress of the reaction was checked by TLC in solvent 10% ethyl acetate: n-hexane (10:90) ratio. The reaction mass was maintained at pH 7 to 8 by addition of the 0.10 N sodium hydroxide. The light green color compound was formed. It was filtered and washed with chilled ethanol. It was dried at 40–45 °C. The compound was re-crystallized in ethanol for purification and improving description.

Reaction scheme preparation of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) nickel dihydrate is given below Scheme 1.



Scheme 1: Reaction scheme preparation of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate

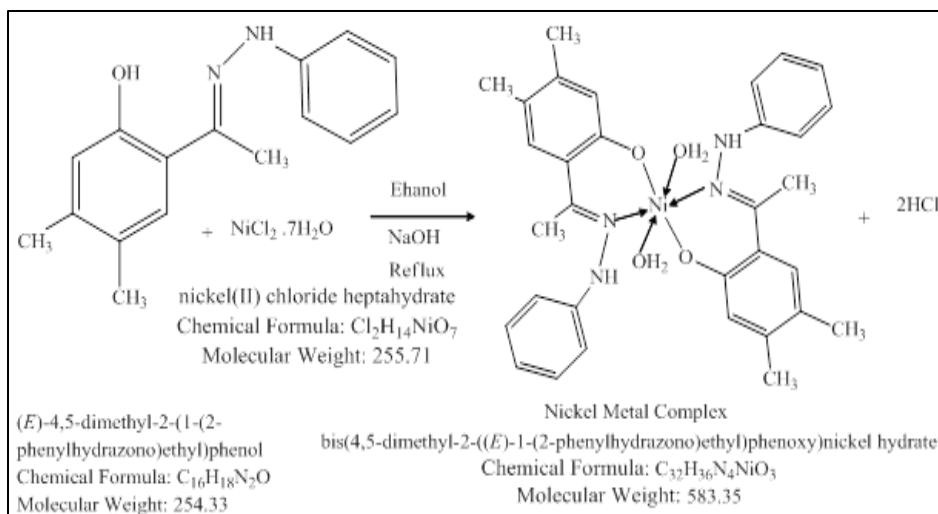
2.3 Preparation of Ni complex with (E)-4,5-dimethyl-2-(1-(2-phenylhydrazono) ethyl) phenol metal complex Or bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate

(E)-4,5-dimethyl-2-(1-(2-phenylhydrazono) ethyl) phenol were synthesized following published procedure [27]. The transition metal complex bis(4,5-dimethyl-2-((E)-1-(2-

phenylhydrazono) ethyl) phenoxy) nickel dihydrate were prepared by dissolution of (E)-4,5-dimethyl-2-(1-(2-phenyl hydrazono) ethyl)phenol ligand (0.0069M) in ethanol at hot condition, an ethanolic solution (0.0035M) of the metal salt nickel chloride heptahydrate was added drop wise with constant stirring and refluxed for 4 to 5 hrs. The progress of the reaction was checked by TLC in solvent 10% ethyl

acetate: n-hexane (10:90) ratio. The reaction mass was maintained at pH 7 to 8 by addition of the 0.10 N sodium hydroxide. The light green color compound was formed. It was filtered and washed with chilled ethanol. It was dried at 50–55 °C. The compound was re-crystallized in ethanol for

purification and improving description. Reaction scheme preparation of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate is given below Scheme 2.



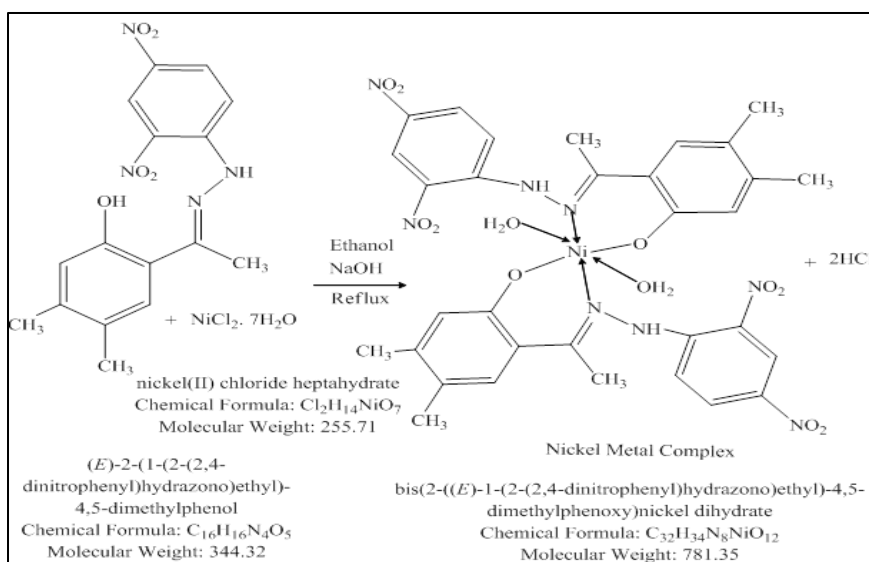
Scheme 2: Reaction scheme preparation of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate

2.4 Preparation of Ni complex with (E)-2-(1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenol metal complex or bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate

(E)-2-(1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenol were synthesized following published procedure [28]. The transition metal complex bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate were prepared by dissolution of (E)-2-(1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenol ligand (0.005M) in ethanol at hot condition, an ethanolic solution (0.0025M) of the metal salt nickel chloride

heptahydrate was added drop wise with constant stirring and refluxed for 5 to 6 hrs. The progress of the reaction was checked by TLC in solvent 10% ethyl acetate: n-hexane (10:90) ratio. The reaction mass was maintained at pH 7 to 8 by addition of the 0.10 N sodium hydroxide. The reddish-brown color compound was formed. It was filtered and washed with chilled ethanol. It was dried at 60–65 °C. The compound was re-crystallized in ethanol for purification and improving description.

Reaction scheme preparation of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate is given below Scheme 3.



Scheme 3: Reaction scheme preparation of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate

3. Results and Discussion

The Ni-Metal complexes were analyzed by Infrared spectroscopy, Ultraviolet-visible spectroscopy, ¹HNMR, Carbon-Hydrogen-Nitrogen-Sulphur elemental analyzer, Decomposition temperature/Melting point, Molar

Conductance, Magnetic susceptibility and Nickel content by Gravimetric technique.

Decomposition temperature / Melting points were recorded using Veego Scientific Device (Model: VMP-AD) in open capillaries and were uncorrected. Some physical properties of Ni-Metal complexes are mentioned in Table 1.

Table 1: Physical properties.

Ni-Metal Complex	Chemical Formula and Molecular formula	Color	Solubility	Decomposition Temperature	Yield
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)nickel dihydrate	C ₂₀ H ₃₀ N ₄ NiO ₄ and 449.17	light green	- Soluble in methanol, ethanol, Acetonitrile DMSO, DMF - Insoluble in water	> 300 °C	83.64%
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl) phenoxy) nickel dihydrate	C ₃₂ H ₃₈ N ₄ NiO ₄ and 601.36	light green	- Soluble in ethanol, methanol, DMSO, DMF - Insoluble in water	> 300°C	81.00%
bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy)nickel dihydrate	C ₃₂ H ₃₄ N ₈ NiO ₁₂ and 781.35	reddish brown	- Soluble in ethanol, methanol, DMSO, DMF Insoluble in water	> 300 °C	78.15%

3.1 Infrared analysis

The IR spectra of Ni-Metal complexes were recorded in the region of 4000–400 cm⁻¹ using FTIR spectrometer of model Agilent Resolutions Pro by direct sampling method.

The IR spectral data along with the possible assignments of Ni-Metal complexes are provided in Table 2, followed by IR spectra performed by IR direct solid method, use of Agilent Resolutions Pro and IR spectrum are showed in Figure 1, 2, 3.

Table 2: Hydrazone Nickel metal complex IR frequency, cm⁻¹

Bond/functional group	frequency, cm ⁻¹		
	bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate	bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) nickel dihydrate	bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate
N–H	3290	3239	3297
C–H	3025	3037	3080
C–H stretch aromatics	2923	2986	3925
C=N	1609	1620	1613
C–C	1559	1574	1588
C=C (aromatic ring)	1494	1431	1423
N–O	---	---	1329
C–O	1256	1256	1260
N–N	1202	1195	1114
O–Ni	Below 600	Below 600	Below 600

**Fig 1:** IR of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) nickel dihydrate

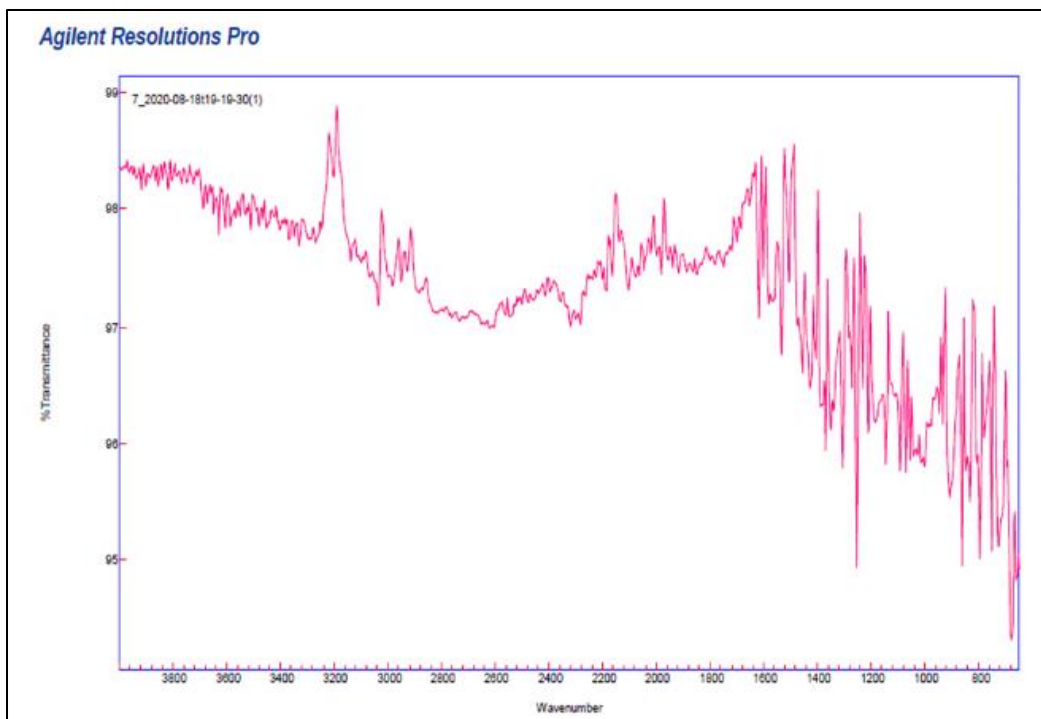


Fig 2: IR of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate

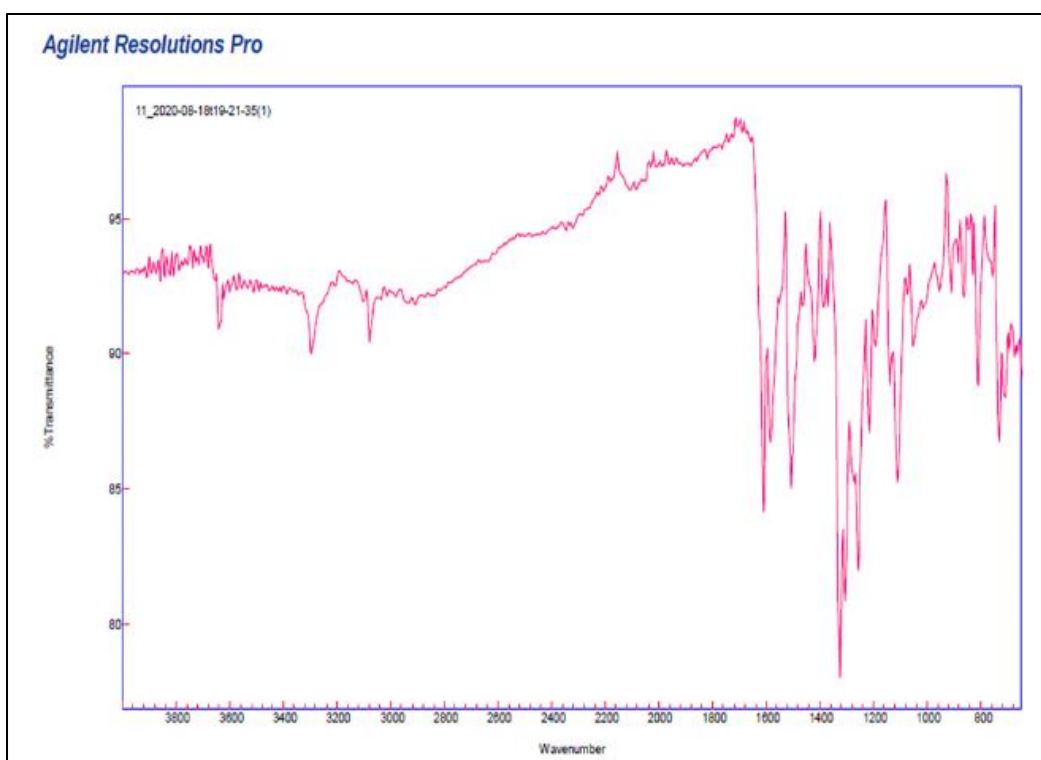


Fig 3: IR of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate

3.2 UV analysis

The UV spectra of the Ni-Metal complexes in methanol were recorded on SPECORD 50 PLUS-233 H 1409C Spectrophotometer, using a quartz cell of 1 cm optical path

where methanol was used as a blank. The UV spectrum is shown in Figure 4, 5, 6. The spectra shows λ (bands maximum in nm) are provided in Table 3.

Table 3: Ni-Metal Complex

	Wavelength nm
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) nickel dihydrate	251, 294, 710, 780, 949
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate	279, 316, 724, 858, 949
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl phenoxy) nickel dihydrate	250, 315, 716, 851, 950

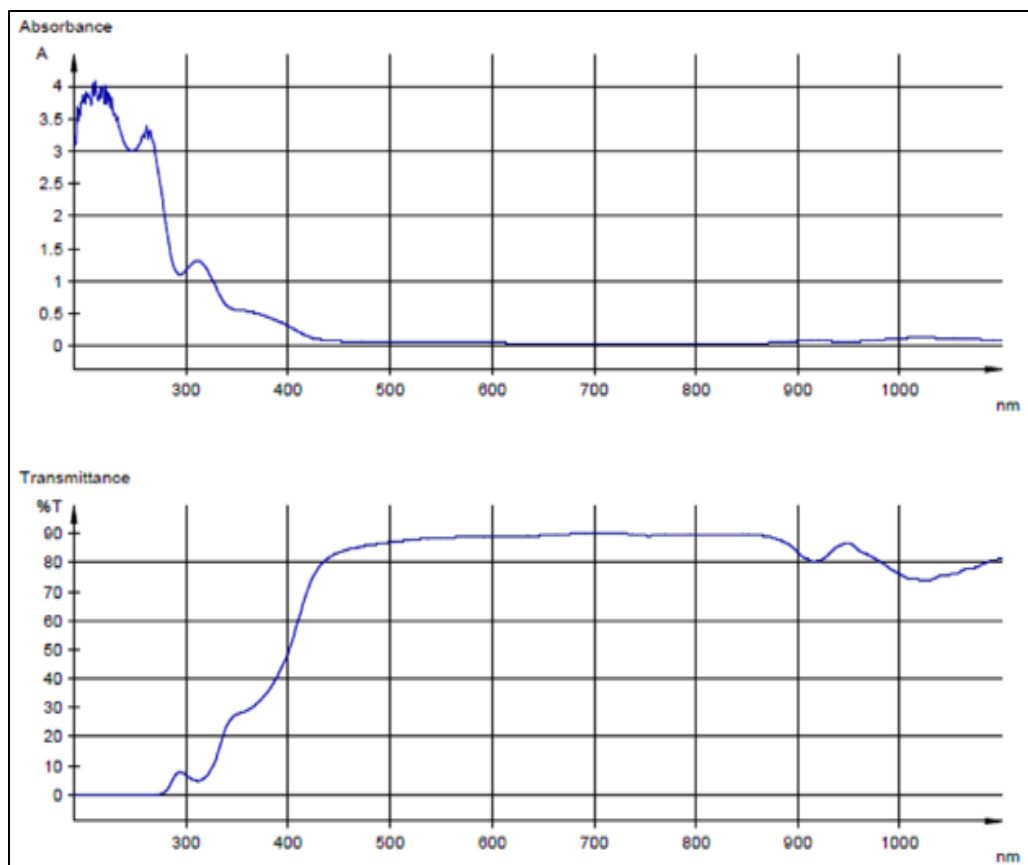


Fig 4: UV-Visible Spectra of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) sethyl) phenoxy) nickel dihydrate

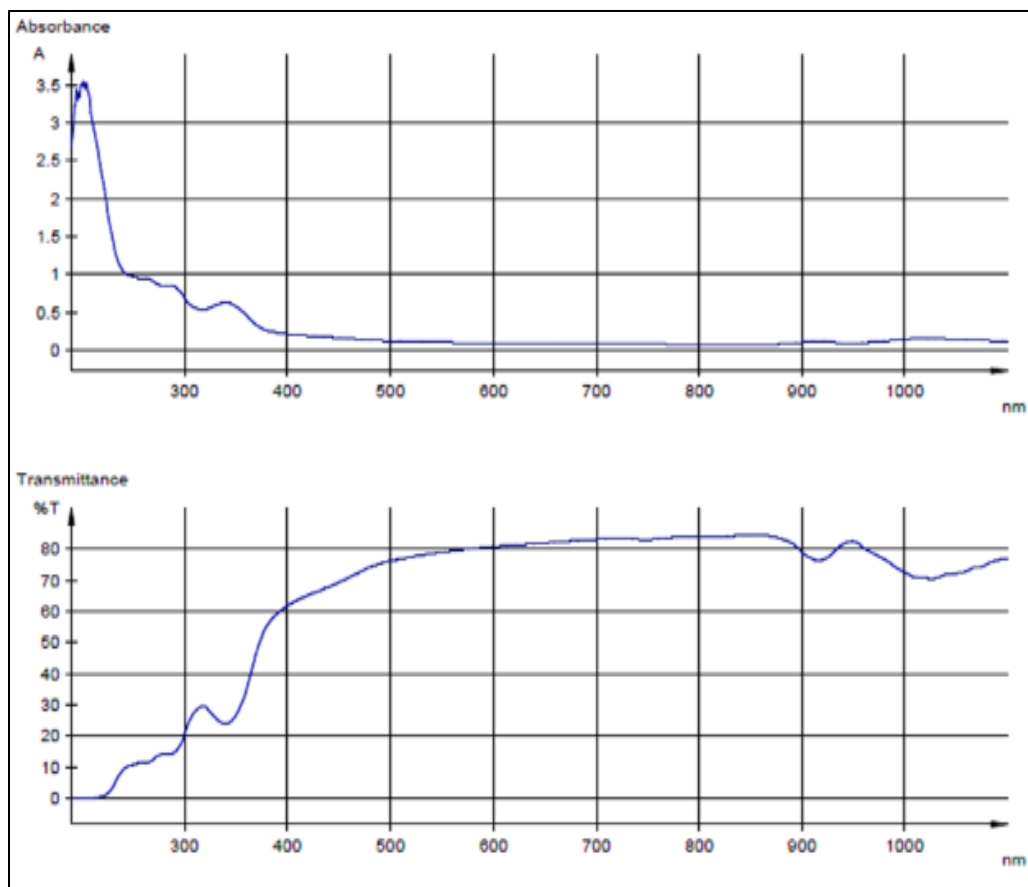


Fig 5: UV-Visible Spectra of bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) nickel dihydrate

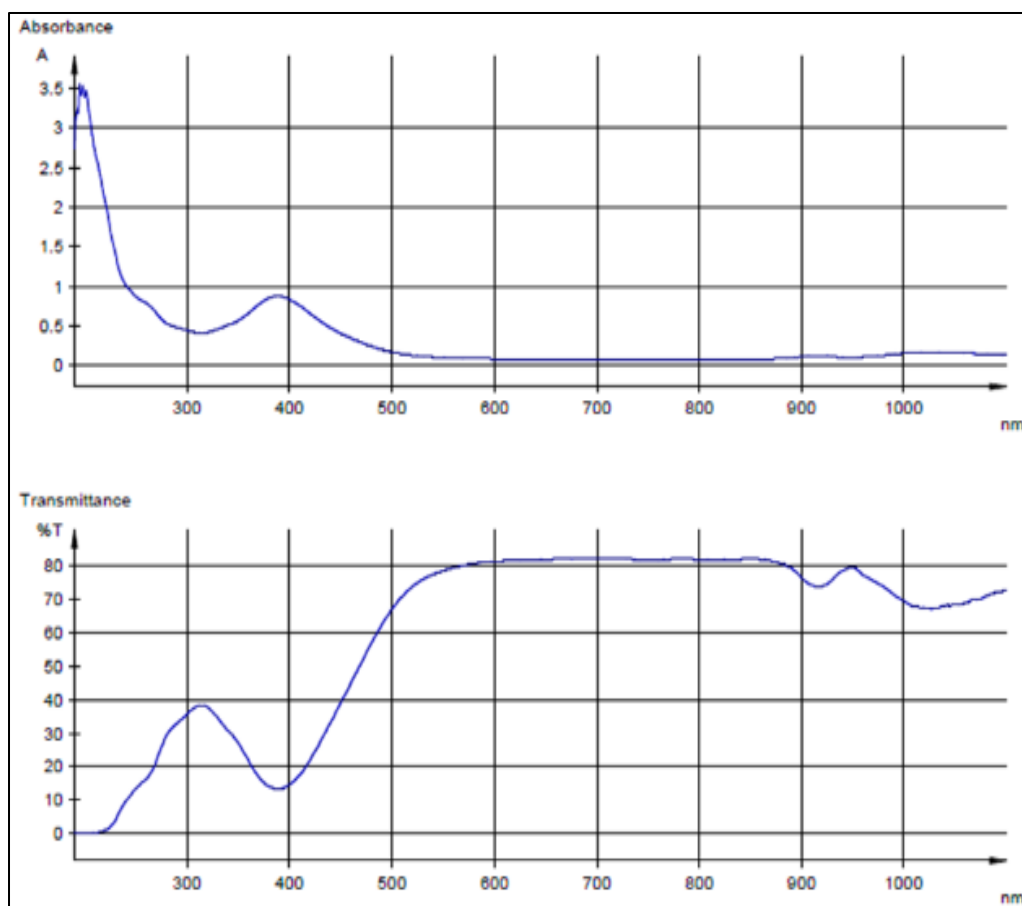


Fig 6: UV-Visible Spectra of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate

3.3 Elemental analysis (CHNS)

Formation of Ni-Metal complexes were further confirmed by elemental analysis which was recorded by Vario MICRO

CHNS analyzer. The elemental analysis data of Ni-Metal complexes and Nickel is confirmed by Gravimetric technique is summarized in Table 4.

Table 4: Carbon, Hydrogen, Nitrogen, Nickel elemental analysis.

Metal Complex	Molecular formula	Molecular weight	Elemental data (required/found)			
			Carbon	Hydrogen	Nitrogen	Nickel
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate	C ₂₀ H ₃₀ N ₄ NiO ₄	449.17	54.06 (53.48)	6.12 (6.27)	12.14 (12.48)	14.21 (13.07)
bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) nickel dihydrate	C ₃₂ H ₃₈ N ₄ NiO ₄	570.18	64.12 (63.91)	6.52 (6.38)	9.55 (9.32)	10.38 (9.76)
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate	C ₃₂ H ₃₄ N ₈ NiO ₁₂	750.17	50.12 (49.19)	4.16 (4.39)	14.16 (14.34)	7.87 (7.51)

3.4 Magnetic Susceptibility

The magnetic moment (μ_{eff}) values at room temperature for Ni (II) complexes complexes was in the range of 2.9–3.3

B.M. These values are expected for Octahedral geometry of Ni(II) complexes at room temperature and further supported by electronic spectral data is summarized in Table 5.

Table 5: Magnetic Susceptibility

Ni-Metal complex	μ_{eff} value
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate	3.12
bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) nickel dihydrate	3.15
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) nickel dihydrate	3.23

3.5 Molar Conductance ($\text{mho.cm}^2.\text{mol}^{-1}$):

The molar conductance values at room temperature were measured in Methanol (10⁻³ M) on an Elico digital direct

reading conductivity meter model CM-180 is summarized in Table 6.

Table 6: Molar Conductance

Ni-Metal complex	Molar Conductance mhos $\text{cm}^2 \text{mol}^{-1}$
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) nickel dihydrate	0.090
bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) nickel dihydrate	0.099
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl phenoxy) nickel dihydrate	0.097

3.6 ^1H NMR

I tried all Ni metal-complexes ^1H NMR and observed the spectrum of the complex differs from that of the free ligand in the following aspects:

- Resolution is very poor due to the low solubility of Metal complexes in DMSO.
- The disappearance of the signal due to the imine group, is attributed to its involvement in coordinating the Iron ion [29].

- Due to the paramagnetic nature of metal the NMR graphs are distorted.
- Due to the metal compound it shows the electrical distortion during NMR analysis.

The ^1H NMR Spectra of metal complexes are shown in Figure 7, 8, 9.

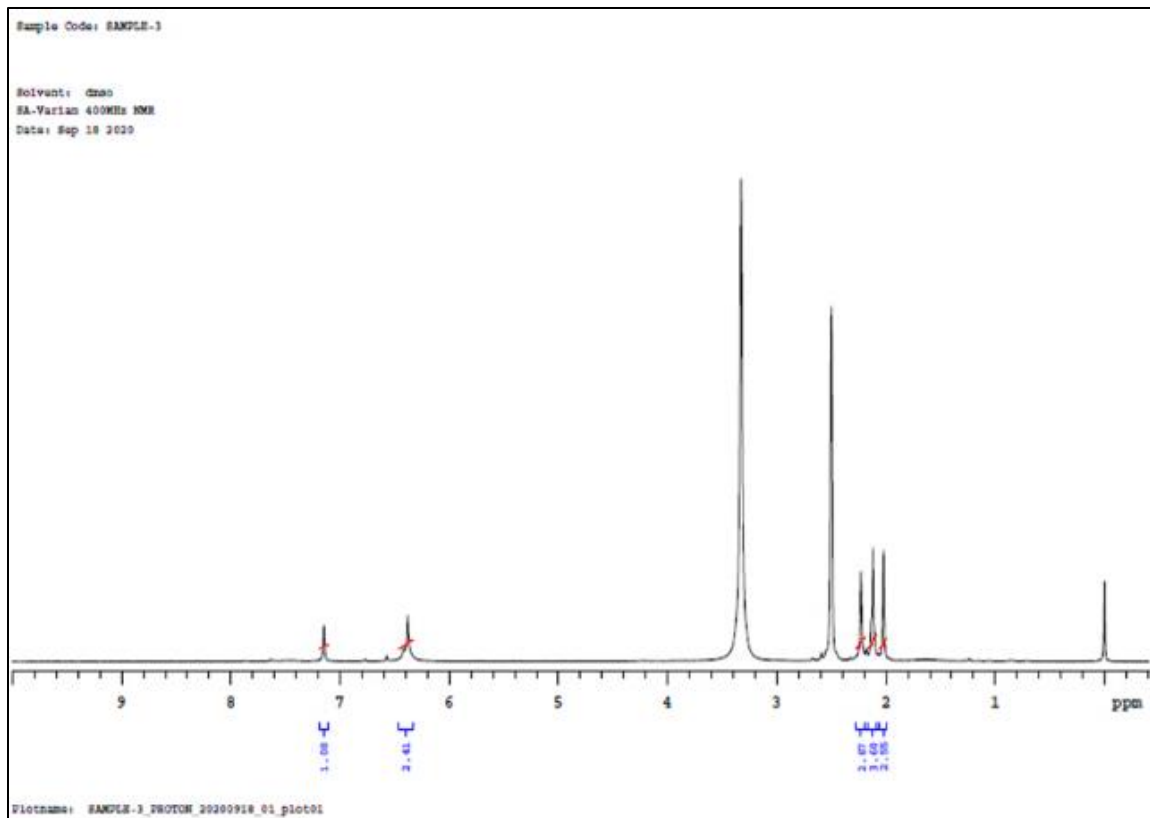


Fig 7: ^1H NMR of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) nickel dihydrate\

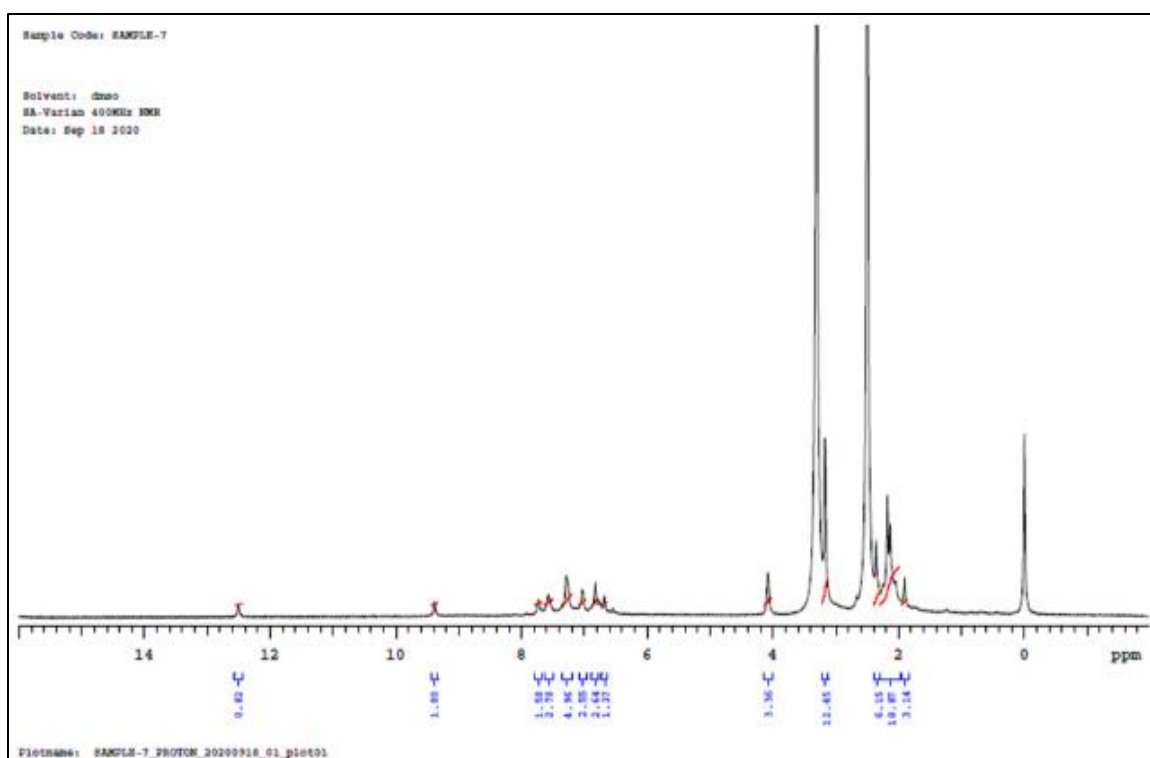


Fig 8: ^1H NMR of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) nickel dihydrate

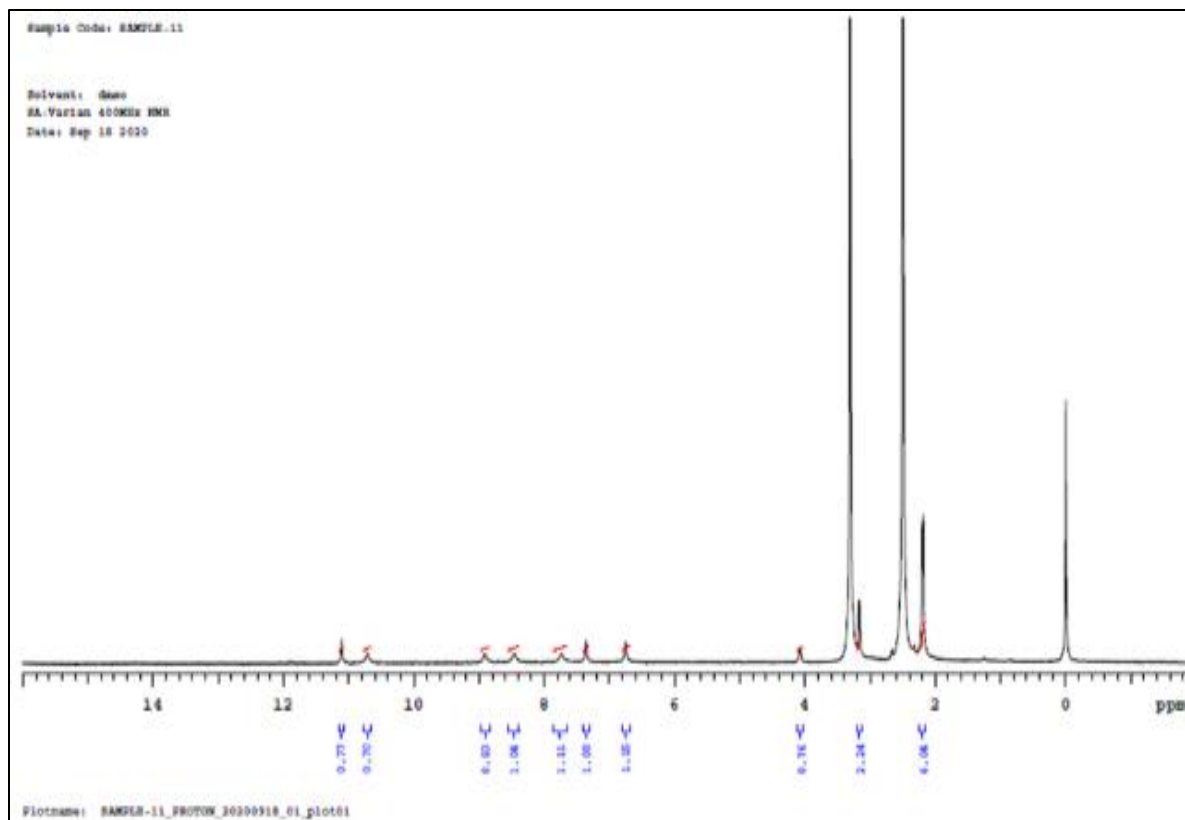


Fig 9: ^1H NMR of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl Phenoxy) nickel dihydrate

4. Conclusion

All Ni complexes are characterized by elemental analysis, IR, UV Visible spectrophotometer, ^1H NMR, Molar Conductance and Magnetic susceptibility measurement, gravimetric analysis. Based on above results it suggests the Octahedral geometry for the Ni (II) complexes and the conductivity data show that each one these complexes are non-electrolytes.

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