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Synthesis and characterization of cobalt (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 Cobalt (II) metal complexes of general formula ML_2 with newly prepared hydrazone synthesized and characterized by several physiochemical 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 Co(II) complexes have Octahedral geometry. The conductivity data show that each one these complexes are non-electrolytes.

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

1. Introduction

The field of coordination chemistry is one of the leading knowledgeable, beautiful and experimentally demanding frontiers in modern chemical sciences. Werner's basic ideas on the stereochemistry of metal complexes, appliance of isomerization etc. remain unobstructed even today despite all the advanced technical developments which have taken place since his days [1]. Recent day's coordination chemistry comprises a large body of inorganic chemistry research. Several synthetic metal complexes that mimic the behavior of complex bio molecules are known and at existing the study of such compounds is getting much consideration.

The part of metal ions in living systems is well recognized in recent years. It has been documented that inorganic biochemistry is a link between the academic disciplines viz., biochemistry and inorganic chemistry. In fact, biochemistry has been considered as the coordination chemistry of living systems. This need is well demonstrated by the observation that one third of all enzymes have a metal ion as essential component [2]. Some assessments, monographs etc., are available on the inorganic aspects of biochemistry [3]. As metal ions show such an significant role in biological systems it is to be estimated that the various aspects of coordination will be studied with deep interest.

The Co(II) ions form coordination compounds with Schiff bases, with octahedral / square planar geometry, capable of revocable binding molecular oxygen [4]. Some of such complexes are ready to bind reversibly molecular oxygen, getting used as abridged models within the study of dioxygen fixation by its natural transporters because they mimic the biological oxidation. According to this principle, cobalt chelate complexes are valuable in highly selective catalytic oxidation reactions using molecular oxygen, as models for oxygenases, peroxydases or mono- and dioxygenases. The complex compounds of cobalt with Schiff bases catalyze the oxidation of phenols, alcohols, flavonoids, nitro alkanes, hydrazines or olefins [5-8]. Some biologically significant substances are 'd'-transition metal coordination compounds that are made from huge organic molecules sure to the metal via coordinate covalent bonds example, hemoglobin (blood protein) is a coordination complex connecting Fe, vitamin B12 is a cobalt complex (cyanocobalamin), complexing agents are used for water softening (Ca, Mg, Fe removal with EDTA), an antidote for some metal poisoning British Anti-Lewisite forms complexes with As, Hg, and Cr, Fe-carbonyls are anti-knock gasoline additives [9].

Cobalt complexes have increased reputation for their applicability in the biological field [10-13]. In the race of making new drugs, cobalt complexes have attracted a great deal of attention amongst the scientific community due to their therapeutic uses as tumor imaging agent [14], transport protein transferrin [15], antimycobacterial [16], antiviral [17], antiparasitic [18], antithrombotic [19], anti-inflammatory activities [20] and as metabolic modifier [21].

2. Experimental

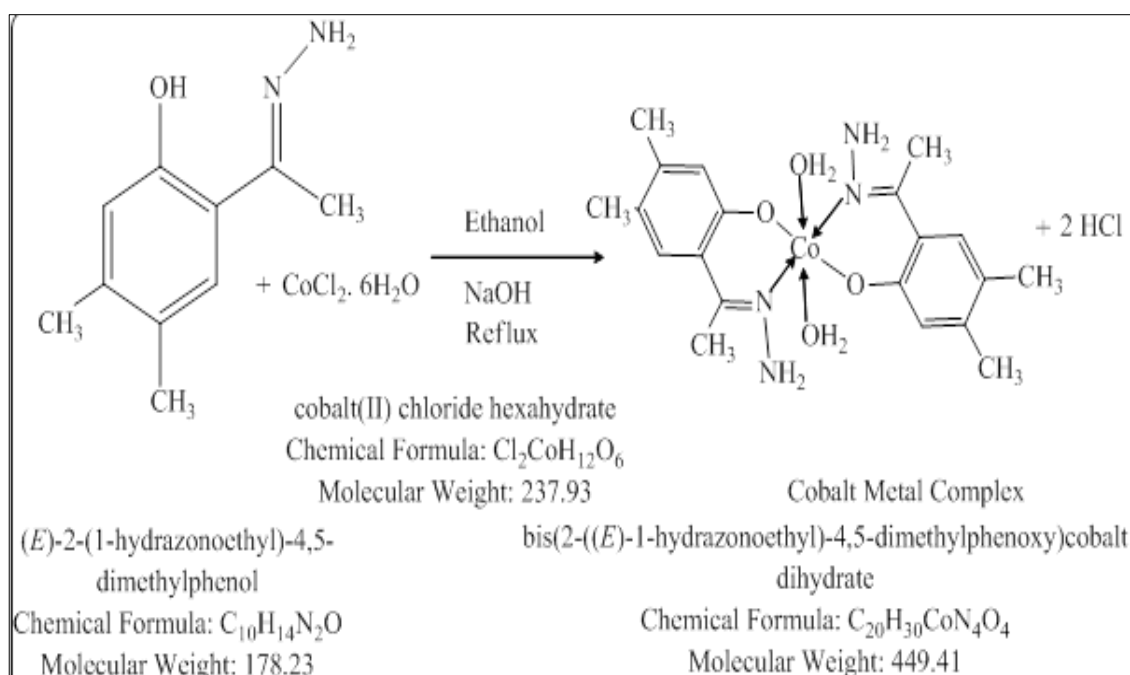
2.1 Materials

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, cobalt chloride hexahydrate, 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 Co complex with bis (2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate

(E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol were synthesized following published procedure [22]. The transition metal complex bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) cobaltdihydrate were prepared by dissolution of (E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol ligand (0.011M) in ethanol at hot condition, an ethanolic solution (0.0056M) of the metal salt cobalt chloride hexahydrate 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 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) cobalt dihydrate refer Scheme 1.



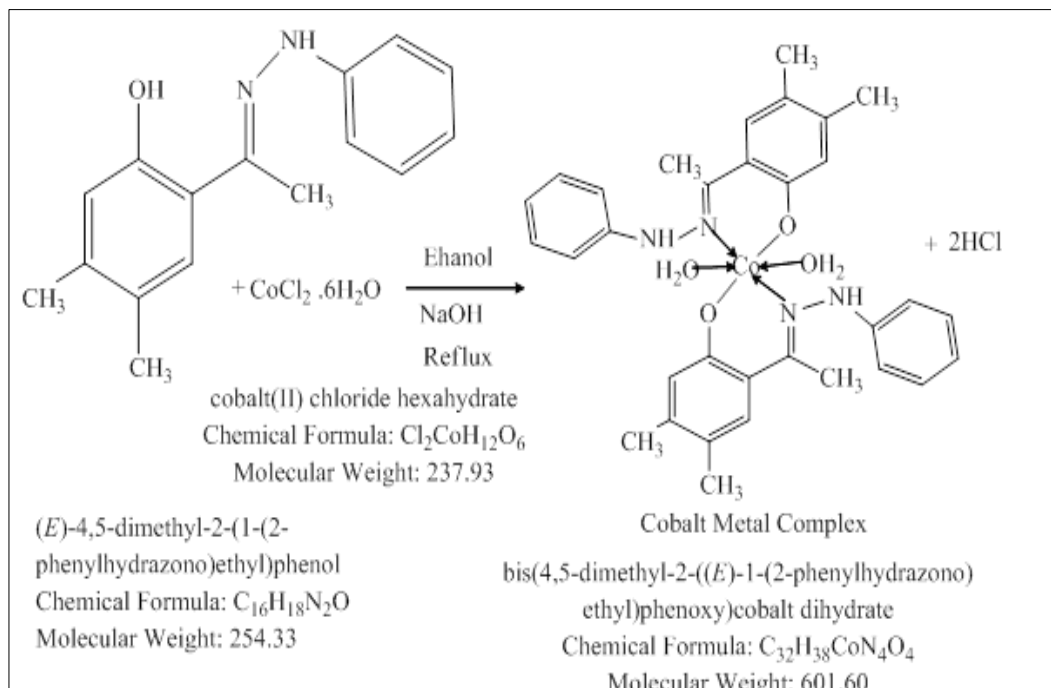
Scheme 1: Reaction scheme preparation of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate.

2.3 Preparation of Co 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)cobalt dihydrate

(E)-4,5-dimethyl-2-(1-(2-phenylhydrazono) ethyl)phenol were synthesized following published procedur [23]. The transition metal complex bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy) cobaltdihydrate were prepared by dissolution of (E)-4,5-dimethyl-2-(1-(2-phenylhydrazono)ethyl)phenol ligand (0.0069M) in ethanol at hot condition, an ethanolic solution (0.0035M) of the metal salt cobalt chloride hexahydrate 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 brown 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) cobaltdihydrate refer Scheme 2.



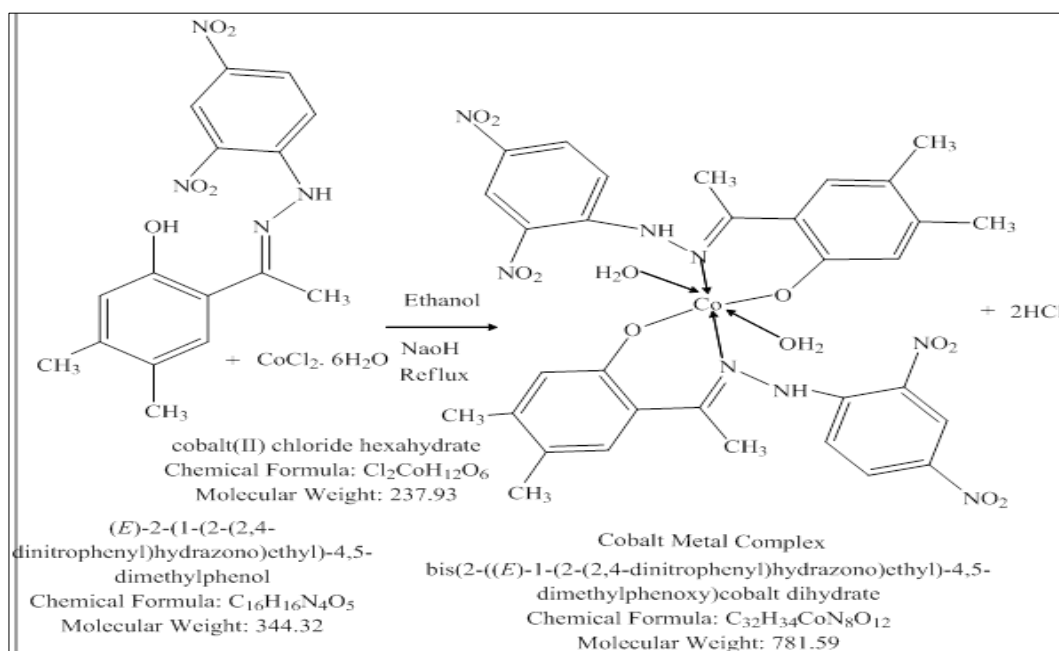
Scheme 2: Reaction scheme preparation of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy)cobaltdihydrate

2.4 Preparation of Co 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)cobalt dihydrate

(E)-2-(1-(2-(2,4-dinitrophenyl) hydrazono)ethyl)-4,5-dimethylphenol were synthesized following published procedure[24]. The transition metal complex bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobaltdihydrate 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 cobalt chloride

hexahydrate 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-dimethyl phenoxy) cobaltdihydrate refer Scheme 3.



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

3. Results and Discussion

The Co-Metal complexes were analyzed by Infrared spectroscopy, Ultraviolet–visible spectroscopy, 1H NMR, Carbon-Hydrogen-Nitrogen-Sulphur elemental analyzer,

Decomposition temperature/Melting point, Molar Conductance, Magnetic susceptibility and Cobalt 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 Co-Metal complexes are mentioned in Table 1.

Table 1: Physical properties

Co-Metal Complex	Chemical Formula and Molecular formula	Color	Solubility	Decomposition Temperature	Yield
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate	C ₂₀ H ₃₀ N ₄ CoO ₄ and 449.41	green	Soluble in methanol, ethanol, Acetonitrile DMSO, DMF Insoluble in water	> 300 °C	86.4%
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) cobaltdihydrate	C ₃₂ H ₃₈ N ₄ CoO ₄ and 601.60	light brown	Soluble in ethanol, methanol, DMSO, DMF Insoluble in water	> 300 °C	82.83 %
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobaltdihydrate	C ₃₂ H ₃₄ N ₈ CoO ₁₂ and 781.59	reddish brown	Soluble in ethanol, methanol, DMSO, DMF Insoluble in water	> 300 °C	79.02 %

3.1 Infrared analysis

The IR spectra of Co-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 Co-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 Cobalt metal complex IR frequency, cm-1

Bond/functional group	Frequency, cm-1		
	bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy) cobalt dihydrate	bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono) ethyl) phenoxy) cobalt dihydrate	bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl phenoxy) cobaltdihydrate
N–H	3282	3315	3305
C–H	3050	3029	3082
C–H stretch aromatics	2921	2917	2915
C=N	1614	1617	1613
C–C	1586	1597	1593
C=C (aromatic ring)	1452	1492	1498
N–O	---	---	1329
C–O	1262	1256	1257
N–N	1111	1144	1113
O–Co	Below 600	Below 600	Below 600

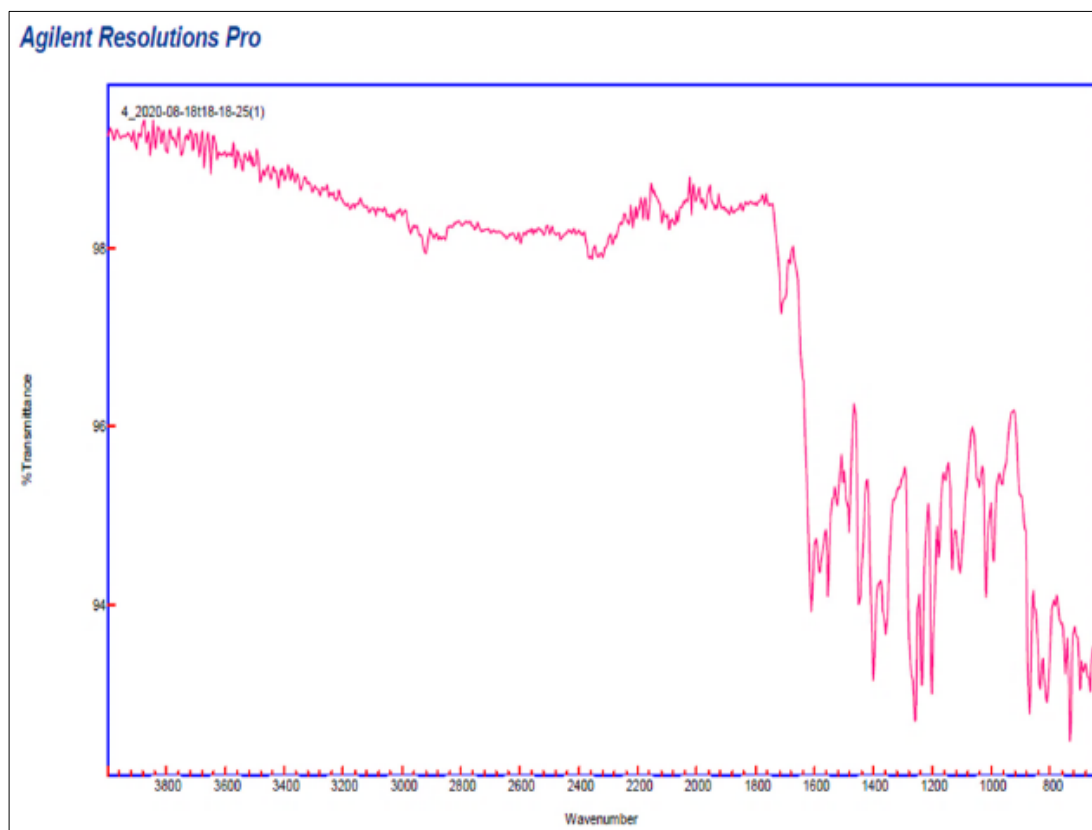


Fig 1: IR Graph of bis (2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)cobalt dihydrate

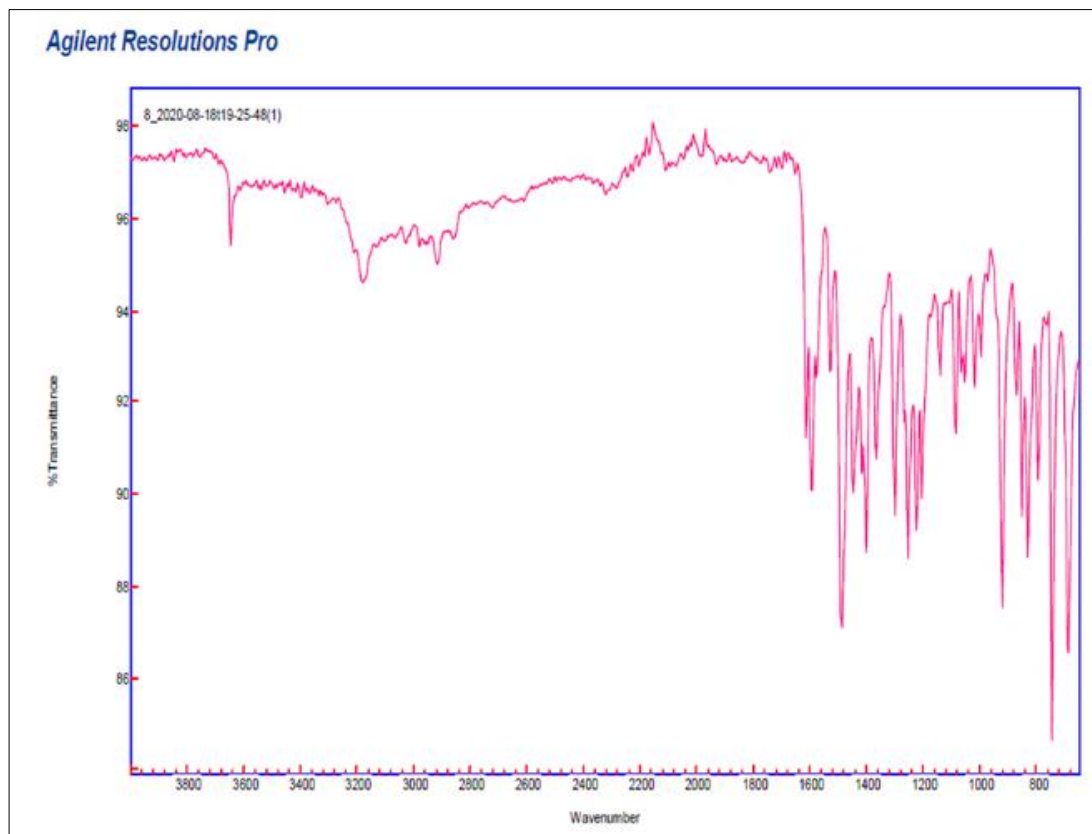


Fig 2: IR Graph of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy) cobalt dihydrate

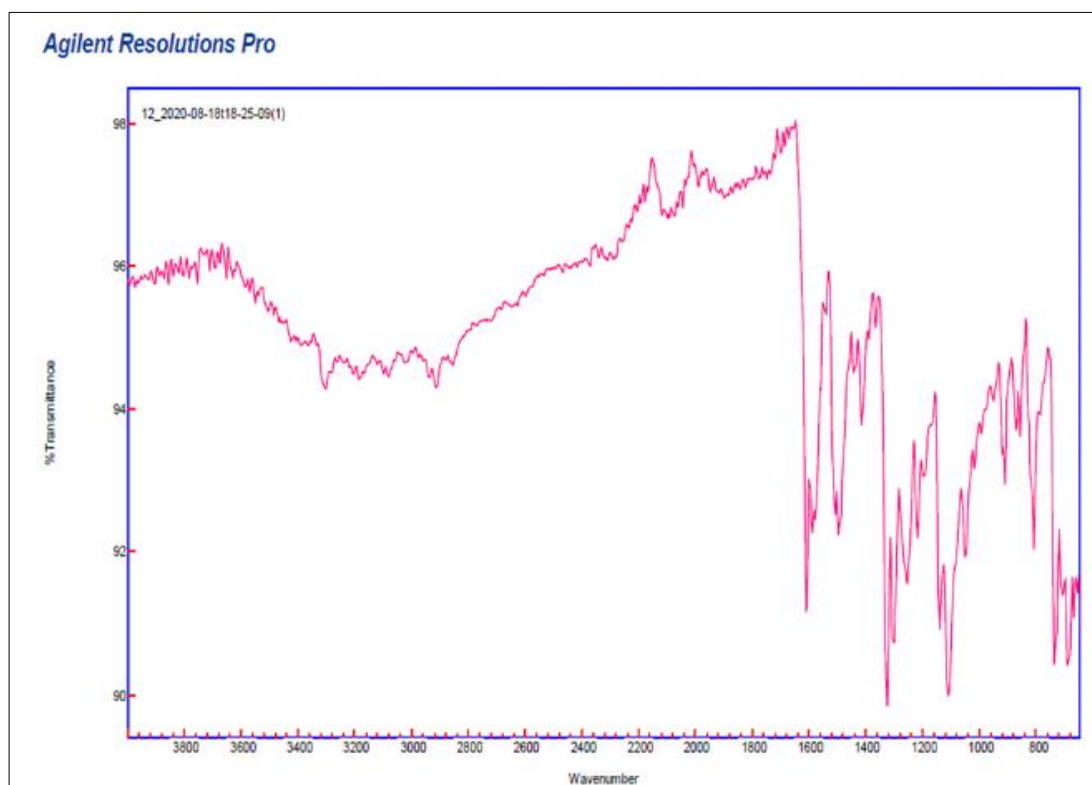


Fig 3: IR Graph of bis (2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl phenoxy) cobaltdihydrate.

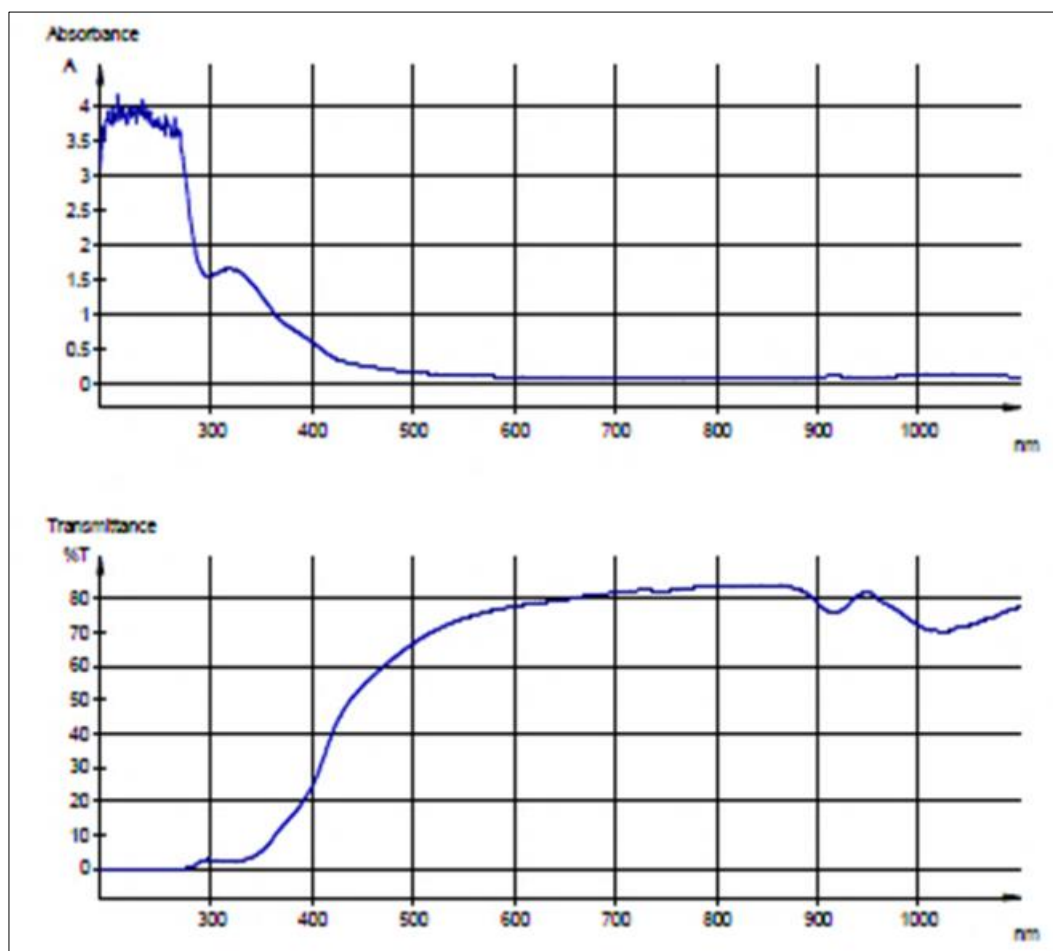
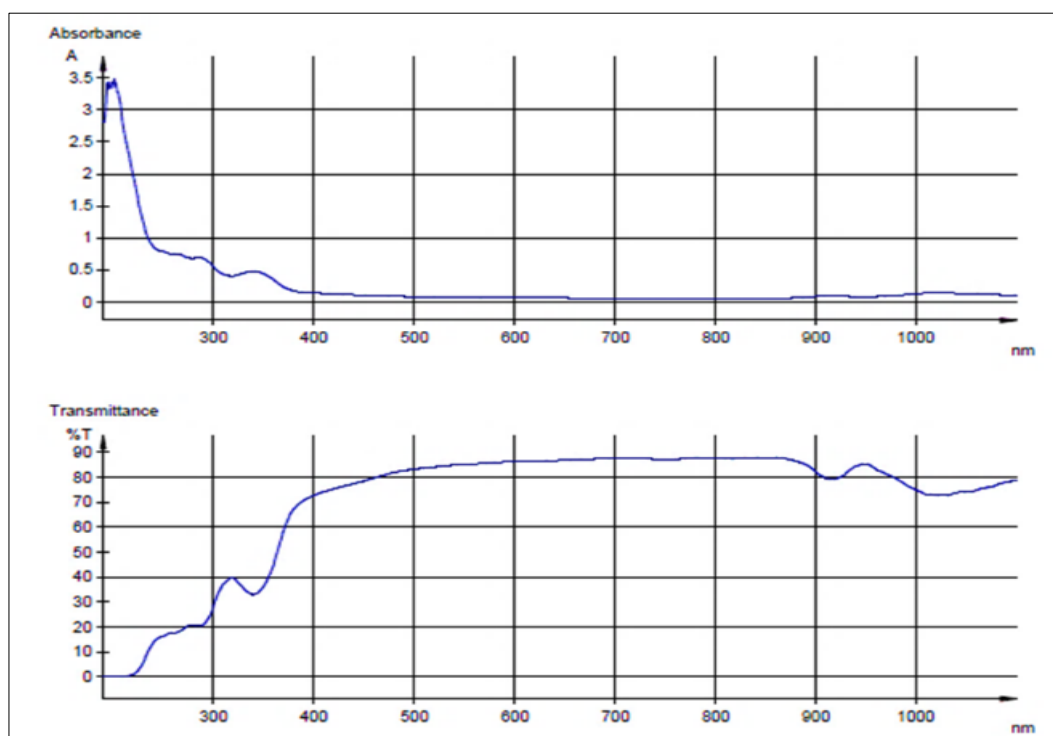
3.2 UV analysis

The UV spectra of the Co-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_{max} (bands maximum in nm) are provided in Table 3.

Table 3: Co-Metal Complex

	Wavelength nm
bis(2-((E)-1-hydrazoneethyl)-4,5-dimethylphenoxy) cobaltdihydrate	254, 298, 524
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) cobalt dihydrate	210, 318, 524
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobaltdihydrate	206, 386, 510

**Fig 4:** UV-Visible Spectra of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) cobaltdihydrate**Fig 5:** UV-Visible Spectra of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy) cobaltdihydrate

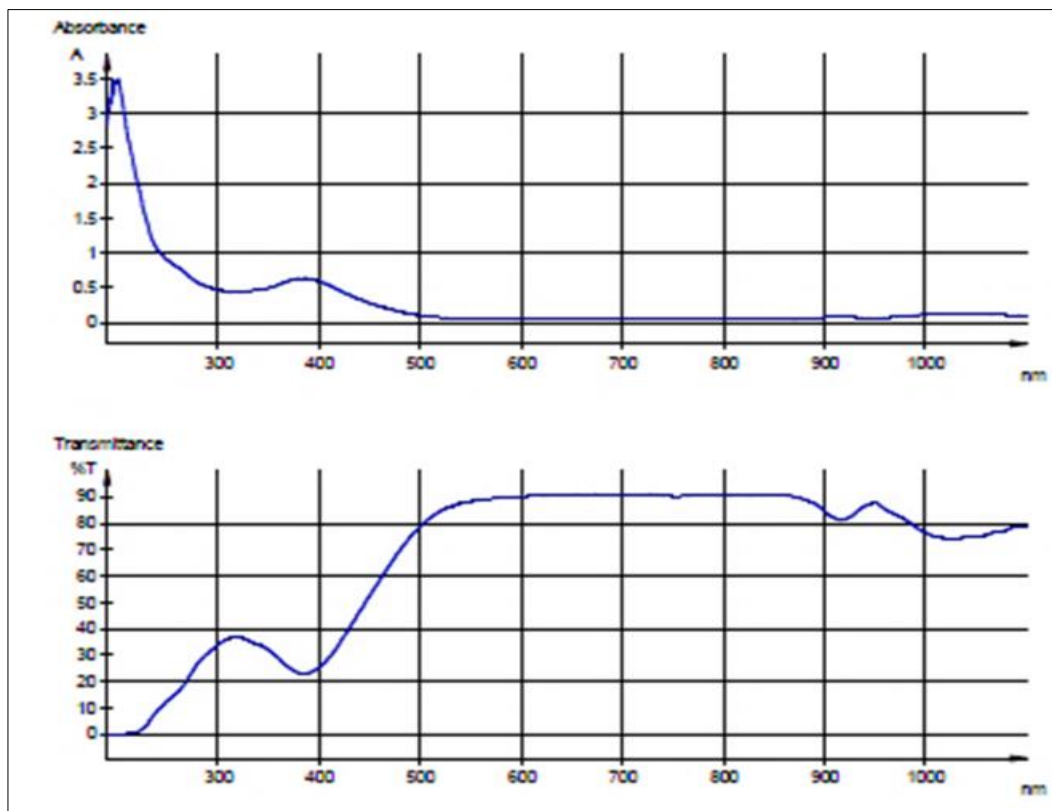


Fig 6: UV-Visible Spectra of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobaltdihydrate

3.3 Elemental analysis (CHNS): Co-Metal complexes

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

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

Table 4: Carbon, Hydrogen, Nitrogen, Cobalt elemental.

Metal Complex	Molecular formula	Molecular weight	Elemental data (required/found)			
			Carbon	Hydrogen	Nitrogen	Cobalt
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobalt dihydrate	C ₂₀ H ₃₀ N ₄ CoO ₄	449.41	54.12 (53.45)	6.96 (6.74)	12.30 (12.47)	14.27 (13.11)
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy) cobaltdihydrate	C ₃₂ H ₃₈ N ₄ CoO ₄	601.60	63.75 (63.88)	6.06 (6.38)	9.46 (9.32)	10.28 (9.80)
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobalt dihydrate	C ₃₂ H ₃₄ N ₈ CoO ₁₂	781.59	49.86 (49.17)	4.55 (4.39)	14.68 (14.34)	7.67 (7.54)

3.4 Magnetic Susceptibility

The magnetic moment (μ_{eff}) values at room temperature for Co (II) complexes complexes was in the range of 4.0 to 5.2

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

Table 5: Magnetic Susceptibility

Co-Metal complex	μ_{eff} value
Bis (2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate	4.37
Bis (4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy) cobaltdihydrate	4.72
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethylphenoxy) cobalt dihydrate	4.96

3.5 Molar Conductance (mho.cm².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

Co-Metal complex	Molar Conductance mhos cm ² mol ⁻¹
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate	0.107
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy)cobaltdihydrate	0.080
bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono) ethyl)-4,5-dimethylphenoxy) cobaltdihydrate	0.32

3.6 $^1\text{H NMR}$

I tried all metal-complexes $^1\text{H NMR}$ 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 [25].

- 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 $^1\text{H NMR}$ Spectra of metal complexes are shown in Figure 7, 8, 9

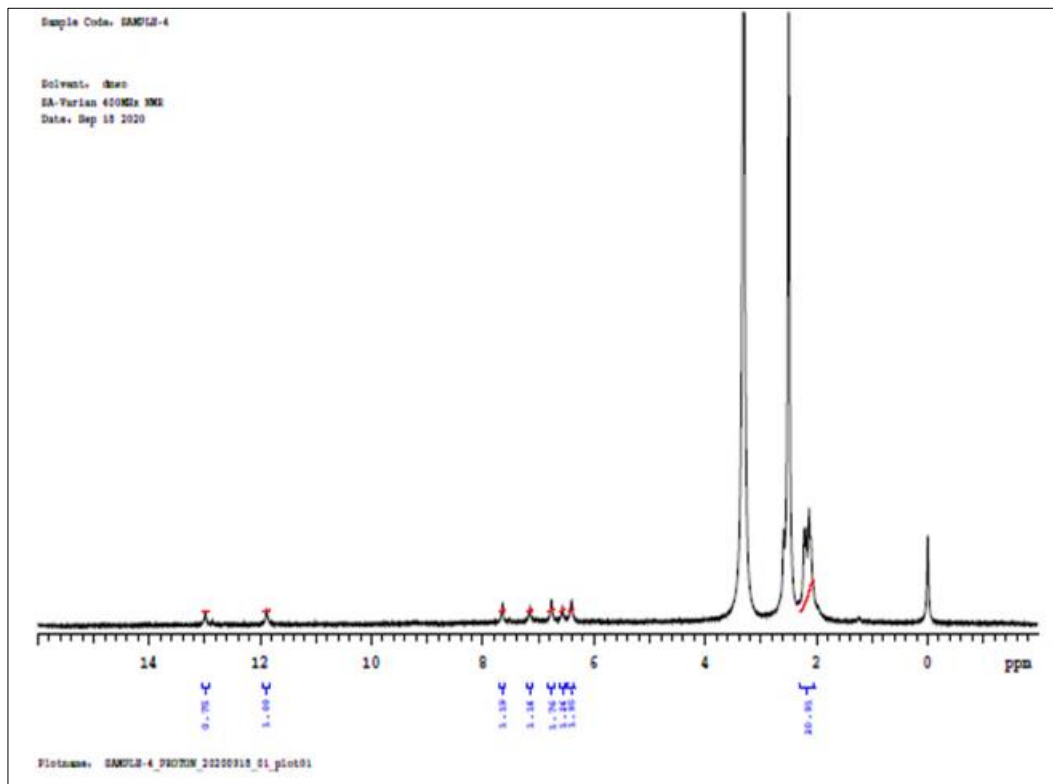


Fig 7: $^1\text{H NMR}$ of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy) cobaltdihydrate

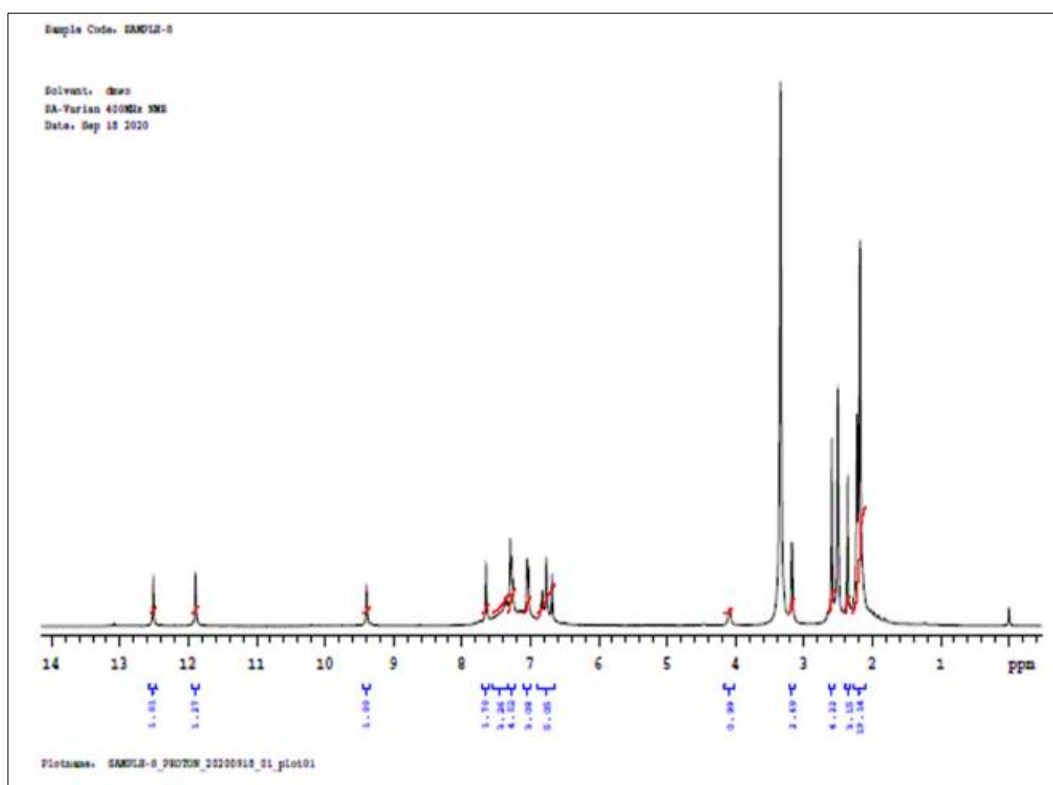


Fig 8: $^1\text{H NMR}$ of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl) phenoxy)cobalt dihydrate

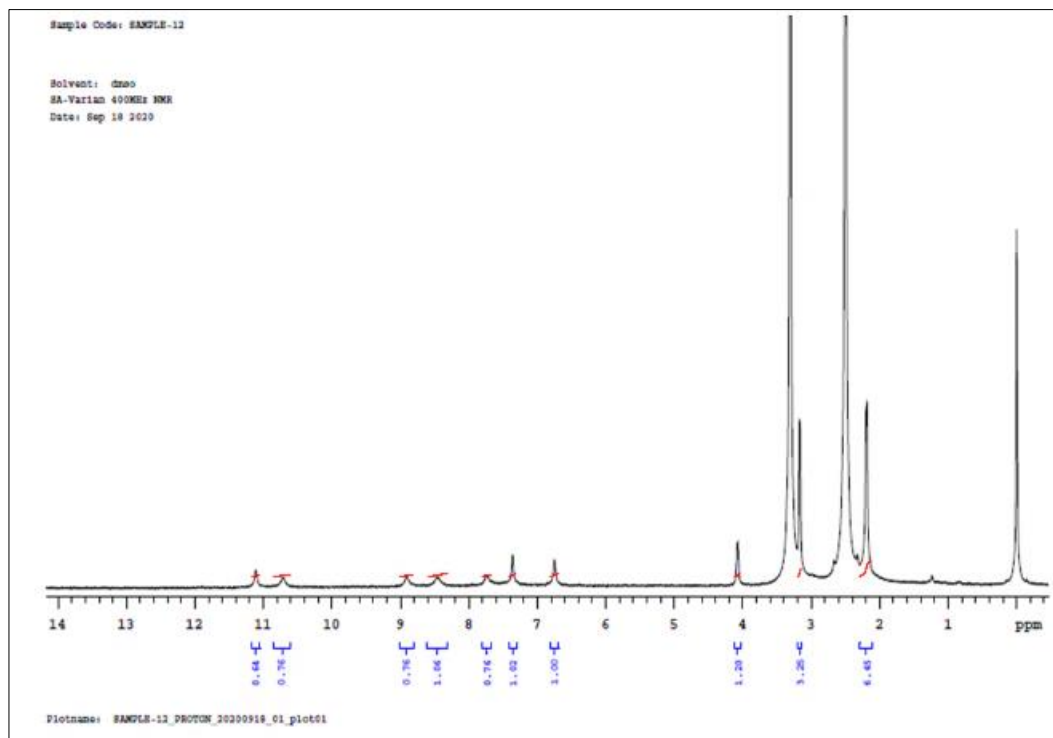


Fig 9: ^1H NMR of bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono) ethyl)-4,5-dimethyl phenoxy) cobaltdihydrate

4. Conclusion

All Cobalt 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 Co (II) complexes and The conductivity data show that each one these complexes are non-electrolytes.

5. Acknowledgements

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