IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429
Volume 6, Issue 1, November 2026
Impact Factor: 8.2

Heterobinuclear Complex Consisting Ni(II) and Rb(I) Derived from Rubidium Salt of Acetyl Salicylic Acid (ASPIRIN) and Nickel(II) Schiff Base N,N'–1,2–ethylenebis(o-hydroxyacetophenoniminato) Nickel(II) as Ligand : Synthesis and Characterization

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Abstract : *In the present research work, a novel hetero-binuclear complex has been synthesized by the complexation reaction between Ni (II) Schiff base N,N'–1,2–ethylenebis(o-hydroxyacetophenoniminato) Nickel(II) and Rubidium salt of Acetyl Salicylic acid (ASPIRIN). The adduct is stable crystalline solid of harvest golden colour, non-electrolytic and diamagnetic in nature, and yield is good. The structure of the synthesized hetero-binuclear complex has been proposed on the basis of investigative results obtained from elemental analysis, solubility, Magnetic moment and Molar conductance measurement, FT-IR spectrum, and Electronic Absorption spectrum. The results show that in adduct Ni (II) Schiff base N,N'–1,2–ethylenebis(ortho-hydroxy acetophenoniminato)Nickel(II) acts as "Bidentate Ligand". The molecular geometry of the synthesized hetero-binuclear complex is found to be square planar about Ni^{2+} ion and there is no change in geometry about Ni^{2+} ion during adduct formation from nickel Schiff base complex. The Formula of the synthesized complex is given by $[M_a\ L\ M_b\ L'].2H_2O$ Where $M_a = Ni^{2+}$, $M_b = Rb^+$, L = N,N'-1,2-ethylenebis(ortho-hydroxy acetophenoniminato) dianion, and L' = Acetyl salicylate.*

Keywords: Hetero-binuclear complex, Square planar geometry, Rubidium acetyl salicylate, Adduct, Schiff base, N,N'–1,2–ethylenebis(ortho-hydroxyacetophenoniminato)Nickel(II), Diamagnetic

1. INTRODUCTION

Due to wide area of applicability of metal complexes such as in catalytic activity, chemical analysis, biological applications including anti-microbial, anti-fungal, anti-HIV, anti-tumour, insecticidal, and in vitro cytotoxic activities as well as DNA binders [1-5], synthesis of new metal complexes has become one of the most active research areas. Salen-type Schiff bases derived from di-primary amine and Salicylaldehyde or its derivatives, act as tetradentate N_2O_2 type ligands. These ligands have the ability to bind two metal atoms adjacent to each other. They coordinate strongly with a wide range of transition and lanthanide metals forming binuclear metal complexes [6-12]. Two distinct metals in hetero-binuclear complexes produce 'Cooperative Synergy Effect' where the distinct physical and chemical properties of each metal complement the other and stems an unique property that is entirely unachievable by either metal alone or by a simple physical mixture of the two [13]. In recent years, a number of hetero-binuclear complexes have been

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DOI: 10.48175/IJARSCT-40543

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synthesized and their applications have been identified. However, a few works have been done on synthesis of hetero-binuclear complexes containing two metal centres – one of transition metal and other of alkali metal. Only a few hetero-binuclear complexes containing Rubidium and Caesium have been reported [14-18]. The present work describes the synthesis of a novel hetero-binuclear complex derived from Rubidium Salt of Acetyl Salicylic Acid (ASPIRIN) and Nickel (II) Schiff Base N,N'–1,2–ethylenebis(o-hydroxy acetophenoniminato)Nickel(II). The adduct yield is good and is crystalline solid. The synthesized complex has been characterized using various physico-chemical techniques such as elemental analysis, melting point determination, solubility determination, magnetic moment measurement, molar conductance measurement, FT-IR Spectroscopy, and UV-Visible Spectroscopy. Investigative results show molecular geometry of the synthesized hetero-binuclear complex to be Square planar about Ni^{2+} ion.

II. MATERIALS AND METHODS

All chemicals used in the present research work were of A.R. Grade. ESICO International Auto Melting Point Apparatus 1934 was used for the determination of melting point. Carbon, Hydrogen, Nitrogen, and Oxygen were estimated by Elemental Analyser EUROVECTOR EA3000 where as Rubidium and Nickel were estimated by using ICP-MS of Agilent Technologies model no.7800. Estimation of Rubidium was further carried out gravimetrically by Sodium tetraphenyl borate method and of Nickel further carried out volumetrically by EDTA titration method in the Departmental Research Laboratory. Vibrating Sample Magnetometer (VSM) was used for the measurement of magnetic moment. Molar conductance of 10^{-3}M solution of the synthesized complex in DMF was measured by Conductometer. FT-IR spectrum of the synthesized sample was recorded on Thermo Nicolet Avatar 370 in the range 4000 – 400 Cm-1 by KBr beam splitter. Electronic absorption spectrum of 10^{-4}M solution of the complex was recorded between 200 -900 nm by Shimadzu UV-Visible Spectrometer Model UV-2450 using UV Probe 2.32 software.

2.1 Synthesis of Nickel (II) Schiff base N,N'–1,2–ethylenebis(o-hydroxyaceto phenoniminato)Nickel(II) [NiEohacp]

Equimolar ethanolic solutions of N,N'-1,2-bis(ortho-hydroxyacetophenonidene) ethylenediamine and nickel acetate tetrahydrate were prepared. In nickel acetate tetrahydrate solution, Schiff base solution was added slowly with constant stirring. The resulting solution was Warmed below 40^0C for 20-25 minutes. Bright yellow coloured crystalline solid precipitated out. Precipitate was filtered, washed with little ethanol, dried and kept in desiccator over anhydrous $CaCl_2$.

2.2 Synthesis of Hetero-binuclear complex [NiEohacpRbAp] from ickel (II) Schiff base [NiEohacp] and Rubidium Acetyl Acetate (RbAp)

Equimolar ethanolic solutions of N,N'–1,2–ethylenebis(o-hydroxyaceto phenoniminato)Nickel(II) and Rubidium acetyl acetate were mixed together and refluxed for 25 minutes at about 40^0C and cooled to ice temperature when the complex was separated out. Precipitate was filtered and washed with little ethanol and dried. Finally, synthesized complex was kept in a desiccator over anhydrous $CaCl_2$.

III. RESULTS AND DISCUSSIONS

The adduct is coloured crystalline solid, non-hygroscopic and stable at room temperature. Physical properties and elemental analysis results of the adduct have been summarised in TABLE-1. Synthesized hetero-binuclear complex, [NiEohacpRbAp] is insoluble in water, ethanol, benzene, petroleum ether solvents but soluble in acetone, DMF and DMSO. Effective magnetic moment of the adduct [NiEohacpRbAp] is found to be zero. This suggests diamagnetic nature of the synthesized complex [NiEohacpRbAp]. Molar conductance of 10^{-3}M solution of the synthesized complex in DMF at $(20\pm0.5)^0$C is found to be 11.90 $S.Cm^2.mole^{-1}$. This indicates non-electrolytic nature of the adduct [19,20].

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TABLE-1: Physical Properties and Analytical Data

	Yield (%)	State	Colour	M.P. (^{0}C)	Effective Magnetic Moment, μ_{eff} (B.M.)	Molar Conductance (S.Cm^2. mole^{-1})
[NiEohacpRbAp]	67	Crystalline Solid	Harvest Gold	225	0	11.90
	ELEMENTAL ANALYSIS Observed (Calculated)					
	C	H	N	O	Ni	Rb
	49.28 (49.56)	4.41 (4.44)	4.17 (4.28)	19.63 (19.58)	9.35 (8.98)	12.97 (13.08)

Empirical Formula as per analytical estimation = $NiRbC_{27}H_{29}N_2O_8$

The prominent I.R. absorption band positions in FT-IR Spectrum [Figure.1] of synthesized complex [NiEohacpRbAp] is given in TABLE – 2. FT-IR spectrum analysis reveals a very strong band at 1595 Cm^{-1} which may be assigned for $\nu_{C=N}$ stretching [21].

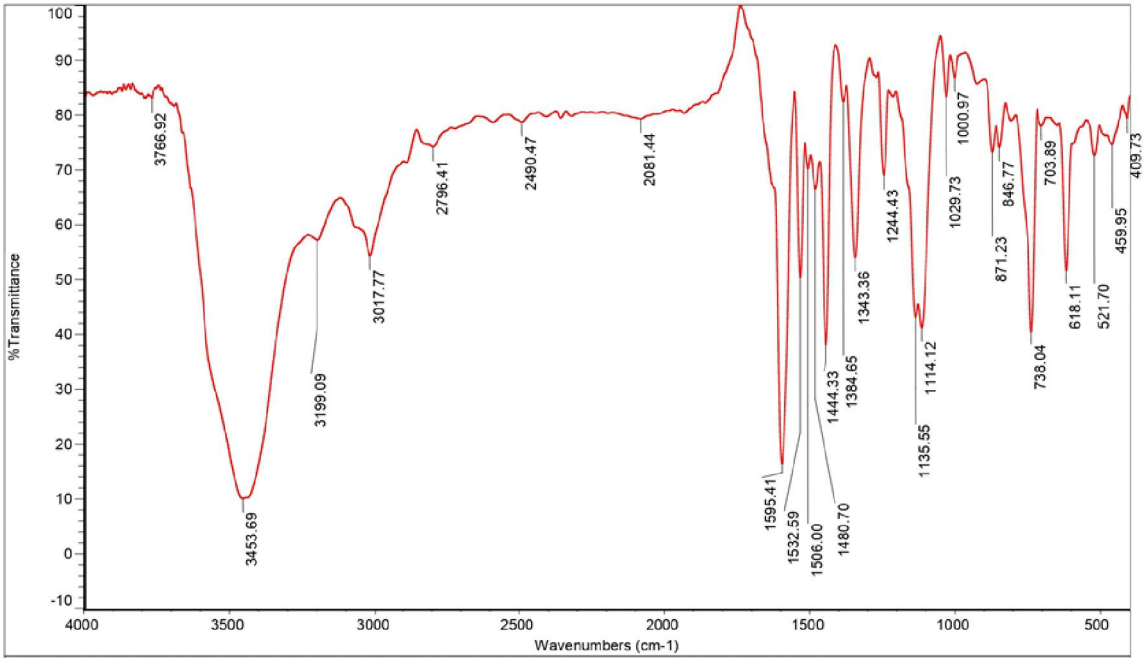


Figure. 1: FT-IR Spectrum of the Synthesized Hetero-binuclear Complex [NiEohacpRbAp]

Many workers in various Schiff bases have reported $\nu_{C=N}$ stretching band for uncoordinated azomethine (C=N) in the range 1650 – 1690 Cm^{-1} [22]. The $\nu_{C=N}$ stretching band in [NiEohacp] FT-IR spectrum appears at 1600 Cm^{-1}. The shift of the absorption band of $\nu_{C=N}$ stretching towards lower wave number indicates coordination of the imine nitrogen to the Ni^{2+} ion during [NiEohacp] complex formation [22,23]. This is further substantiated by the presence of a new band at 618 Cm^{-1} assignable to ν_{N-M} stretching vibrations. Sharp, medium to weak bands in the range 500 – 700 Cm^{-1} have been reported for nitrogen-metal, ν_{N-M} stretching vibrations [24]. The absence of sharp characteristic absorption band in the region 3600 – 3700 Cm^{-1} due to phenolic O – H stretching vibrations and also the absence of broad band

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in the region $3200 - 3300\ Cm^{-1}$ due to intermolecular hydrogen bond, indicates deprotonation during formation of nickel complex of Schiff base [NiEohacp]. Absorption band position of certain chemical bonds of interest have been identified in FT-IR Spectrum of Aspirin, [NiEohacp] and Synthesized complex [NiEohacpRbAp], and is summarized in TABLE – 3.

TABLE. 2: Prominent I.R. absorption band positions in FT-IR Spectrum of synthesized complex [NiEohacpRbAp]

[NiEohacpRbAp]	
Absorption Band Position (in cm^{-1})	Assignment
3453 br	Water of crystallisation
3199 w, 3017 w	ν(Ar-H) Str
1595 vs	ν(C=O) Str. ester, ν(C=O) Str. carboxylic, ν(C=C) Str. aromatic, ν(C=N) Str.
1532 s	ν(C=C) Str. aromatic
1506 w, 1480 w	ν(C=C) Str. aromatic
1444 s	ν (C – O) Str. carboxylic, ν(C-H) Bending aromatic
1384 s	ν(C-H) def. in CH_2/CH_3, ν (C-N) Str.
1343 vs	ν (C-O) Str. ester, ν (C-O) Str. phenolic
1244 s	ν(C – H) skeletal vib., ν(C – CH_3) rocking vibration
1135 s	In plane bending of the ring C – H bonds
1114 s	ν (C – N) Bending
1029 m, 1000 m	In plane bending of the ring C – H bonds
925 w, 871 m, 846 m	Out – of – plane bending of the ring C – H bonds
738 s	ν(C – C) def. ortho-disubstituted benzene
703 w	Out of the plane ring bending of substituted benzene
618 s	ν(N – M) Str.
521 m, 459 w, 409 w	ν(O – M) Str.

Br = Broad, s = Strong, vs = Very strong, m = Medium, w = Weak

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TABLE – 3: Absorption band position of certain chemical bonds of interest in FT-IR Spectrum of Aspirin, [NiEohacp] and Synthesized complex [NiEohacpRbAp]

Chemical Bond	Mode of Vibration	Absorption Band Position (in cm^{-1})		
		Aspirin	[NiEohacp]	[NiEohacpRbAp]
O – H Carboxylic	Stretching	2649br, 2821 br	NA	NP
C – O Carboxylic	Stretching	1313 s	NA	1444 s
C = O Carboxylic	Stretching	1591 s	NA	1595 vs
C = O Ester	Stretching	1684 s	NA	1595 vs
C – O Ester	Stretching	1265 s	NA	1343 vs
C – O Phenolic	Stretching	NA	1244 s	1343 vs

NA = Not Applicable, NP = Not Present, br = broad,vs = very strong, s = strong

A sharp ir absorption band appears at 1343 Cm^{-1} which may be assigned for phenolic C - O stretching vibrations. However, phenolic C – O stretching vibrations absorption band in complex [NiEohacp] appears at 1244 Cm^{-1}. A strong absorption band in region $1200 - 1350\ Cm^{-1}$ has been reported for phenolic C – O stretching mode by research workers [25,26,27]. Shifting of phenolic C – O stretching vibration towards higher wave number indicates coordination of phenolic oxygen atom to Rb^{+} ion during hetero-binuclear complex [NiEohacpRbAp] formation from ligand [NiEohacp]. This fact is further substantiated by the presence of oxygen – metal bond stretching vibration (ν_{O-M}) bands in the region 409 – 521 Cm^{-1}.

Absence of absorption band of carboxylic O – H bond (Str) in [NiEohacpRbAp] indicates deprotonation of carboxylic O – H bond during Rubidium salt formation, and shift of absorption band of carboxylic C – O (Str) towards higher wave number in [NiEohacpRbAp] as compared to aspirin further substantiates the fact of deprotonation of carboxylic O – H and conversion of it into O – Rb. Comparison of absorption band position of carboxylic C = O (Str) in FT-IR spectra of Aspirin and [NiEohacpRbAp], we find no major shift in wave number which indicates non-involvement of oxygen atom lone pair of carboxylic C = O in coordination during complex [NiEohacpRbAp] formation. However, slight shift of absorption band of ester C = O (Str) towards lower wave number from aspirin to [NiEohacpRbAp] indicates presence of hydrogen bonding between ester C = O group and H_2O (water of crystallisation). On comparison of FT-IR spectrum of Aspirin and [NiEohacpRbAp], we find major shift in absorption band of ester C – O (Str) towards higher wave number which indicates coordination of oxygen atom of ester C – O with Rubidium during complex [NiEohacpRbAp] formation. This fact is supported by the presence of oxygen-metal bond stretching vibration (ν_{O-M}) bands in the region 409 – 521Cm^{-1} . A broad band at 3453 Cm^{-1} indicates presence of H_2O as water of crystallisation in [NiEohacpRbAp] molecule.

Band positions in electronic spectrum of the complex [NiEohacpRbAp] is summarised in TABLE – 4.

TABLE – 4: Band Positions in Electronic Absorption Spectrum of [NiEohacpRbAp]

[NiEohacpRbAp]	
Absorption Band Position (in cm^{-1})	Assignment
Broad band ranging 256 – 472 nm with a peak at λ_{max} = 409 nm and a shoulder at 348 nm	(1) Intraligand transitions: (a) Benzene Chromophore $\pi \longrightarrow \pi^{*}$ transition (b) Azomethine Chromophore

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	$\pi \longrightarrow \pi^*$ transition (c) Co-ordinated azomethine n $\longrightarrow \pi^*$ transition (2) Ligand to Metal Charge Transfer (LMCT) (3) d $\rightarrow$ d transition
Band with a peak at λ_{max} = 544nm	d $\rightarrow$ d transition

Appearance of no bands between 500 – 1000 nm excludes possibility of square pyramidal and octahedral geometry around Ni^{2+} ion. Further appearance of no band above 1000 nm excludes possibility of tetrahedral geometry around Ni^{2+} ion. For a square planar geometry around Ni^{2+} ion there must be appearance of two bands in region 420 – 500 nm & 500 – 600 nm assignable to $^1A_{1g} \rightarrow {}^1A_{2g}$ and $^1A_{1g} \rightarrow {}^1B_{1g}$ transitions, respectively. In this case a band appears at 544nm corresponding to $^1A_{1g} \rightarrow {}^1B_{1g}$ d – d transition and another band of $^1A_{1g} \rightarrow {}^1A_{2g}$ d-d transition has been obscured by the CT band at that region [21,28]. Hence, in the complex [NiEohacpRbAp] there is square planar geometry around Ni^{2+} ion. This is further supported by zero magnetic moment of complex [NiEohacpRbAp] which indicates its diamagnetic nature with no unpaired electron, and hence a low spin complex. Empirical formula suggests two water of crystallisation molecules present in the complex molecule, [NiEohacpRbAp].$2H_2O$.

On the basis of above investigation results and discussions, following structure and general formula of the synthesized hetero-binuclear complex is proposed:

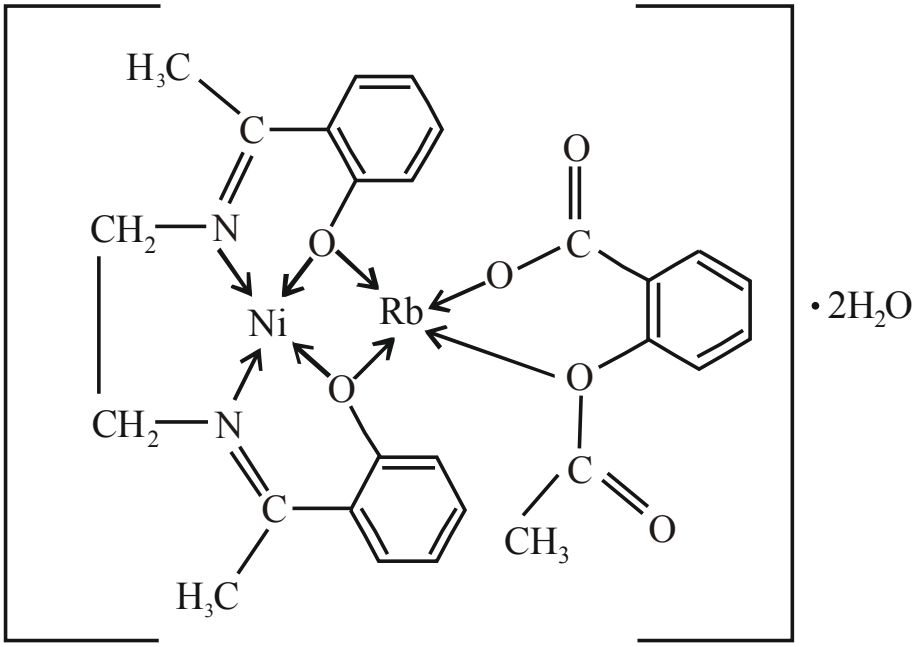


Figure. 2: Proposed Structure of Synthesized Hetero-binuclear Complex

A general formula of the synthesized complex is given as [Ma L M_b L']$.2H_2O$

Where $M_a = Ni^{2+}$, $M_b = Rb^+$, L = N,N'-1,2-ethylenebis(o-hydroxyaceto phenoniminato) dianion and L' = Acetyl salicylate.

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IV. CONCLUSION

The synthesized hetero-binuclear complex is an adduct of Rubidium Acetyl Salicylate and N,N'–1,2–ethylenebis(o-hydroxyacetophenoniminato) Nickel(II) in which Nickel (II) Schiff base acts as bidentate ligand. The molecular geometry of the synthesized complex is Square planar about Ni^{2+} ion, and there is no change in geometry about Ni^{2+} ion during adduct formation from nickel Schiff base complex [NiEohacp]. The adduct is non-electrolytic and diamagnetic in nature.

V. ACKNOWLEDGEMENT

Author is grateful to Civil Engineering Department IIT Patna, SAIF CSIR-CDRI LUCKNOW, SAIF STIC COCHIN and SAIC TEZPUR UNIVERSITY for doing elemental analysis, UV-Visible, FT-IR spectral analysis and magnetic measurements.

VI. CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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