A COMPENDIUM OF ORTHO AND META SUBSTITUTED HYDRAZONE LIGANDS AND THEIR METAL COMPLEXES

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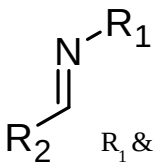
ABSTRACT

A compendium of N-substituted hydrazone salts as well as transition metal developments remained synthesized by benzaldehyde and its disubstituted isomeric derivatives i.e. 3-hydroxybenzaldehyde and 2-hydroxybenzaldehyde (Salicylaldehyde). The bidentate ligands synthesized 2-[(Z)-(2-phenylhydrazinylidene) methyl] phenol, (1*E*)-1-benzylidene-2-phenylhydrazine, Hydroxy {4-[(Z)-(-hydroxybenzylidene) amino] phenyl}, 3-[(*Z*)-(2-phenylhydrazinylidene) methyl] phenol and their Cd (II) and Zn (II) developments remained also organized. Completely the respective products were characterized as well as evaluated by means of analytical parameters and various spectral techniques like IR spectroscopy, UV visible spectroscopy, anti-oxidant and anti-bacterial assay as the Schiff bases were analysed to be very agile products which refers towards the novelty and main intent of this synthetic research work, as featured study of isomeric Schiff bases to get the proficient sequels for anti-bacterial and anti-oxidant values.

Keywords: Schiff bases, transition metal complexes, synthesis, Salicylaldehyde, IR spectroscopy, UV visible spectroscopy, anti-bacterial, anti-oxidant, and 3-hydroxybenzaldehyde.

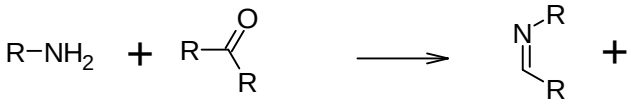
INTRODUCTION

Schiff-bases have C=N bond as well as remain appreciated to be multi-purpose dynamic agents in numerous field as well as viewing comprehensive range for organic possessions such as antifungal activities, antibacterial properties (R. R. Gupta, Kumar, & Gupta, 1998).A typical Schiff-Base is a *N*-analogue of carbon-based complex in which C=O set remains side chain by RHC=N-R group (Comey, 1896) and these remained first reportable by Hugo Schiff (a German chemist, Noble Laureate) in 1864 (Taggi et al., 2002).The cooperative portion of complexes remains that the imine set (Azomethine) (Cimerman, Miljanić, & Galić, 2000), wherever R as well as R_1 group, aryl, cyclo group or heterocyclic groups, which can be multifariously side chain. All these complexes remain named Anils or Imines or azomethine as shown in scheme (Elmali, 2000). These remain accurately concentration creation of RCHO or ROR by main amines as exposed in figure (Hine & Yeh, 1967).



R_1 & R_2 = Aryl or Acyl group

Figure *General formula of Schiff-base (Hine & Yeh, 1967)*



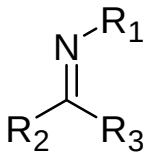
primary amine carbonyl compound Schiff base

Scheme *General man-made process of Schiff-base (Elmali, 2000)*

Schiff-bases typically remain metallic, tridentate or tetradentate chelate salts as well as manufacture stable developments sideways with the metallic ions. Their physical as well as chemical properties in diverse fields such as preparatory uses, identification procedures, protection procedures as well as also purpose dealings of carbon-based complex, de-contamination of Carbonyl-compounds as well as also amino-based complexes of those compounds in difficult process remain calculated by different organizations (Xavier & Srividhya, 2014; Yoshizawa et al., 2005).

Schiff base salts

General formula of Schiff-bases is $R_1N=CR_2R_3$ as shown in figure, in which R_1 can be alkyl or aryl group. The groups attached remain liable for stabilizing the Schiff-bases.



R_1= Aryl or alkyl group
R_2&R_3= Heterocyclic group

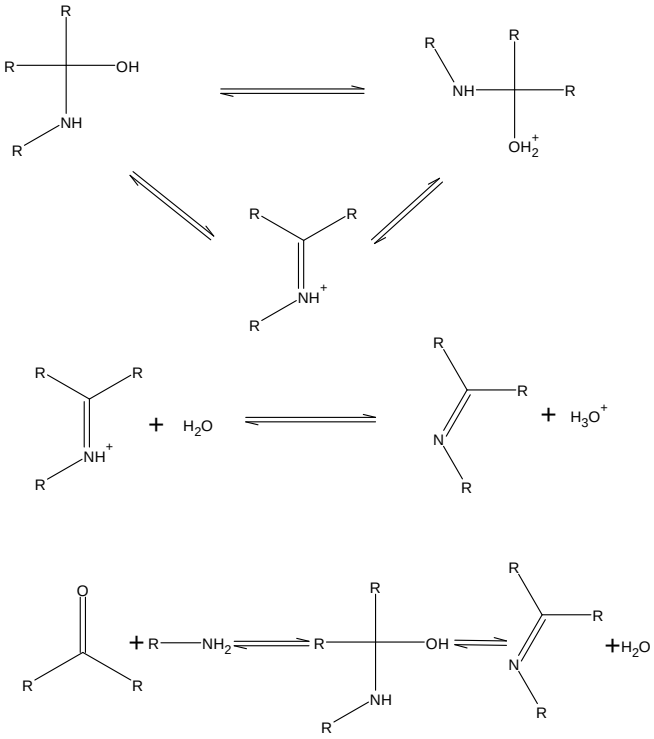
Figure Imine group (Al Zoubi, 2013)

Schiff base Metal Complexes

New anti-microbial representatives meanwhile the pathogens producing infectious syndromes progress confrontation to the existing anti-biotics (Ibrahim, Mohamed, & Refat, 2014; Mohanan & Murukan, 2005; Sobola, Watkins, & Van Brecht, 2014). Efficiency of Schiff-base remains improved in salt form. Metal ions accept lone-pair of e^- from Nitrogen molecule of imine bond. Salicylaldehyde as well as certain hetero-cyclic rings act as a basis of another functional-group as well as of *N*-atom correspondingly (K. Gupta & Sutar, 2008; K. Gupta, Sutar, & Lin, 2009).

General production of Schiff-bases

Production of Schiff-bases remains inverted because certain applications remain as follows (i) acid catalysis (ii) base catalysis or (iii) heating. The artificial mechanism of Schiff-bases with amines as well as carbonyl complexes continuously procedures an adduct named Carbinolamine as the Nu addition remains not tracked (Stehlik, 2009). The development of a Schiff-base from an RCHO (or) ROR remains a reversible process as well as usually taking under H^+ or OH^- catalysis (Baldaniya & Patel, 2009).



Scheme *General synthetic framework (Schiff, 1864)*

1. METHOD

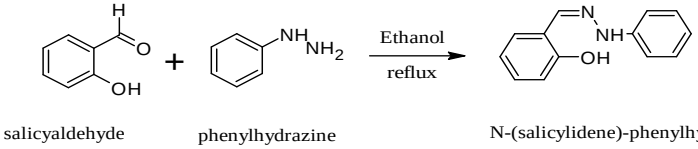
Chemicals and Apparatus

Salicylaldehyde, Benzaldehyde, p-nitroaniline, Phenyl hydrazine, $CdCl_2$ as well as $ZnCl_2$. CH_3CH_2OH, CH_3OH, Diethyl ether, Ethyl acetate, Petroleum ether, benzene, Distilled water. Hot plate with magnetic stirrer, condenser, desiccator, round bottom flask, beakers, thermometer, measuring cylinder, pipette, pipette pump, separating funnel, TLC plates, silica gel, aluminium foil. Weighing balance, Gallen Kamp, ALPHA FT-IR Spectrometer, Conductometer, UV/Visible spectrophotometer.

Preparation of salts:

Synthesis of *N*-(salicylidene)-phenyl hydrazine [R]

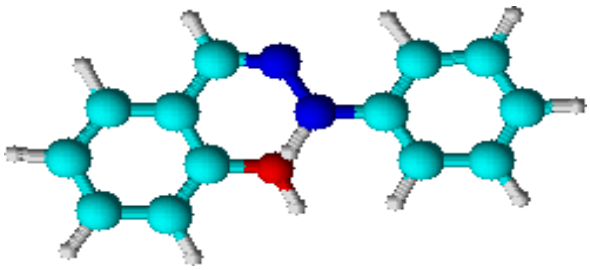
Melted Salicylaldehyde (2 mL) as well as phenyl hydrazine 2 mL in 20 mL of CH_3CH_2OH as well as occupied in round lowest flask as well as refluxed for 2-3 hours. The development of process remained check done thin layer chrmatography plates after each half hour. After the conclusion of process, the warm mixture remained transferred in ice-cold H_2O. Then the creation remained sieved below suction, washed by condensed CH_3CH_2OH as well as dehydrated in desiccator below reduced pressure over an-hydrous $CaCl_2$. The product got remained yellow coloured crystalline solid, Yield 70-75%, M.P. 185 ºC (Iftikhar et al., 2018).



salicyaldehyde phenylhydrazine N-(salicylidene)-phenylhydrazine

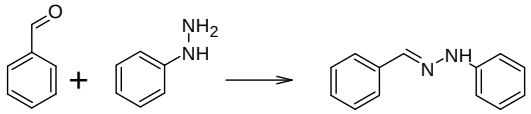
Scheme *Synthesis of salt [R]*

Figure *Three dimensional structure of salt [R]*



Synthesis of *N*-(benzylidene)-phenyl hydrazine [S]

Benzaldehyde (2 mL) as well as phenyl-hydrazine 1 mL remained melted in a round bottommost flask comprising 20 mL of CH_3CH_2OH mixture as well as then heated as well as stirred for 3-4 hours on the warm plate by reflux. The conclusion of process remained check over thin layer chromatography plates after half hour. The product moulded in process solvent remained occupied in beaker, sieved as well as eroded with di-ethyl ether as well as CH_3CH_2OH to get the pure product. The product remained dehydrated in desiccator below reduced pressure over an-hydrous $CaCl_2$. The subsequent product remained white crystalline solid, Yield 70% and M.P. 154 ºC (Mohapatra et al., 2018).



benzaldehyde phenylhydrazine (1*E*)-1-benzylidene-2-phenylhydrazine

Scheme *Synthesis of salt [S]*

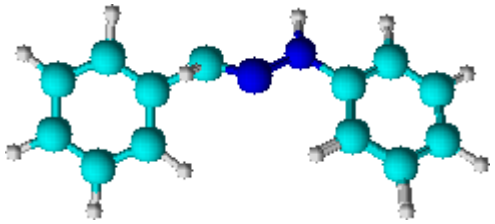
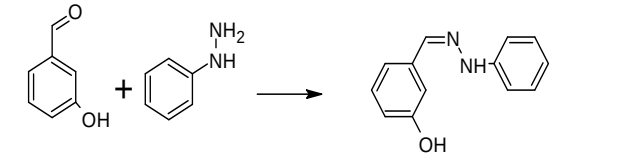


Figure *Three dimensional structure of salts [S]*

Synthesis of *N*-(3-hydroxybenzylidene)-phenyl hydrazine [Q]

3-hydroxybenzaldehyde (2 mL) as well as phenyl-hydrazine (2 mL) remained melted in 20 mL of CH_3CH_2OH as well as occupied in round lowest flask as well as refluxed for three hours. The development of process remained check done thin layer chromatography plates later half hour. Next the accomplishment of process, the warm mixture remained discharged in ice-cold H_2O. Formerly the creation remained sieved below suction, eroded with purified H_2O as well as dehydrated in desiccator below reduced pressure over an-hydrous $CaCl_2$. The subsequent product remained dark brown sticky-

waxy solid, Yield 75-79%, M.P. 187 ºC (Pawanoji & Mehta, 2009).



Benzaldehyde phenylhydrazine N-(3-hydroxybenzylidene)-phenyl hydrazine

Scheme *Synthesis of salts [Q]*

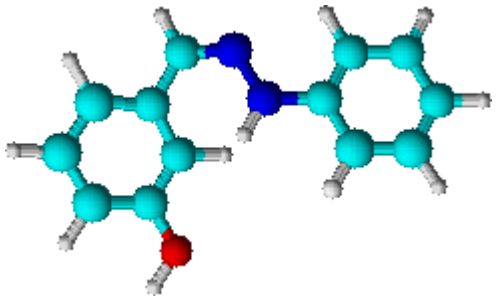
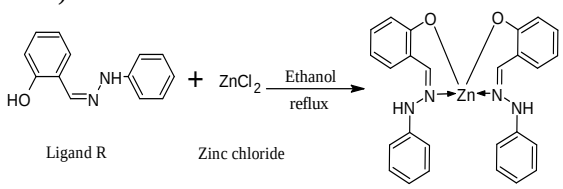


Figure *Three dimensional structure of salt[Q]*

Synthesis of Zn compound of salts R [R-1]

To the warm mixture of Schiff-base [R] (0.43g) in CH_3CH_2OH (20.00 mL), mixture of 1gram of metallic ligands ($ZnCl_2$) in CH_3CH_2OH remained more with continuous stirring. Stirring remained continuous till the accomplishment of process. The metallic compound thus moulded remained sieved as well as splashed by CH_3CH_2OH to eliminate the un-reacted salts as well as remained dehydrated over an-hydrous $CaCl_2$ in a desiccator. The colour of consequential composite remained pale yellow, Yield 50-55%, M.P.205 ºC (Yu et al., 2006).



Ligand R Zinc chloride [R-1]

Scheme *Zn-complex of R [R-1]*

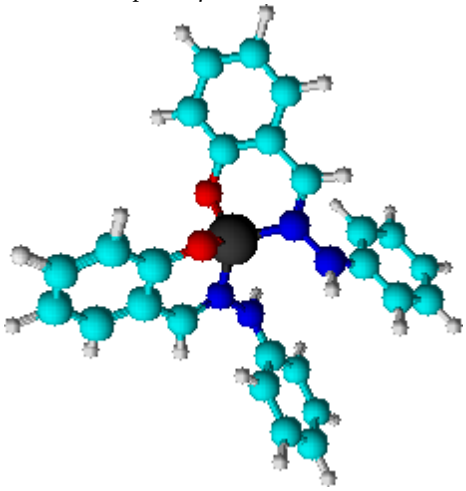
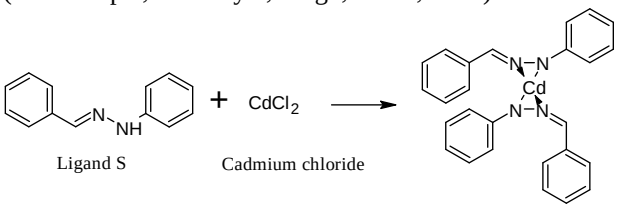


Figure *Three dimensional structure of [R-1]*

Synthesis of Cd complex of ligand S [S-1]

A mixture of metallic ligands ($CdCl_2$) (1.2g of $CdCl_2$ in 10 mL CH_3CH_2OH) remained initially ready. To the warm mixture of Schiff-base [S] (0.47g) in CH_3CH_2OH (20.00 mL), metallic ligand mixture remained additional by continuous stirring. Stirring remained constant till the conclusion of process. The metallic compound thus moulded remained sieved as well as splashed with CH_3CH_2OH to eliminate the un-reacted salts as well as remained dehydrated in desiccator with an-hydrous $CaCl_2$.the produce remained white-solid, Yield 65-70 %, M.P. 189 ºC (V. K. Gupta, Al Khayat, Singh, & Pal, 2009).



Ligand S Cadmium chloride [S-1]

Scheme *Cd-complex of S [S-1]*

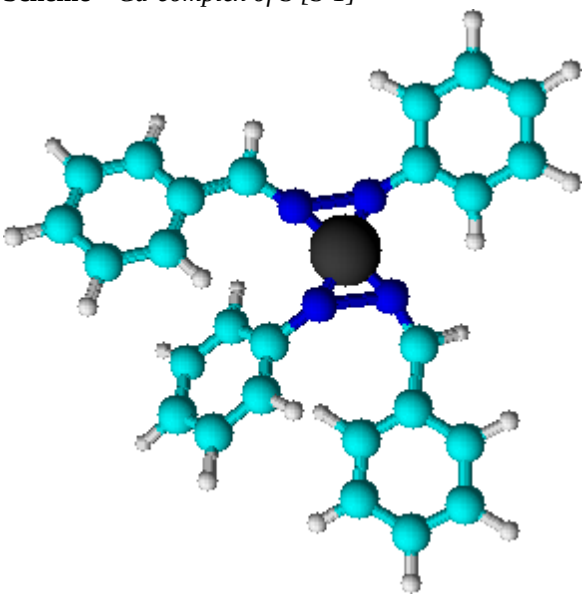
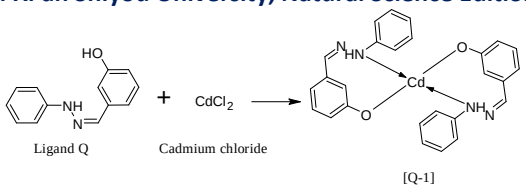


Figure *Three dimensional structure of [S-1]*

Synthesis of Cd complex of ligand Q [Q-1]

To the warm mixture of Schiff-base [Q] (0.49g) in total CH_3CH_2OH (20.00 mL), 1.5g of metallic ligand ($CdCl_2$) in CH_3CH_2OH remained further with continuous magnificent. Stirring remained constant till the conclusion of process. The metallic compound thus moulded remained sifted as well as eroded with CH_3CH_2OH to eliminate the un-reacted salts as well as remained dehydrated in desiccator below condensed gravity over an-hydrous $CaCl_2$. The resulting product remained dark brownish black sticky-waxy solid, Produce 73-78%, M.P. 210 ºC (V. K. Gupta et al., 2009).



Scheme *Cd-complex of Q [Q-1]*

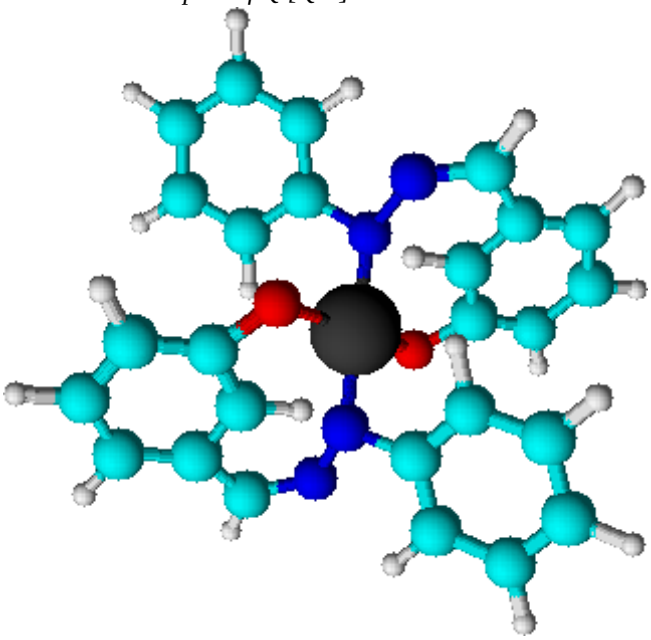


Figure Three dimensional structure of [Q-1]

RESULTS AND DISCUSSION

The chief objective of this article remains to manufacture the Schiff-base metal as well as salts by capable consequences for anti-oxidant as well as anti-bacterial actions (Sotiriou & Pratsinis, 2010) (biological study). This article offerings the manmade effort of certain Schiff-base salts as well as multi-dentate d-block developments Cd as well as Zn in command to attain some responsibilities such as antibacterial as well as antioxidant accomplishments (Shahidi & Zhong, 2015).

This study obtains the development of new di-valent metal-ion files (Devi, Yadav, Kumar, Naik, & Jindal, 2019) use minor molecular-weight complexes which comprise imine-groups as well as in certain belongings, likewise 1 phenolic-group. Phenyl-hydrazine (Al-Salami, 2018) remains create to be appropriate for the combination of d-block developments since of the occurrence of additional than 1 Nitrogen donor particles on every associating lateral which offers a "Compact" for the metallic ion to detained composed. Salicylaldehyde (Cimerman, Galic, & Bosner, 1997) as well as 3-hydroxybenzaldehyde (Mahon, McGinley, Rooney, & Walsh, 2009) (m-isomer of Salicylaldehyde) go faster the development of d-block developments since of the existence of hydroxyl-group in which O tolerates a lone-pair of e^- which container be given to the metallic ion. Dissimilar di-valent metallic ion ligands (MX_2) with,

M = Cd (II) and Zn (II) and X = Cl

The classification as well as consequences remained helped through Ultraviolet-Visible spectro-scopy, Infrared Spectro-scopy, Antibacterial as well as Antioxidant examination. In those portion possessions as well as consequences related to special-studies like ultraviolet, infrared spectro-scopy, m.p, Conductivity remain announced. All the created complexes like Schiff-base as well as their metallic complexes remained coloured, stable in air as well as non-hygroscopic(Pravin, Kumaravel, Senthilkumar, & Raman, 2017).

Physical Data

Table Molecular formulas for ligands and metal complexes

o.	ompounds No.	ompounds
1	[R]	$_{13}H_{12}N_2O$
2	[R-1]	$n(R)_2Cl_2]$
3	[S]	$_{13}H_{12}N_2$
4	[S-1]	$d(S)_2Cl_2]$
5	[Q]	$_{13}H_{12}N_2O$
6	[Q-1]	$d(Q)_2Cl_2]$

Table Colour, m.p as well as p*resence of*

No.	ands	Metal omplex	Colour	elting point)	cal appearance
1	[R]		y orange	185	Crystalline
2		[R-1]	e yellow	205	morphous
3	[S]		White	154	Crystalline
4		[S-1]	White	189	morphous
5	[Q]		k brown	187	ky waxy-solid
6		[Q-1]	rownish black	210	ky waxy-solid

manufactured complexes

The manufactured complexes remained contained of great dyes as well as sharp m.p by stable somatic conditions exposed in Table. All metal as well as salts developments remained processed figures except [Q] salts as well as [Q-1] metal-complex which gummy waxy hard as well as also these display uppermost m.p which exhibition the stability of complexes.

Solubilities

Solubilities of Schiff base salts as well as their metal developments remained checked in various mixture by sheeting down a slight quantity of composite in a test-tube as well as disbanding in a categorization of mixture such as H_2O, CH_3CH_2OH (Iftikhar et al., 2018).

Table *Solubilities of metal compounds as well as salts*

No.	ands	Metal plexes	ater	anol	thyl ther	thyl etate	oleum Ether	zene
1	[R]		IS	S	S	S	IS	S
2		[R-1]	S	PS	PS	PS	PS	PS
3	[S]		IS	IS	IS	S	IS	PS
4		[S-1]	IS	IS	IS	PS	PS	PS
5	[Q]		S	S	S	PS	S	PS
6		[Q-1]	S	S	S	PS	S	PS

Table clarifies the solubility belongings of dissimilar Schiff-base metal compounds as well as salts in altered solutions. Salt [Q], [R] as well as [Q-1] metal complex displayed high solubilities in Ethanol, Diethyl ether and Ethyl acetate while [Q] as well as [Q-1] remained incompletely soluble in Ethyl acetate level on warming.

Formula mass and yield

The formula form of a chemical-complex remains distinct as proportion amongst mass as well as the quantity of element of some example of that complex. The formula mass remains substance not molecular possessions of a constituent (Al Zoubi, Al-Hamdani, Ahmed, & Ko, 2018). It remains a amount of number of moles of a creation made in relation to the reactant expended got in a chemical feedback articulated as %age (Davis & May, 2005).

IR-spectrum

IR-spectra (range: 4000-400cm^{-1}) remained noted in FT-IR Spectro-photometer in command to check the functional-group of complex. IR spectro-scopy remains the extremely used implement for the acknowledgment of functional-groups in pure complexes as well as solution as well as mechanisms confirmation on molecular-vibrations. Absorption of radioactivity in the IR section consequences in the excitation of link distortions, either stretching or bending (Pervaiz et al., 2014).

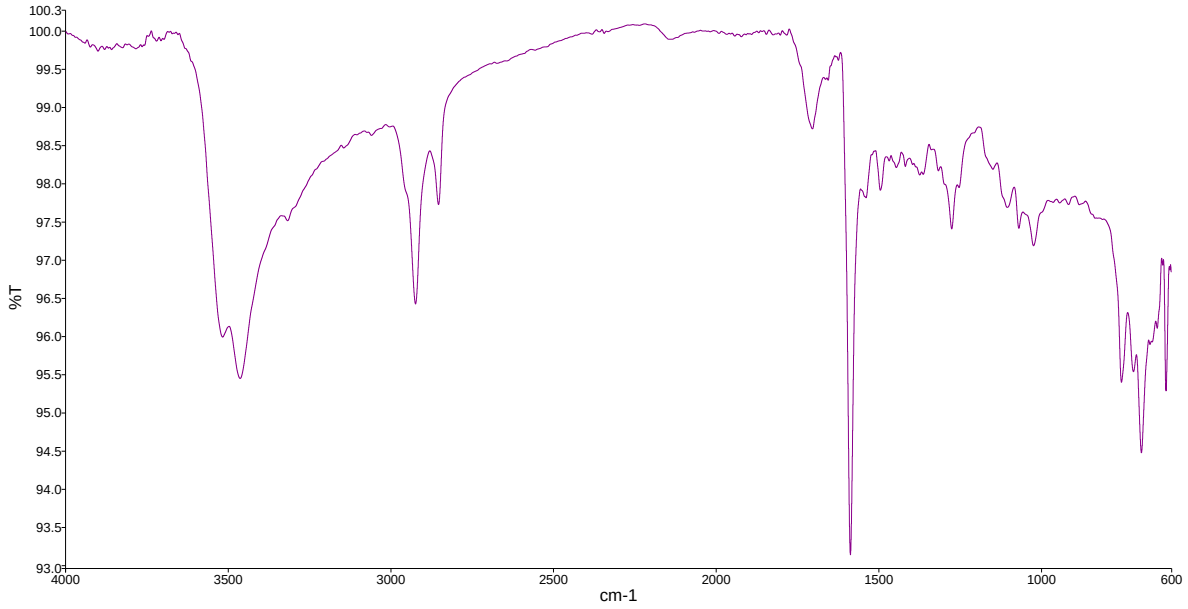


Figure *IR Spectra of Ligand [R]*

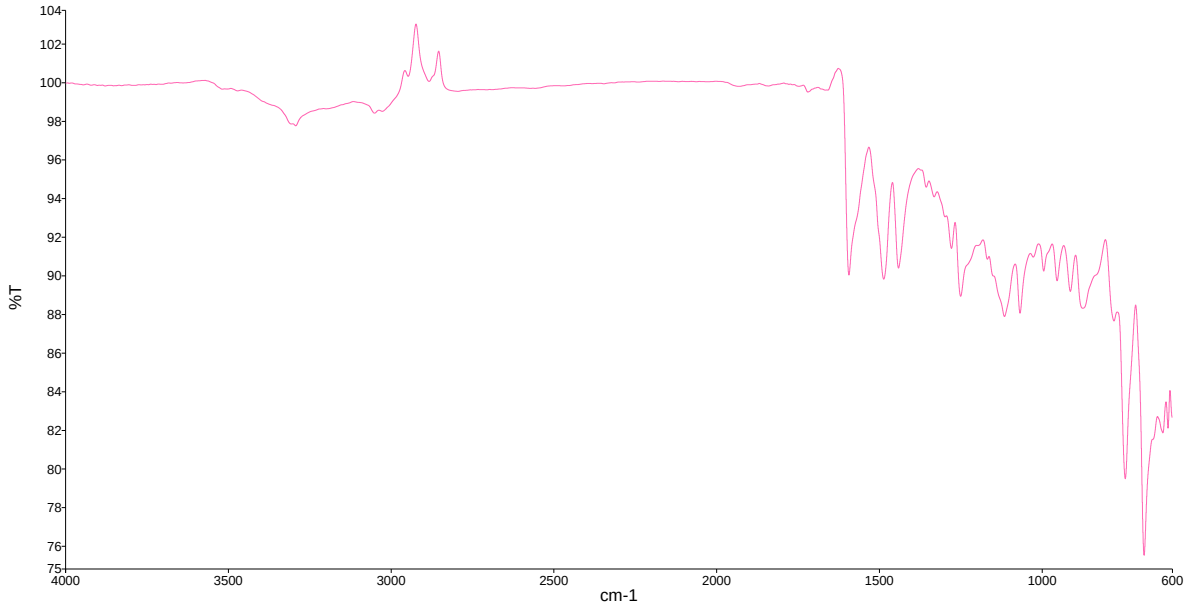


Figure *IR Spectra of Ligand [S]*

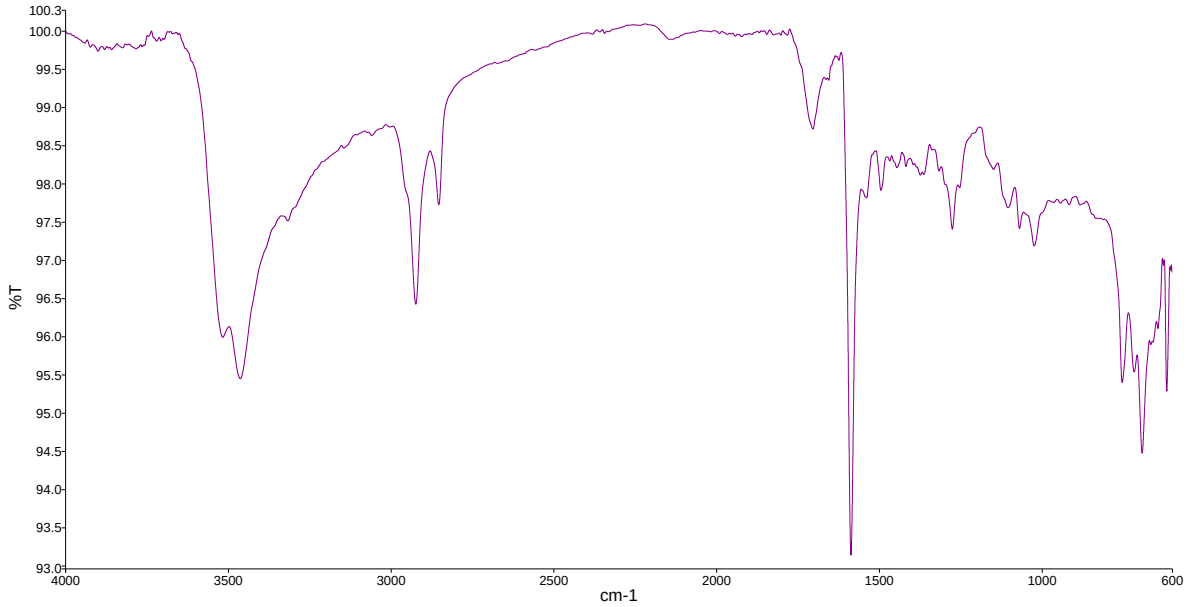


Figure *IR spectra of Ligand [Q]*

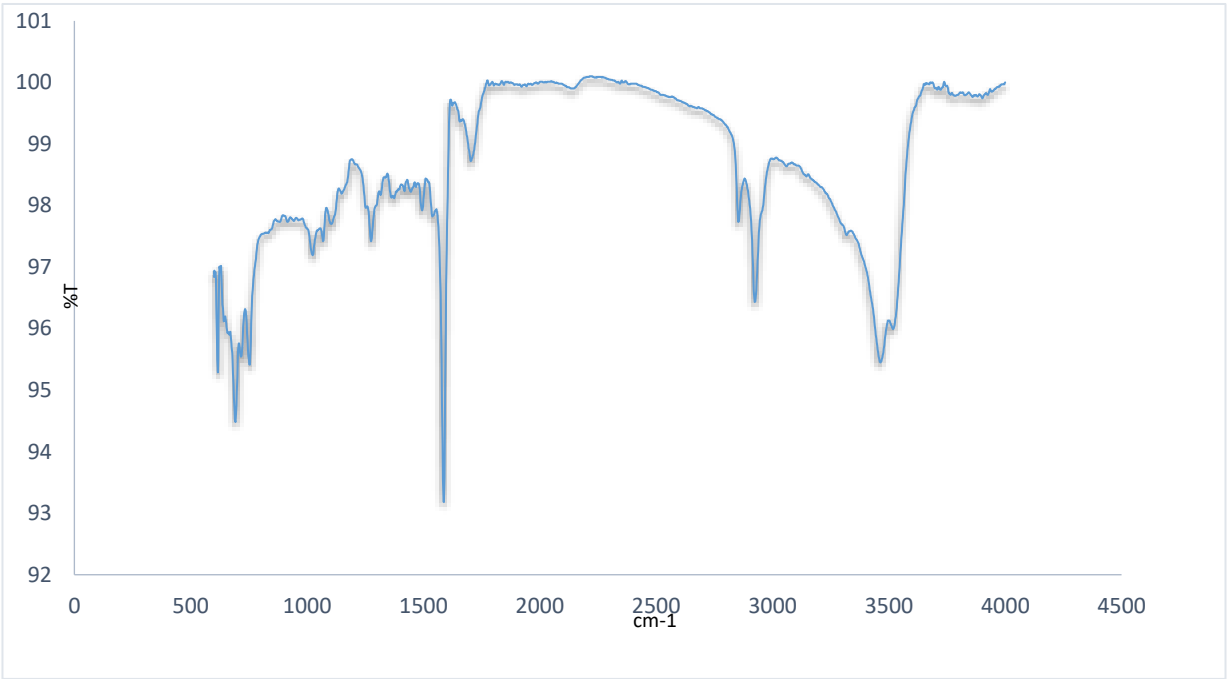


Figure *IR Spectra of complex [R-1]*

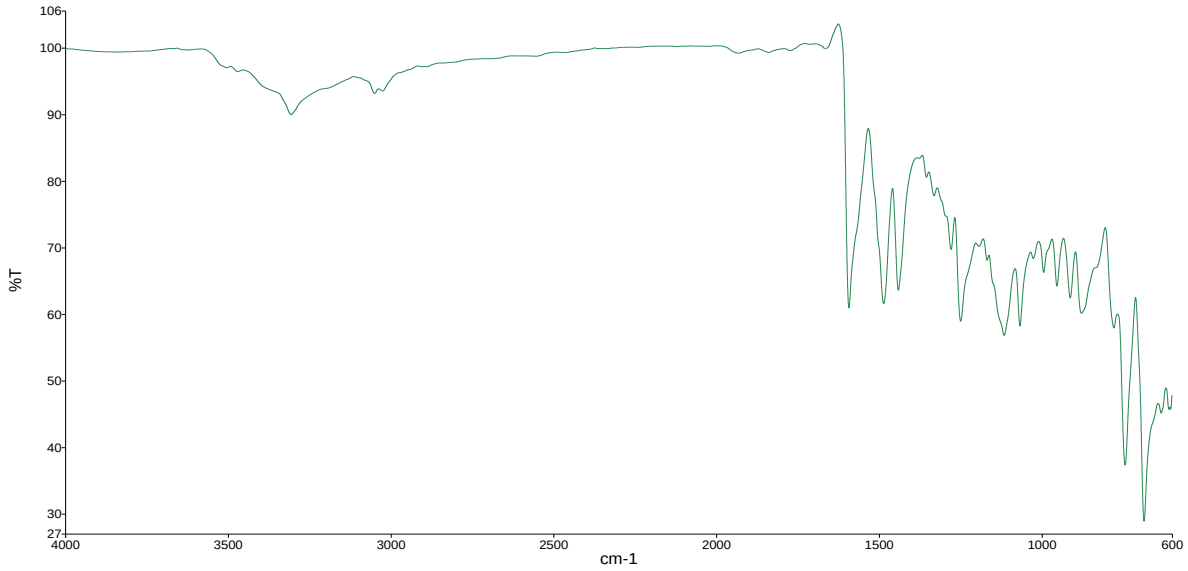


Figure *IR Spectra of complex [S-1]*

The definite structures of 2-[(Z)-(2-phenylhydrazinylidene) methyl] OH remained demonstrated by spectral examination as well as Infra-red group amount as follows: C=N (1640 cm^{-1}), C-N (1380 cm^{-1}), N-H (3382 cm^{-1}), O-H (3450 cm^{-1}), C-O (1220 cm^{-1}).

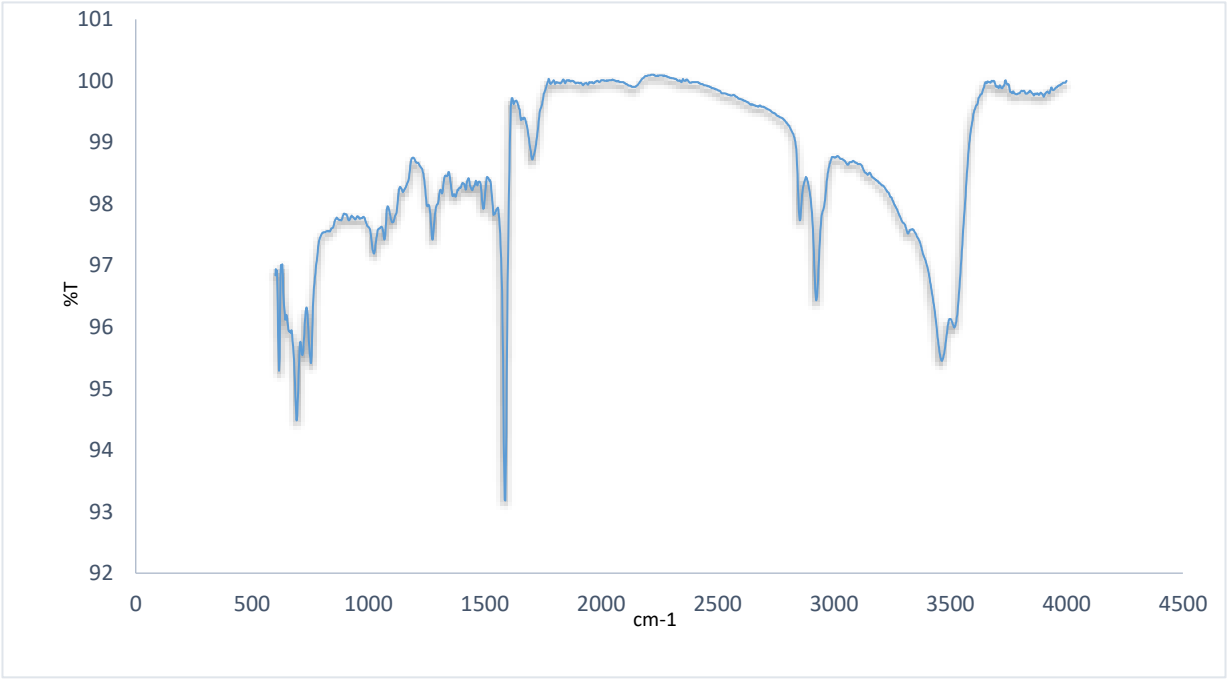


Figure *IR Spectra of complex [Q-1]*

The featured structure of (1*E*)-1-benzylidene-2-phenylhydrazine had band values of as follows: C=N (1650 cm^{-1}), C-N (1340 cm^{-1}), N-H (3320 cm^{-1}). The definite organization of 3-[(*Z*)-(2-phenylhydrazinylidene) methyl] OH remained confirmed by IR band values as follows: C=N (1641 cm^{-1}), C-N (1320 cm^{-1}), N-H (3400 cm^{-1}), O-H (3350 cm^{-1}), C-O (1220 cm^{-1}).

The conclusive features Zn-complex of Ligand [R] were evaluated by following IR data: C=N (1644 cm^{-1}), C-N (1290 cm^{-1}), N-H (3480 cm^{-1}), O-H (3500 cm^{-1}), C-O (1120 cm^{-1}), Zn-N (432 cm^{-1}), Zn-O (530 cm^{-1}). The features of Cd-complex of Ligand [S] were evaluated by following IR data: C=N (1652 cm^{-1}), C-N (1320 cm^{-1}), N-H (3390 cm^{-1}), Cd-N (452 cm^{-1}). The features of Cd-complex of Ligand [Q] remained estimated by following Infrared data: C=N (1647 cm^{-1}), C-N (1280 cm^{-1}), N-H (3482 cm^{-1}), O-H (3440 cm^{-1}), C-O (1210 cm^{-1}), Cd-N (440 cm^{-1}), Cd-O (544 cm^{-1}).

Anti-bacterial study

Anti-bacterial assay (Mary, 2007) of mock complexes remained consummate differing to 3-strains of bacteria like Two-gram negative bacteria (*Escherichia coli, pseudomonas)* as well as one gram-positive bacteria *(Staphylococcus aureus)* casted-off in modern research. That immobile is a lack of significance around awareness of influence cleanliness. Microorganisms remain unremittingly now to pollute the hands of individuals throughout several kind of accomplishments for which they remain insentient. The complexes that obtain anti-bacterial actions remains that cell decease happens when anti-bacterial agents reason demolition in the manmade method of cellular-walls as well as association of lipo-proteins because those structures of cell's perviousness remain demolished. The manufactured complexes remained capable of hampering microbial development as well as action by inquisition by the metabolic procedures in the microorganisms.

Table *Anti-bacterial activities of manufactured complexes*

o.	pounds	coli	domonas	reus
1	R]	nm	ND	nm
2	[S]	5mm	.7mm	nm
3	Q]	mm	10mm	nm
4	R-1]	nm	6mm	D
5	S-1]	D	ND	D
6	Q-1]	mm	12mm	mm

Table illustrations that most of complexes consumed anti-bacterial activities against all strains, whereas certain determined activities against specific strain.

Antioxidant study

Anti-oxidant research of the manufactured salts as well as their analogous di-valent metal developments remained accomplished by 1, 1-diphenyl-2-picryl hydrazyl radical (DPPH) method (Gyamfi et al., 1999). These complexes remained accomplished of inhibiting oxidation method (Noureen, Saleem, Fatima, Siddiqi, & Mirza, 2013).

Table *Anti-oxidant DPPH values for metal as well as salts*

No.	Compounds	PH Scavenging activity
1	[R]	52.81±1.29
2	[S]	31.24±1.23
3	[T]	32.16±1.23
4	[Q]	55.85±1.17
5	[R-1]	47.50±1.92
6	[S-1]	34.56±0.67
7	[T-1]	14.19±0.72
8	[Q-1]	58.57±0.87

The consequences displayed that furthermost of complexes consumed anti-oxidant actions where as certain has exposed great capacity to act as anti-oxidant. Table exposed that determined oxidant significance remained showed by salt [Q] and [Q-1] metallic complex as well as a while of fewer remained [R] salt.

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