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Synthesis, Characterization and Biological Activity of Some Mixed Ligand Transitionmetal Complexes

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Abstract: *The Schiff bases derived from Salicylaldehyde with substituted amines and alpha benzoin oxime as a primary ligand are prepared by mixed with Cobalt (II), Nickel(II) and Copper (II). Structure have been proposed from elemental examination, like IR, NMR, thermal examination and magnetic susceptibility, Spectroscopic studies suggests that coordination occurs through azomethine nitrogen, hydroxyl group and oxime of the ligand to the metal ions. Elemental analysis of Schiff bases, alpha benzoin oxime and metal complexes are confirmed to stoichiometry of the type ML_1L_2 where Metals are Cobalt (II) and Nickel (II) L_1 are Schiff bases and L_2 are alpha benzoin oxime. The mixed ligand Co(II) complexes were synthesize by using α-benzoin oxime as a primary ligand and Schiff base prepared from salicylaldehyde and chloroanilines with hydroxyanilines as a secondary ligand. The metal complexes have been characterized different elemental analyses and various chemical techniques such as molar conductance, magnetic susceptibility, infrared, NMR, spectral studies and thermal analysis. The elemental analysis data is consistent with their general formulation as mixed ligand complexes MLL'.xH_2O. The bondings and structures of the complexes are discussed in detail on the basis of the results of various chemical studies. All of these metal complexes and ligands have been screened for their biological activities against selected pathogenic microbial strains. The Ditch Plate Method has been used to study the antibacterial activity against E. coli, S. typhi, S. aureusandS. pyogenes.*

Keywords: Schiff base, Benzoin Derivative, Metal complexes, biological activity

I. INTRODUCTION

Cobalt, Nickel and other transition of metal complexes have very good color and having very nice empirical formula [1]these are called as the coordination complexes [2], the presence of the very 2,4,5,6 and more good chemical groups which having the good geometrically placed around the metal ion and having the good proposed features. these groups are the coordinating groups change the chemical behavior of the metal with significantly as the electronic properties that the various metal or the free metal or ion does not which absorb the visible light.[3] large area of the biochemistry are mainly used in metal chemistry. Schiff base and their metal complexes are largely widespread because of their chelating abilities [4]they have very significant in the analytical structural studies as the consequence of various fundamental towards the various metal atoms and metal complexes of various Schiff base are considered. The result of the various flexibility with good sensitivity having the synthetic selectivity, these having the very complex structures. These can be used as the catalyst, in the dye industry and various intermediates,these complexes and employed as the catalyst ,dyes, intermediates in the various biological formation and polymer stabilizers. The large number of the Schiff base molecules have organic action of the ant diabetic ,anticancer, anti-proliferative antitumor, antifungal, antiseptic, anti-inflammatory showing the good performance.[5-6]

Alpha benzoinoxime and other is one of the Schiff Base compounds which is useful in the synthesis of the various Schiff base ligand that have very good application in the pharmaceutical industry ,industrial uses, and antimicrobial [7] as its consequences this work has dedicated to the various groups Cu (II), Ni(II) Co(II) as metal ions.[7-9]

II. MATERIALS AND METHODS

Chemical and the various instrument required are used of the good quality the most of the chemicals are purchased from the company like Sigma Aldrich, S. D. Fine, LOBA, they have very good purity mostly the elemental analysis

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carried using the EA300A the absorption spectrum was studied using the Shimadzu UV-Vis 1800 spectrophotometer, this can done at room temperature and 1 cm quartz cell was used and the IR spectrum using the Shimadzu FT IR without KBrpalate and with ATR at the frequency at 4000-400 cm-1.Electrial Conductivity was determined using the conductivity meter in ethanol as solvent room temperature. The Gouy's technique used to study the magnetic susceptibility, NMR Spectrum was studied using the NMR Spectrometer, Solvent used is the Ethanol, thin layer chromatography was studied in the aluminum plates covered with the gel of the silica.

The practical Procedures as the Schiff base ligand preparation. This occur in two steps as the step one is the preparation of the Schiff base, this is prepared by the condensation of the Aromatic amine with the different carbonyl functions this reaction catalyzed by the acid or the base in the absolute alcohol.

2.1 Preparation of Schiff base

In the round bottom container take the 0.01 mol of the substituted aromatic amines in 15 ml of the ethanol and the Salicylaldehyde in 15 ml of the absolute alcohol with few drop of the acetic acid and refluxed for the 4-5 hrs.,then the product obtained recrystalled with the hot ethanol, it gives 80 % yield.[10]

2.2 Formulation of Metal Complexes

The chelate and metal taken in the ration of the 1 as to 2 .complexes are of the metal and the ligand are ready by taking and solubilised (0.002 mol)Schiff base in (15 ml) of heated absolute ethanol. The corresponding hydrated metal chloride salts of ($CoC_{l2}.6H_2O$, $NiCl_2.6H_2O$ and $CuCl_2.2H_2O$) of (0.001 mol) was placed in hot absolute alcohol (15 mL) was placed with heated absolute alcohol as solution of the ligand and refluxed for 1 hour on a water bath and the contents was cooled. The complexes was separated out in each case. The product filtered, placed with alcohol and dried in vacuum.

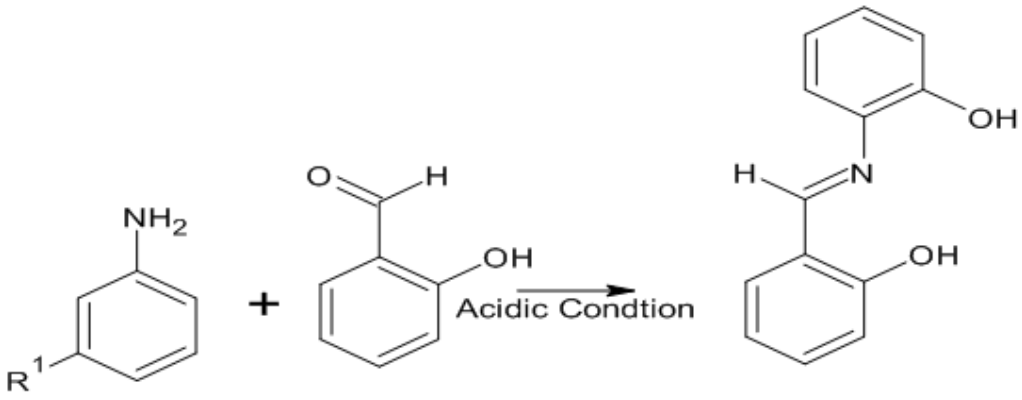
2.3 Biological Activity Study

The newly synthesized ligand of all complexes are investigated with certain gram positive bacteria and certain gram-negative bacteria $1x10^{-3}$M. The dishes are incubated at a temperature of 37°C for complete day. The formed inhibiting region by compounds against the specific tested bacterial strain can be used to evaluate the antibacterial activities of the synthetic compounds. The obtained mean magnitude for three individual replicates has been employed to compute growth inhibition zone of every sample

III. Results and Discussion

The Schiff base ligand have yellow to brown crystals which are partially soluble in water for the biological solvent ,it can form the different color sample ,all of these complexes arethese complexes have the good air stability, they are insoluble in the water ,they have good solubility in the solvent like DMSO and DMF,like chloroform and methanol and ethanol.

Reactions:



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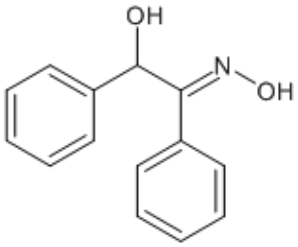
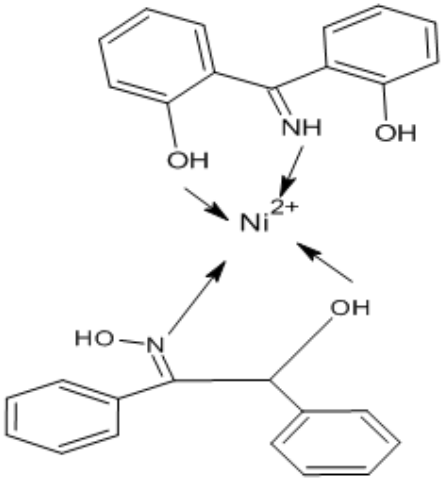
Ligand L1

Schiff Base of the various Substituted Aromatic amines with theSalicylaldehydeR1 is –OH Ortho,Meta,Para Substituted Aromatic Amines are used

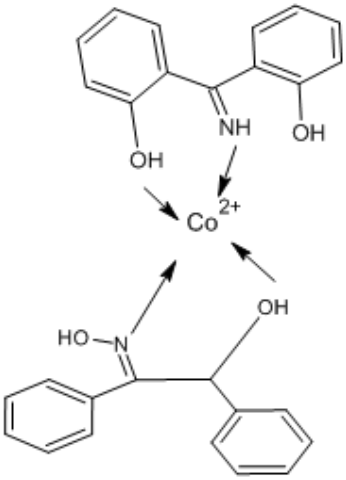


Ligand 2 is a alpha benzoin oxime (Primary Schiff's Base)

Formation of the metal complex with different Complexes with the metal like Nickel, Cobalt and Copper:



Ortho, meta, para substituted complexes of various ligand with metal Ni(II)



Ortho, meta, para substituted complexes of various ligand with metal Co(II)

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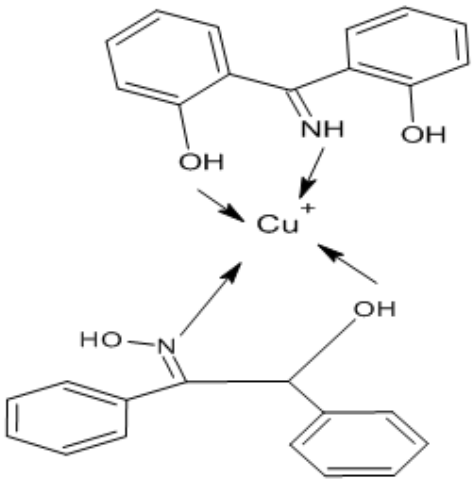

Ortho, meta, para substituted complexes of various ligand with metal Cu(II)

3.1 Elemental Investigation:

A. Physical Characteristics and Elemental Investigation

Physical characteristics and elemental data obtained from the C,H,N investigation and metal substance.

The physical characteristics and outcomes taken from C,H,N investigation and metal substances of the arranged complexes are in table. From this the structure has been confirmed ,two different ligands are used with different metal like Nickel, Cobalt and copper.

Sr. No.	Compound	Color	M.W	M.P. °C	Yield	Founded Calculations in %				Metal
--	--	--	--		--	C	H	O	N	M
1	$C_{14}H_{13}NO_2$ (L_1)	Yellow	227	153-155	--	77.99	5.77	14.08	6.16	-
2	$C_{13}H_{10}N_2O_4$(L_2)	Yellow	258	178	85	77.99	5.77	14.08	6.16	-
3	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Ortho)	Yellow	543	198	86	59.59	4.26	17.64	7.72	Ni(10.79)
5	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Meta)	Yellow	543	202	85	59.59	4.26	17.64	7.72	Ni(10.79)
6	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Para)	Yellow	543	204	94	59.59	4.26	17.64	7.72	Ni(10.79)
7	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Ortho)	Faint Yellow	543.93	206	90	59.59	4.26	17.64	7.72	Co(10.79)
8	[[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Meta)	Faint Yellow	543.93	208	92	59.59	4.26	17.64	7.72	Co(10.79)
9	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H13NO_2$ (Para)	Yellow	543.93	202	95	59.59	4.26	17.64	7.72	Co(10.79)
10	[Cu($C_{13}H_{10}N_2O_4$)]	Yellow	548.53	209	90	59.59	4.26	17.64	7.72	Cu(10.79)

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	and $C_{14}H_{13}NO_2$ (Ortho)									
11	$[Cu(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$ (Meta)	Yellow	548.53	211	93	59.59	4.26	17.64	7.72	Cu(10.79)
12	$[Cu(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$ (Para)	Faint Yellow	548.53	207	93	59.59	4.26	17.64	7.72	Cu(10.79)

C= Carbon, H= Hydrogen, N= Nitrogen M = Metal

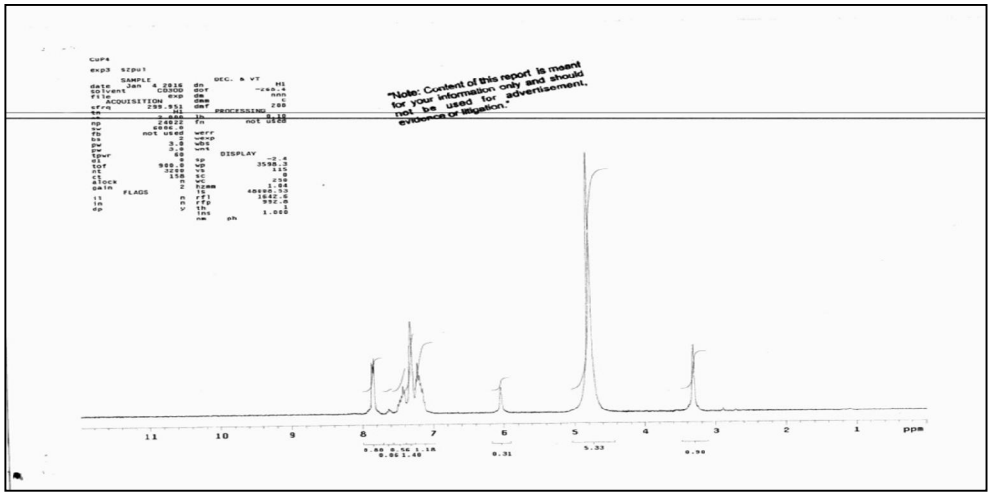
B. Thin Layer Chromatography

The thin layer chromatography was carried in the ethanol as solvent, it shows the single isomer indicate the single isomer.

Sr. No.	Ligands and mixed ligand complexes	Color
1	$C_{14}H_{13}NO_2$ (L_1)	0.84
2	$C_{13}H_{10}N_2O_4$(L_2)	0.78
3	$[Ni(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Ortho)	0.56
5	$[Ni(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Meta)	0.58
5	$[Ni(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Para)	0.60
6	$[Co(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Ortho)	0.65
7	$[Co(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Meta)	0.67
8	$[[Co(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Para)	0.69
9	$[[Cu(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Ortho)	0.70
10	$[Cu(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Meta)	0.72
11	$[Cu(C_{13}H_{10}N_2O_4)]$ and $C_{14}H_{13}NO_2$(Para)	0.72

C. NMR Spectra for Complex with the Para of Hydroxyl Aniline Substitution

The NMR band of the complex was exhibited the major signal in the aromatic region indicate the presence of the aromatic ring ,indicating the 16 protons in the range of the 7 -8 δ.the δ value around the 4.5 -5 δ indicate the presence of the hydroxyl group in the structure ,and NMR peak 3.5-4.2 δ.



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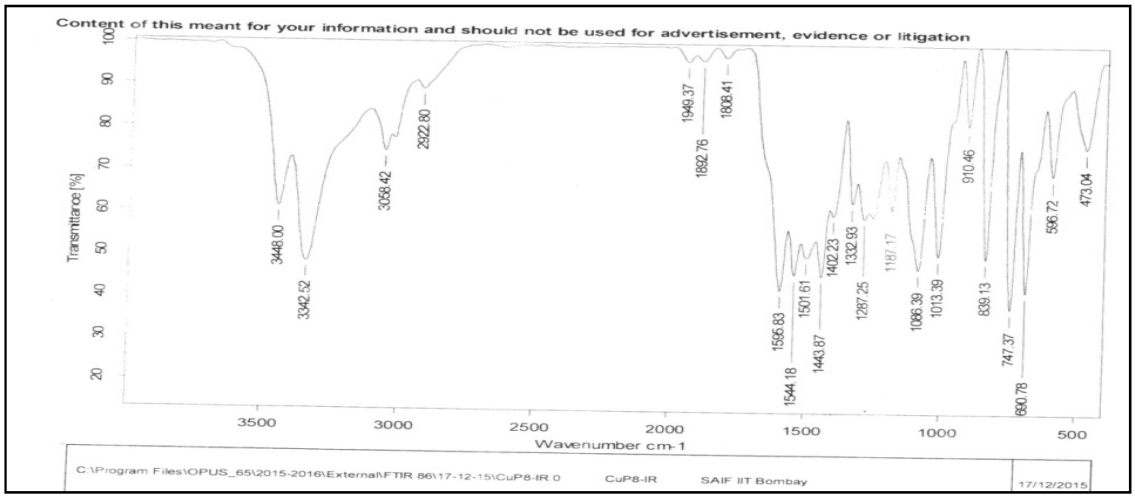
Spectrums of Ligand and its Complexes

The IR Spectral data of different Schiff base and its complex were studied ,it shows the typical band at the ,the free ligand spectrum has strong band in 3300cm^{-1}-3400 cm^{-1} indicate the being there of the hydroxyl group in the structure,the IR Spectrum at the 1600-1700cm^{-1} indicate presence of the C=N,it also presence of the aromatic ring The presence of a new two bands around (455-428) cm^{-1} and (558-509) cm^{-1} in IR spectrums of all complexes owing to υ(M-N) and υ(M-O) substantiates respectively.



Magnetic Measurements

The Co (II) complex having the good magnetic μeffis around,4.16 B.M. based on the uncombined electrons,this is related to the octahedral magnetic moment Ni (II) complex exhibits magnetic moment around the 3.18 B.M. at the room temperature, with the octahedral geometry based on the unpaired electrons at room temperature based on dualistic uncombined electrons. Cu (II) complex exhibits magnetic moment around the 3.58 B.M. at the room temperature, with the octahedral geometry based on the unpaired electrons at room temperature based on dualistic uncombined electrons.

Conductivity Measurements

Every one soluble complexes show molar conductivity with values at range (16.03 – 11.14) S.cm^2. mol^{-1}in ethanol medium in 10^{-3}M at room temperature. These magnitudes point to small conductivity and nonionic structure of these complex [30]. The conductivity were values in table

Sr .No.	Compound	**Molar Conductivity S.cm2.mol-1**	**μeff B.M**
1	$C_{14}H_{13}NO_2$ (L_1)	--	--
2	$C_{13}H_{10}N_2O_4$(L_2)	--	--
3	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Ortho)	23.22	3.18
5	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Meta)	24.25	3.20
5	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Para)	23.45	3.22
6	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Ortho)	19.25	4.16
7	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Meta)	18.78	4.10
8	[[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Para)	19.27	4.12
9	[[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Ortho)	22	3.56
10	[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Meta)	22.78	3.48
11	[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H1_3NO_2$(Para)	22.89	3.50

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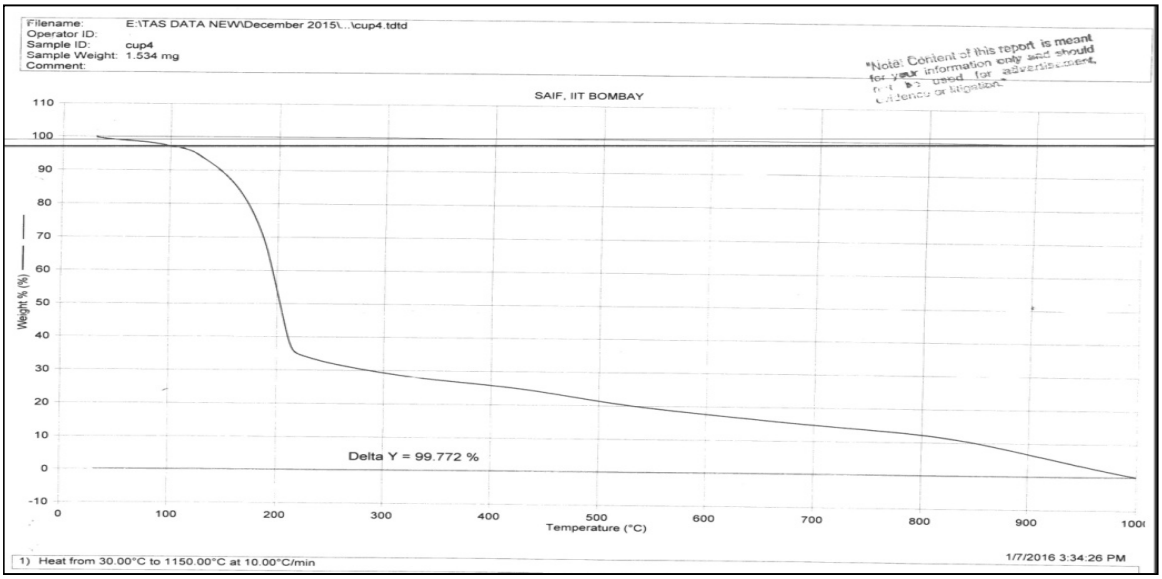
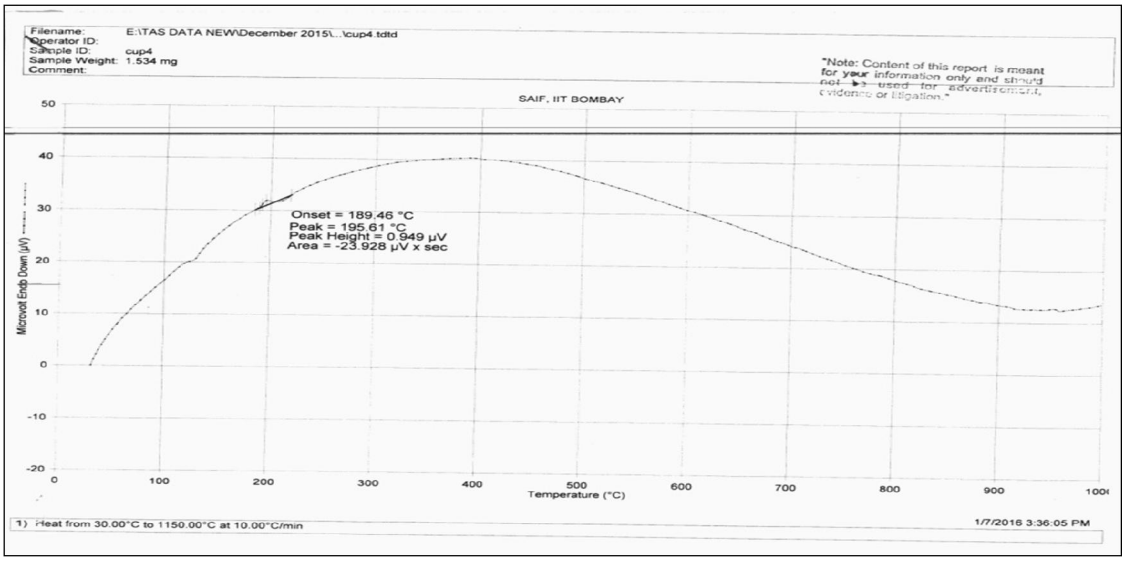
Thermal Analysis for Many Metal Complexes

The TGA and the DTA are the method from first to last the range (0-1000 ^{0}c) at very short heating rate in the nitrogen ambiance The different consequences of TGA and DTA are and represent thermo grams . the TGA curves are refer to the dissimilar thermal constancy of the complexes .these curves shows the stepwise decomposition of the mixed ligand occur in the primary step there is the weight loss with taking absent of molecules with very low molecular weight such as ammonia and water in which is established. While The second and third steps involved the thermal decomposition of the remaining parts of complexes are taking place . On the additional DTA of the curves evidence the change that cause by temperature such as vaporization ,melting, sublimation, or change that credited weight wounded are represented an endothermic natureas the processes crystallization in the change of crystal structure and oxidation are represent exothermic natural world

TGA Data



DTA Analysis Data



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Biological Activity

The biological activity was studied using the disc plate method for the various metal complexes in this the ligand with the complexes contain that *S. aureus* and *S. pyogenes* susceptible to ligand and its complexes while *E. coli* and *K. pneumonia* showed a reverse activity which resistance Co(II) complex but to ligand and Ni(II)

Sr. No.	**Inhibition area of the bacterial sensitivity (zone of inhibition in mm)**	*E-Coli* In mm	*Samplella* In mm	*S,Aureus* In mm	*pyogenes* In mm
1	$C_{14}H_{13}NO_2$ (L_1)	19	18	20	20
2	$C_{13}H_{10}N_2O_4$(L_2)	20	21	28	23
3	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Ortho)	22	21	21	21
4	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Meta)	26	2 7	28	26
5	[Ni($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Para)	24	23	22	26
6	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Ortho)	25	27	25	28
7	[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Meta)	26	27	28	26
8	[[Co($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Para)	24	23	22	26
9	[[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Ortho)	28	28	30	30
10	[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Meta)	27	29	30	30
11	[Cu($C_{13}H_{10}N_2O_4$)] and $C_{14}H_{13}NO_2$(Para)	29	30	31	32

IV. CONCLUSION

In this research, the research and representation of Cobalt (II), Nickel (II) and Copper (II) complexes of ligand were accomplished by means of elemental analyses, metal content, mass spectrum, 1H-NMR, IR, and UV-Vis spectral measurements. Magnetic susceptibility measured results verify octahedral structure of the complexes. They are have greater biological actions than the respective ligand.

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