

# Synthesis, Characterization and Spectroscopic Technique Analysis of 3-[3-(1H-Indol-3-yl)Acryloyl]-4-Hydroxy-6-Methyl-2H-Pyran-2-one and their metal complexes

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**Abstract :** In the present research article, the some of first transition series metal complexes derived from (4-hydroxy-3-[3-(1H-indole-3-yl)-acryloyl]-6-methyl-2H-pyran-2-one) were synthesized from Dehydroacetic acid and Indole-2-carboxaldehyde. They correspond well with the general formula  $[M(L)_2(H_2O)_2]$ , where  $M = Mn(II), Co(II), Ni(II)$  and  $Cu(II)$  and  $[M(L)_2(Cl)(H_2O)]$ , where  $M = Fe(III)$  and  $L = C_{17}H_{13}NO_4$ . The ligand was characterized on the basis of elemental analysis, UV, IR, Mass, <sup>1</sup>H NMR and antimicrobial activity. All the complexes were characterized by elemental analysis, UV, magnetic susceptibility measurements, IR, XRD & antimicrobial activity. The ligand acts as a bidentate chelate and coordinates through two oxygen atoms of ligand i.e complex formed by 1:2 (metal: ligand) ratio. The ligand and its metal complexes were screened for antimicrobial activity against *Bacillus Cereus*, *Bacillus Megaterium*, *Shigella boydii* and *Escherichia Coli* bacteria, and *Saccharomyces Cerevisiae*, *Aspergillus Oryzae* and *Penicillium notatum* fungi were studied.

**Keywords:** Magnetic susceptibility; Chalcone; ligand; XRD; Antimicrobial activity.

## I. INTRODUCTION

A number of schemes for the preparation of the chalcone built on the formation of carbon-carbon bond have been described. Amongst the straight aldol condensation and Claisen-Schmidt condensation static take place prominent site<sup>1,2</sup>. The leading way for the preparation of chalcones is the conventional Claisen-Schmidt condensation in the existence of aqueous alkali  $Ba(OH)_2$ <sup>3,4</sup>. On the other hand several of this approach is hurt from strict reaction circumstances, poisonous reagents, strong acidic as well as basic conditions, lengthy reaction period, low yield and low selectivity. While, some changes have been complete to counter these difficulties. There is yet essential for the improvement of choosy and improved schemes for the preparation of  $\alpha, \beta$ -unsaturated carbonyl compounds<sup>1,2</sup>. A investigation of the literature review shown that no work has been complete on transition metal chelates of the chalcone resulting from dehydroacetic acid plus Indole-3-carboxaldehyde. The chelates of Copper(II), Nickel(II), Cobalt(II) Manganese(II) and Iron(III) through this ligand were too synthesized in the solid state then characterized by different analytical, spectral and thermal techniques.

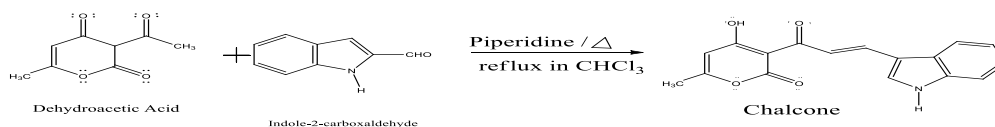
## II. MATERIAL AND METHODS

Dehydroacetic acid for synthesis was obtained from Merck, Germany & used as supplied. Indole-3-Carboxaldehyde of A.R. grade obtained from AVRA chemicals were used for the synthesis of the ligands. A.R. grade hydrated metal chlorides from Thomas Baker were used for the preparation of the complexes. The carbon, hydrogen & nitrogen content in each sample were measured on a Perkin Elmer (2400) CHNS Analyzer. The IR spectra (KBr), in the range of 4000-450  $cm^{-1}$  were recorded on a Perkin Elmer (C-75430) IR spectrometer. The <sup>1</sup>H-NMR spectrum of the ligand was measured in  $CDCl_3$  on Bruker instrument. The mass spectrum of the ligand was measured in Qc-01 DAD Mass-spectrometer, X-ray diffractograms of the all chelates was recorded in  $2\theta$  range of 5-70° by copper Ka radiation initiator at a wavelength of 1.540598 Å at normal temperature. The Ultra-Violet visible spectra of the complexes were recorded on a Shimadzu UV-2202 Spectrophotometer. Magnetic susceptibility measurements of the complexes were performed using a Gouy balance at room temperature using  $Hg [Co (SCN)_4]$  as the calibrant.

### Synthesis of the Ligand :

A 0.01 mol of Dehydroacetic acid (DHA), then add few drops of piperidine base with constant stirring and then lastly add 0.01 mole of Indole-3-Carboxaldehyde in 30ml of chloroform solvent were refluxed for 6-8 hours, after some times approximately 10 ml of the chloroform-water azeotrope combination was separated by purification<sup>1,2</sup>. Solid type of crystal product separated on sluggish vanishing of the residual chloroform solvent. The subsequent precipitate was filtered using standard funnel, simultaneously washed some times through ethanol and recrystallization carried out by the chloroform solution<sup>5,6</sup>.

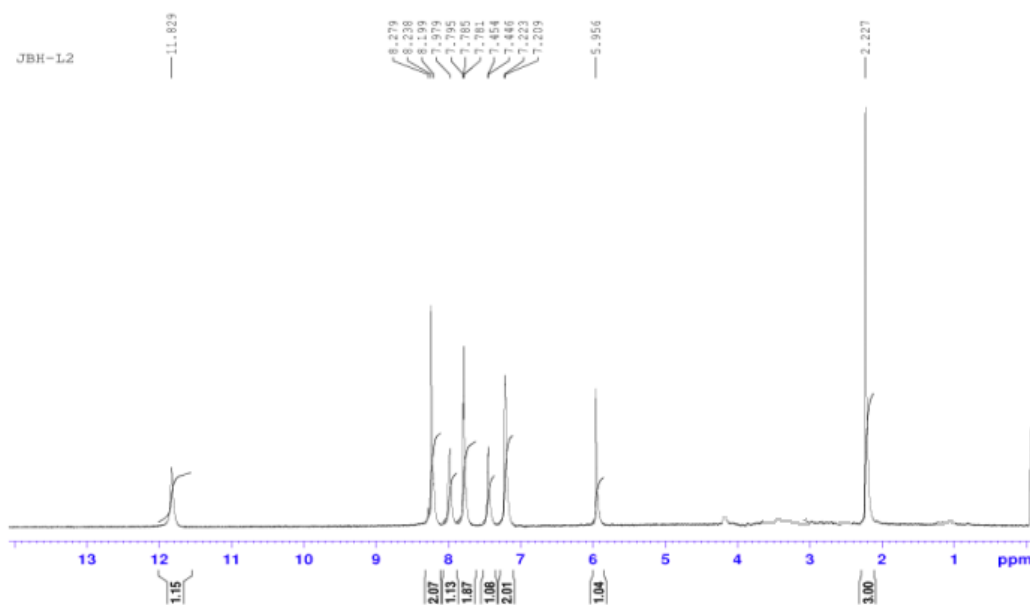
### Reaction :



**Scheme 1: Synthesis of Ligand**

Analysis of Chalcone Ligand is carried out through CHN analyser, UV spectra, IR spectra, <sup>1</sup>H-NMR Spectra and Mass Spectra.



**Fig.1.01 :- <sup>1</sup>HNMR Spectrum of Chalcone Ligand :****IV) IR Spectrum of Ligand and Their Chelates :**

Appropriate infrared bands that deliver extensive physical sign for the formation of ligand and their metal chelates are particular in Table 1.02. The infrared spectra of ligand displays specific bands on 3402, 3132, 1747, 1697, 1242-1196  $\text{cm}^{-1}$  indicated to the  $\nu(\text{N-H})$  stretching in indole moiety,  $\nu(\text{OH})$  of the intramolecular phenolic assembly of the dehydroacetic acid,  $\nu(\text{C=O})$  (lactone carbonyl in the ring of DHA),  $\nu(\text{C=O})$  (acetyl carbonyl in the dehydroacetic acid moiety) and  $\nu(\text{C-O})$  (stretching in phenolic group on DHA) stretching approach, respectively<sup>1,2,12,13</sup>. In the FTIR spectrum of every metal complex, no band was detected in the area of 3165-3100  $\text{cm}^{-1}$ . As an alternative, a wide-ranging band typical of  $\nu(\text{OH})$  of chelation water molecule was detected in the area of 3400-3170  $\text{cm}^{-1}$ . The existence of chelated water molecule was supplementary definite by the presence of a non-ligand band now the area 835-843  $\text{cm}^{-1}$ . The nonappearance of  $\nu(\text{OH})$  (Phenolic group) at 3120-3100  $\text{cm}^{-1}$  recommends successive deprotonating of the phenolic functional group and direction of phenolic oxygen atom to the transition metal ion. This was reinforced by an rising shift in  $\nu(\text{C-O})$  (phenolic group)<sup>14</sup> by 15-40  $\text{cm}^{-1}$ . The  $\nu(\text{C=O})$  (acetyl carbonyl of DHA) was moved to lesser energy with related to the free ligand, signifying the involvement of the acetyl carbonyl of DHA in the chelation<sup>1,2,12,13</sup>. The infrared spectrum of entirely the compounds displayed a noticeable band at  $\approx 1376$  &  $\approx 972$   $\text{cm}^{-1}$ , distinctive of  $\nu(\text{C-O-C})$  as well as *trans* -CH=CH- absorption. The occurrence of novel bands in the area of 640-460  $\text{cm}^{-1}$  can be allotted to  $\nu(\text{Metal-Oxygen})$  vibration<sup>1,2,15</sup>.

**Table 1.02: Infrared spectroscopic figures of ligand and their metal chelates**

Ligand and Complex	$\nu(\text{N-H})$ (indole ring)	$\nu(\text{OH})$ (dehydroacetic acid moiety)	$\nu(\text{C=O})$ (lactone)	$\nu(\text{C=O})$ (acetyl carbonyl)	$\nu(\text{C-O})$ (phenolic)	$\nu(\text{C=C})$ (trans)	$\nu(\text{M-O})$
Ligand HL $\text{C}_{17}\text{H}_{13}\text{NO}_4$	3402 <sub>(s)</sub>	3132 <sub>(s)</sub>	1747 <sub>(s)</sub>	1707 <sub>(m)</sub>	1242 <sub>(s)</sub>	997 <sub>(m)</sub>	-
$\text{C}_{34}\text{H}_{30}\text{CoN}_2\text{O}_{10}$	3287 <sub>(s)</sub>	-	1678 <sub>(s)</sub>	1649 <sub>(s)</sub>	1268 <sub>(w)</sub>	971 <sub>(w)</sub>	531 <sub>(w)</sub> 476 <sub>(m)</sub>
$\text{C}_{34}\text{H}_{30}\text{CuN}_2\text{O}_{10}$	3214 <sub>(m)</sub>	-	1680 <sub>(m)</sub>	1654 <sub>(s)</sub>	1270 <sub>(m)</sub>	975 <sub>(m)</sub>	560 <sub>(m)</sub> 480 <sub>(m)</sub>
$\text{C}_{34}\text{H}_{28}\text{ClFeN}_2\text{O}_9$	3296 <sub>(s)</sub>	-	1681 <sub>(s)</sub>	1647 <sub>(m)</sub>	1270 <sub>(s)</sub>	972 <sub>(s)</sub>	530 <sub>(s)</sub> 488 <sub>(s)</sub>
$\text{C}_{34}\text{H}_{30}\text{MnN}_2\text{O}_{10}$	3170 <sub>(m)</sub>	-	1675 <sub>(m)</sub>	1645 <sub>(w)</sub>	1268 <sub>(w)</sub>	974 <sub>(m)</sub>	560 <sub>(w)</sub> 462 <sub>(s)</sub>
$\text{C}_{34}\text{H}_{30}\text{N}_2\text{NiO}_{10}$	3307 <sub>(s)</sub>	-	1681 <sub>(s)</sub>	1647 <sub>(s)</sub>	1268 <sub>(s)</sub>	972 <sub>(s)</sub>	551 <sub>(m)</sub> 531 <sub>(s)</sub>

Fig.1.02 :- FTIR Spectra of Chalcone Ligand :

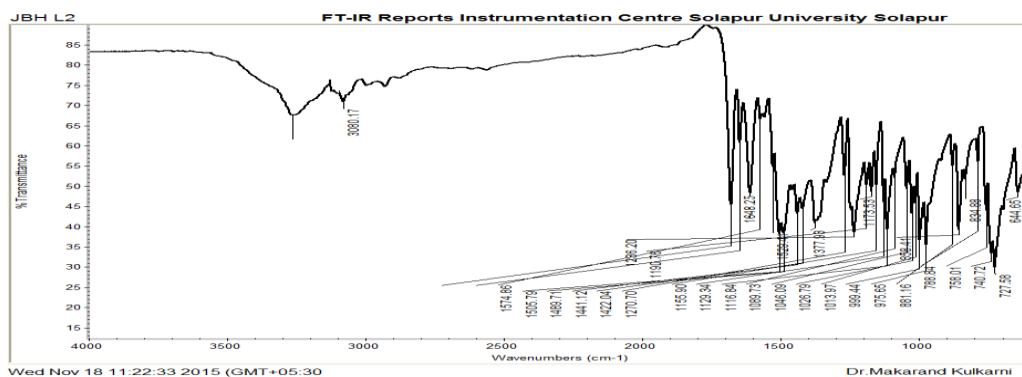
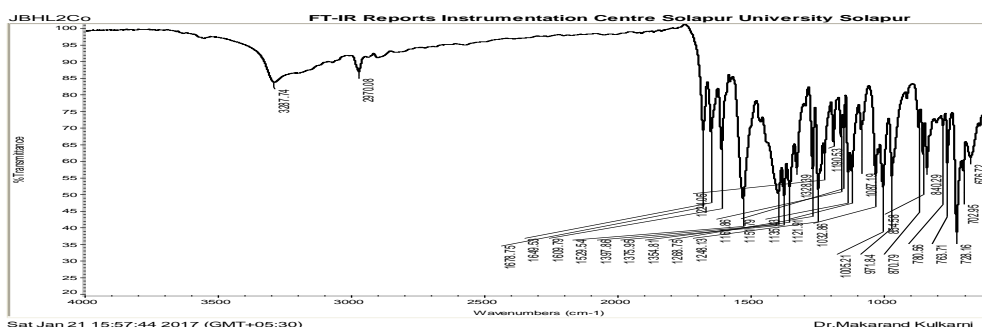


Fig.1.03 :- FTIR Spectra of ComplexCobalt with ligand :



V)Magnetic Measurements And UltravioletSpectra :

The magnetic susceptibility and UV spectral facts are specified in Table 1.03. The UV-visible spectrum of the copper(II) chelate in dimethylformamide discovered one broad band at 15130 and 25226 cm<sup>-1</sup> for Chalcone ligand, predictable to an<sup>2</sup>E<sub>g</sub> → <sup>2</sup>T<sub>2g</sub> transition and charge transfer spectra. The practical magnetic susceptibility value for the copper(II) chelate remained in the 2.08 μ<sub>B</sub>. The UV spectroscopic facts<sup>16</sup> combined by the magnetic value propose a distorted octahedral confirmation for the copper(II) chelates<sup>1,2,17</sup>. The ultraviolet spectrum of Nickel(II) chelate spectacle three bands at 9412, 15622 then 24200 cm<sup>-1</sup> for ligand, predictable to the <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>2g</sub>(F) (v<sub>1</sub>), <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>1g</sub>(F) (v<sub>2</sub>) also <sup>3</sup>A<sub>2g</sub> → <sup>3</sup>T<sub>1g</sub>(P) (v<sub>3</sub>) alterations. This is in conformity by previously described values for octahedral Nickel(II) chelates<sup>1,2,18,19</sup>. The nickel(II) chelate magnetic moment is 3.03 μ<sub>B</sub> checks the suggested geometry. On the accordance this value the range stated for octahedral confirmation. The Cobalt(II) chelate display three bands in the range of 9559, 18448 and 22665 cm<sup>-1</sup> for ligand predictable to <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>2g</sub>(F) (v<sub>1</sub>), <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>A<sub>2g</sub>(F) (v<sub>2</sub>) and <sup>4</sup>T<sub>1g</sub>(F) → <sup>4</sup>T<sub>1g</sub>(P) (v<sub>3</sub>) transitions<sup>1,2,17,20</sup>. The magnetic moment of the Cobalt(II) chelate is 4.66 μ<sub>B</sub> it proposed octahedral configuration.

Table1.03. Magnetic Susceptibility and UV spectra records (in DMSO) of the ligand and complexes.

Ligand and Complex	ν /cm <sup>-1</sup>	Band assignment	μ <sub>eff</sub> / μ <sub>B</sub>	Geometry
Ligand HL C <sub>17</sub> H <sub>13</sub> NO <sub>4</sub>	32442 40545	INCT <sup>a</sup> INCT	-	-
C <sub>34</sub> H <sub>28</sub> ClCoN <sub>2</sub> O <sub>9</sub>	9559 18448 22665	<sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>2g</sub> (F)(v <sub>1</sub> ) <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> A <sub>2g</sub> (F)(v <sub>2</sub> ) <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P)(v <sub>3</sub> )	4.66	Octahedral
C <sub>34</sub> H <sub>30</sub> CuN <sub>2</sub> O <sub>10</sub>	15130 25226	<sup>2</sup> E <sub>g</sub> → <sup>2</sup> T <sub>2g</sub> INCT	2.08	Distorted Octahedral
C <sub>34</sub> H <sub>30</sub> FeN <sub>2</sub> O <sub>10</sub>	14546 21801 24449	<sup>6</sup> A <sub>1</sub> → <sup>4</sup> T <sub>1</sub> (G) <sup>6</sup> A <sub>1</sub> → <sup>4</sup> T <sub>2</sub> (G) <sup>6</sup> A <sub>1</sub> → <sup>4</sup> E(G)	5.92	Distorted Octahedral
C <sub>34</sub> H <sub>30</sub> MnN <sub>2</sub> O <sub>10</sub>	17789 19571 31060	Laporte and spin forbidden	5.77	Distorted Octahedral
C <sub>34</sub> H <sub>30</sub> N <sub>2</sub> NiO <sub>10</sub>	9412 15622 24200	<sup>3</sup> A <sub>2g</sub> → <sup>3</sup> T <sub>2g</sub> (F)(v <sub>1</sub> ) <sup>3</sup> A <sub>2g</sub> → <sup>3</sup> T <sub>1g</sub> (F)(v <sub>2</sub> ) <sup>3</sup> A <sub>2g</sub> → <sup>3</sup> T <sub>1g</sub> (P)(v <sub>3</sub> )	3.03	Octahedral

The Iron(III) chelate of Chalcone ligand appearance three bands at 14546, 21801, 24449 cm<sup>-1</sup> predictable to the <sup>6</sup>A<sub>1</sub> → <sup>4</sup>T<sub>1</sub>(G), <sup>6</sup>A<sub>1</sub> → <sup>4</sup>T<sub>2</sub>(G) also <sup>6</sup>A<sub>1</sub> → <sup>4</sup>E(G) transitions. The UV spectra put forward distorted octahedral geometry<sup>1,2,20,21</sup>. The ultraviolet spectrum of Manganese(II) chelate of ligand spectacle feeble bands at the 17789, 19571 plus 31060 cm<sup>-1</sup>. These

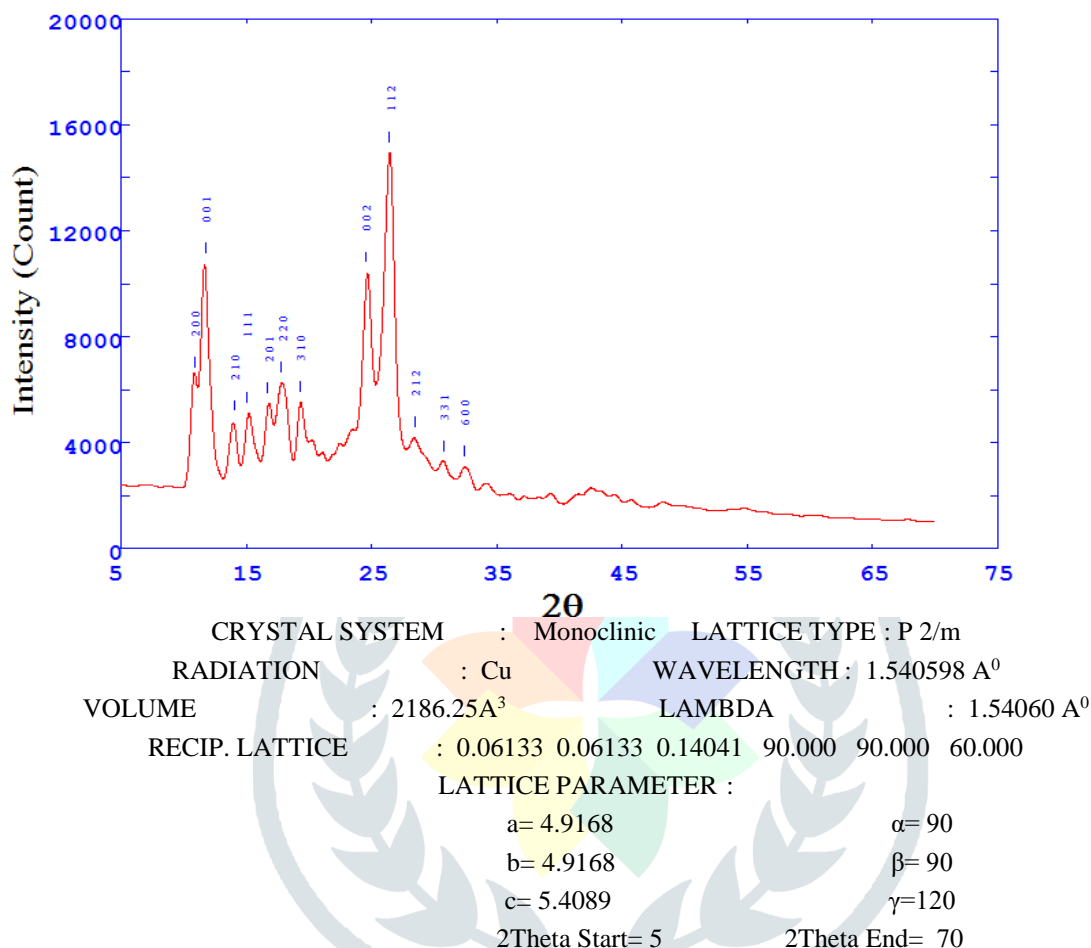
transitions are together Laporte plus spin-forbidden. On the other hand, caused by prompt distortion of the octahedral configuration nearby the metal cation, feeble transitions occasionally do seem<sup>1,2,17,18</sup>.

#### VII) XRD Study of Chalcone Ligand and its Metal Complexes :

The X-ray diffraction studies of newly prepared Copper(II), Nickel(II), Cobalt(II) Manganese(II) and Iron(III) complex with ligand was taken from Instrumentation Centre of Solapur University, Solapur. The powder X-ray diffractograms of the all chelates was recorded in  $2\theta$  range of  $5-70^\circ$  by copper Ka radiation initiator at a wavelength of  $1.540598 \text{ \AA}$  at normal temperature.

The leading reflexes remained used to conclude resultant inter planar distances. The XRD was then indexed individually, miller indices were measured, lattice parameter a, b, c in addition to interfacial angles  $\alpha$ ,  $\beta$ ,  $\gamma$  remained define by powder-X computer software programme<sup>1,2</sup>. The above X-ray diffraction data of transition metal complex has been tabulated in the following Fig.1.04 and tables 1.04 are as follows.

**Fig. 1.04 : X-ray Diffractogram of Cu-Ligand :**



**Table 1.04 :XRD data of Cu-Ligand Complex**

H	K	L	TH(OBS)	TH-ZERO	TH(CALC)	D(OBS)	D(CALC)	RI%
2	0	0	5.41986	5.62366	5.42190	7.86069	8.15228	33.2
0	0	1	5.87839	6.08218	6.20896	7.27008	7.12219	54.2
2	1	0	7.02469	7.22848	7.18058	6.12192	6.16254	23.1
1	1	1	7.52906	7.73285	7.79464	5.72482	5.67971	23.6
2	0	1	8.35440	8.55819	8.25715	5.17626	5.36360	24.2
2	2	0	8.90463	9.10842	9.41937	4.86597	4.70672	30.0
3	1	0	9.63826	9.84205	9.80771	4.50645	4.52207	24.1
0	0	2	12.25183	12.45562	12.49237	3.57143	3.56110	52.8
1	1	2	13.21473	13.41852	13.37187	3.31936	3.33073	76.5
2	1	2	14.22348	14.42727	14.46734	3.09170	3.08332	17.4
3	3	1	15.36978	15.57357	15.56064	2.86916	2.87149	13.4
6	0	0	16.19512	16.39891	16.46720	2.72843	2.71743	12.4

From the X-ray diffraction spectrum analyzed data were illustrated in above Table 1.04 that the Cu(II) complex have a Monoclinic crystal system<sup>22</sup>.

#### Antimicrobial Activity :

Antimicrobial activity was assayed by paper disc plate method by measuring inhibition zones in mm. in vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against four strains of bacteria which include *Bacillus Megaterium*, *Bacillus Cereus* and Gram-negative i.e. *Escherichia Coli*, *Shigella boydii* and against three

fungal strains like *Penicilliumnotatum*, *Saccharomyces Cerevisiae* and *AspergillusOryzae*. The standard used was *Ciprofloxacin* and *Grysofulvin*.

Compound	Inhibition zone of bacterial & fungal growth in mm						
	Antimicrobial activity				Antifungal activity		
	<i>Bacillus Megaterium</i>	<i>Bacillus Cereus</i>	<i>Escherichia Coli</i>	<i>Shigella boydii</i>	<i>Penicilliumnotatum</i>	<i>Saccharomyces Cerevisiae</i>	<i>AspergillusOryzae</i>
Conc <sup>n</sup> ,1m g/ ml	Conc <sup>n</sup> ,1 mg/ ml	Conc <sup>n</sup> ,1m g/ ml	Conc <sup>n</sup> ,1m g/ ml	Conc <sup>n</sup> ,0.5mg/ ml	Conc <sup>n</sup> ,0.5mg/ ml	Conc <sup>n</sup> ,0.5mg/ ml	
Ligand HL C <sub>17</sub> H <sub>13</sub> NO <sub>4</sub>	10	08	08	12	04	-	02
C <sub>34</sub> H <sub>28</sub> ClCo N <sub>2</sub> O <sub>9</sub>	22	12	19	13	12	10	10
C <sub>34</sub> H <sub>30</sub> CuN <sub>2</sub> O <sub>10</sub>	24	14	24	23	14	12	14
C <sub>34</sub> H <sub>30</sub> FeN <sub>2</sub> O 10	20	16	08	12	10	-	-
C <sub>34</sub> H <sub>30</sub> MnN <sub>2</sub> O <sub>10</sub>	26	20	10	16	11	18	16
C <sub>34</sub> H <sub>30</sub> NiN <sub>2</sub> O 10	16	14	18	18	12	20	18
<i>Ciprofloxacin</i>	36	54	32	30	-	-	-
<i>Grysofulvin</i>	-	-	-	-	34	40	42

From the results of antimicrobial activity of ligand and complex it is clear that the complexes shows enhance activity than the ligands. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligand<sup>23</sup>.

#### IV. CONCLUSION

Preparation of Chalconeligand, physicochemical and spectroscopiclikes UV, <sup>1</sup>H-NMR, IR and Mass was studied.<sup>1</sup>H-NMR spectrum suggest decent involvement of trans hydrogen in CH=CH vinyl moiety as chalcone formation.Molecular ion forms at equal to the mass of corresponding ligandthis was a strong evidence for the formation of the ligand.The Chalconeligand has one phenolic –OH group from Dehydroacetic acid (DHA) and one α,β unsaturated carbonyl moiety which are connected to the Indole ring. The chalcone ligand forms metal complex having coordination integer six. ChalconeLigand acts as uninegativebidentate in nature.The complexes remain insoluble in common organic solvents, however soluble in Dimethylsulfoxide as well as Dimethylformamide. Ultraviolet spectrum values of the complexes shifted towards lower or higher frequencies compare to the ChalconeLigand, which confirm the chelation of the ligand to the transition metal ions.Studies of FTIR spectrum of the chelates tells that the Chalcone Ligand coordinates to transition metal ions via phenolic –OH group, carbonyl oxygen as well as coordinated water molecules. Existence of metal-oxygen stretching vibrations was confirmed by this type bonding in chelates.The X-ray diffractogramoutcomes of the chelates shown monoclinic crystal system for Cu(II), Ni(II), Mn(II) and Co(II) chelates while hexagonal for Fe(III) chelate with P-type lattice.

The analytical data shows 1:2 metal to ligand stoichiometry, we have proposed distorted octahedral geometry for Cu(II), Mn(II) & Fe(III), other Ni(II), Co(II) are octahedral geometry. Antimicrobial activity it is found that the complexes are more active than their parent ligand.

#### V. ACKNOWLEDGMENT

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#### REFERENCES

- Balaji H. Jawale and shridhar D. salunke, Research Journal of Pharmaceutical, Biological and Chemical Sciences,2017,8(2),1260.
- Patangevaibhav, Ph.D Thesis, Dr.B. A. M. University, Aug 2008.
- Prasad, Y.R., Rao, A.L., Rambabu, R., and Kumar, P.R. Oriental J. Chem., 2007;3:927-937.
- Rao, S.M., Kotesch, J., Marukulla, R., and Duddeck, H. ARKIVOC 2004;xiv;96-102.
- Patange,V.N., B.R. Arbad, V.G. Mane & S.D. SalunkeTrans Met. Chem.2007;32; 944.

6. Munde A.S., Jagdale, A.N., Jadhav,S.M., Chondhekar, T.K., J.Korean Chem. Soc.2009;53;4.
7. Thornberry, H.H.,*Phytopathology* 1950;40;419.
8. Bauer, A.W.,W.M.M. Kirby, J.C. Shesies, M. Turck, *Am. J. Clin. Pathol.* 1966;44;93.
9. R.Gren, *Dent.Apoth.Ztg*, 111 (1971) 219.
10. S.Forsen and M.Nilsson, *Acta.Chem.Scand*, 14 (1960) 1333.
11. V.Drevenkar, A.Deljac and Z.Stefanac, *Polyhedron*, 2(16) (1983) 447-453.
12. Ramarao,N., V.P. Rao, V.J. TyagaRaju, M.C. Ganorkar, *Indian J.Chem.*1985;A24;877.
13. Carugo,O., C.B. Castellani, M.Rizzi, *Polyhedron* 1990;9;2061.
14. Rao,P.V., A.V. Narasaiah, *Indian J. chem. A* 2003;42;1896.
15. Nakamoto,K., *Infrared spectra of inorganic & coordination compounds*, Wiley, New York, 1970;159;167;214.
16. Eichhorn G. L., J. C. Bailar, *J. Am. Chem. Soc.* 1953;75;2905
17. Figgis B. N., *Interscience*, London, 1966;56;654.
18. Lever A. B. P., *Inorganic Electronic Spectroscopy*, Elsevier, Amsterdam, 1968;89;789.
19. Satpatty K. C., Panda A. K., Mishra R., Pande I., *Transition Met. Chem.* 1991;16;410.
20. Gudasi K. B., Patil S. A., Vadavi R. S., Shenoy R. V., Patil M. S., *J. Serb. Chem. Soc.* 2006;71;529.
21. Prasad R. N., Mathur M., *J. Serb. Chem. Soc.* 2002;67; 825.
22. AmelDjedouani, AbderrahemBendaas, SoflaneBoucida, Adel beghidja and TaharDouadi, *Acta.Cryst*, 62E 2006 m133-m135.
23. Harsfall J.G, *Bot Rev.* 1945;11:357.

