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X-Ray Diffraction and Anti-Microbial Study of Synthesized Ru(II) and Rh(II) Complexes with Ciprofloxacin

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Abstract: *New two metal complex from Ru(III) and Rh(III) with ciprofloxacin where synthesized in proportion of 1:2. Synthesized complexes where analyzed by elemental analysis, which help in predicting molecular formula of complexes. X-ray diffraction data help in analyzing atomic arrangement inside the co-ordination complexes. Infrared spectral data give idea about coordinating atom with Ru(III) and Rh(III). The synthesized metal complex was evaluated for in-vitro antibacterial and antifungal activity against the drug resistant pathogens such as Pseudomonas aeruginosa, E. coli, Proteus vulgaris, Streptococcus pneumoniae, Vibrio cholerae, Salmonella typhi, Aspergillusniger, and Candida albicans. The metal complex showed the significant antibacterial and antifungal activity comparison with commercial antibiotics.*

Keywords: Ciprofloxacin, Metal, Antimicrobial, Antifungal, X-ray Diffraction.

I. INTRODUCTION

Coordination chemistry is important role in medical field after the discovery of Cis-platin as Anti-cancer drug. From this important milestone researcher are interested to develop drug by forming its complexes. In coordination complexes metal ion directly connected to ligand through coordinate bond to form stable coordination complexes. Ciprofloxacin drug is good ligand which form chelate ring around central metal ion to form stable complex.

The application of quinoline and its derivatives in areas of medicine, food, catalyst, dye, materials, refineries and electronics is well known [1]Metal ion plays very important role in healthy growth of human body and various biological process . Metal drug coordination complexes are more biologically active than that of standard drug molecule[2]. In many cases coordinated drug with metal ion is more active than that of original drug molecule[3]. It was found that metal ligand shows biological activity viz., antibacterial, anti-fungicidal, antiviral and anticancer activity.i.e. cis-platin[4]. Due to these reason I try to synthesize Rh(III) and Ru(III) complexes with ciprofloxacin drug and to check their anti-microbial activity against drug resistant bacterial species.

II. EXPERIMENTAL

2.1 Synthesis of Metal Complexes

Previously reported method utilized by some modification (Ogunniran et al., 2008; Osowole et al., 2015 Nadira, 1987, Galvan–Tejada, 2002).A hot ethanolic solution of Ciprofloxacin Drug (10 mmol, 3.14 gm) was added to ethanolic solution $Ru(Cl)_3 .5H_2O$(5mmol, 1.45gm). The resulting reaction mixture was refluxed for 6 hours. After cooling, the colored precipitate obtained was collected, filtered, recrystallized from ethanol and dried over anhydrous CaCl2 in desiccator. The metal drug coordination complexes are insoluble in common polar organic solvents and soluble in DMF, DMSO[5-10].

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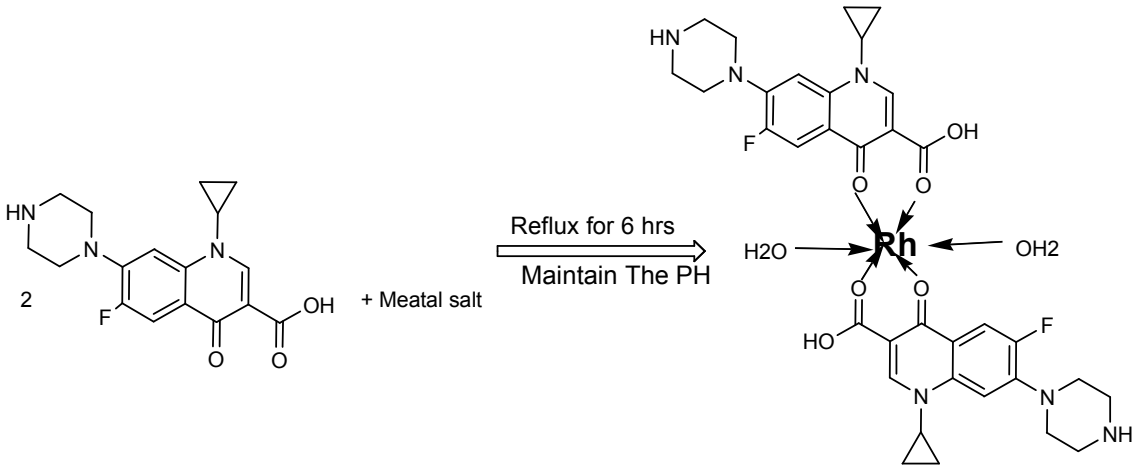
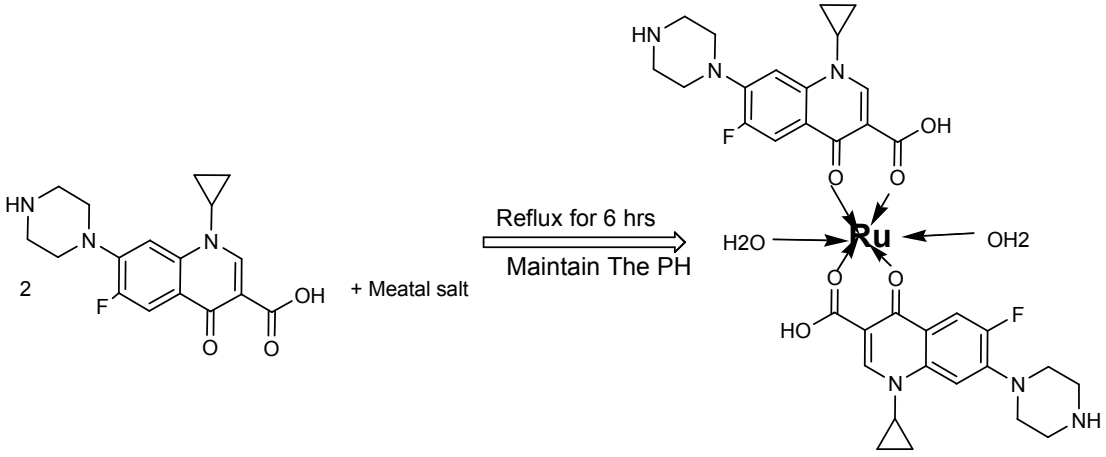


ciproflxacin Rh(III) complexes



ciproflxacin Ru(III) complexes

Physical, analytical, molar conductivity data and magnetic moment of Ciprofloxacin with Ru(III) and Rh(III) complexes.

Physical data of metal drug coordination complexes indicates formation of complexes in which melting point increased it indicates the stability of complexes and color indicates formation of complexes. Molar conductance indicates ionic nature of coordination complexes in which RU(III) and Rh(III) complexes shows higher conductance as compare to reaming coordination complexes given in **Table No.01**. The calculated elemental analysis of coordination complexes and obtained data is having same percentage composition.

Table 1: Elemental analysis, molar conductance and physical parameters

Compounds	Mol. Wt.	Colour (%Yield)	M.P.	Molar Cond. Ohm^{-1} cm^2 mol^{-1}	% Found (cald)					
					C	H	N	F	O	M
Ciprofloxacin (L)	331.34	White	289	---	(61.62)	(5.48)	(12.68)	(5.75)	(14.49)	--
$[Ru(L)_2(H2O)_2]Cl_3$	799.79	Brown (72)	292	17.04	52.01 (51.06)	4.93 (5.04)	10.32 (10.51)	4.52 (4.75)	15.66 (16)	12.98 (12.64)

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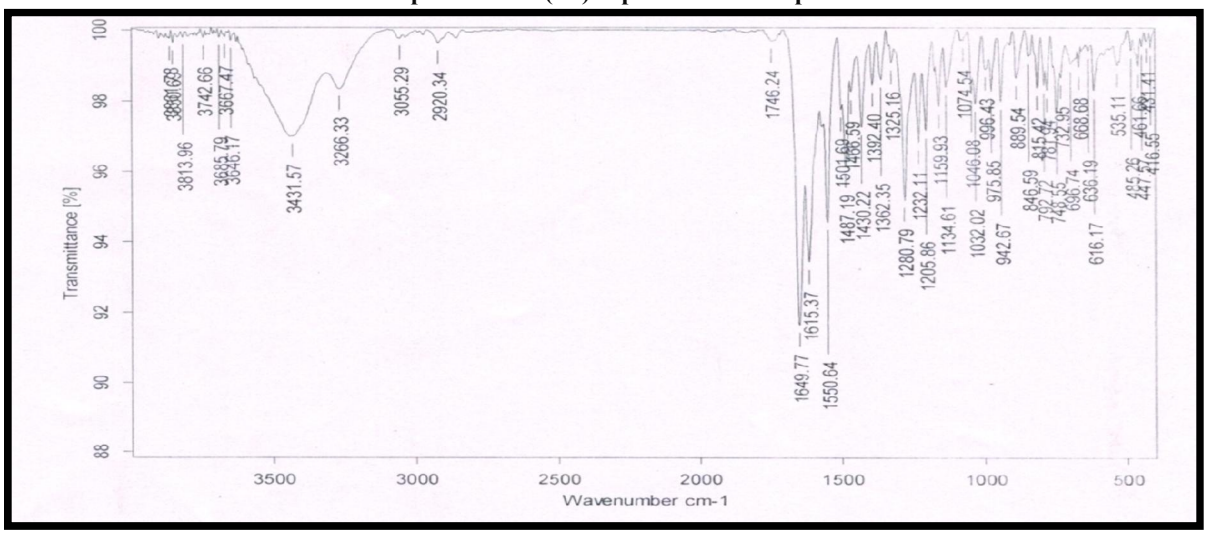
$[Rh(L)_2(H2O)_2]Cl_3$	801.63	Orange (69)	310	19.4	51.02 (50.94)	4.82 (5.03)	10.04 (10.48)	4.54 (4.74)	15.67 (15.97)	12.65 (12.84)

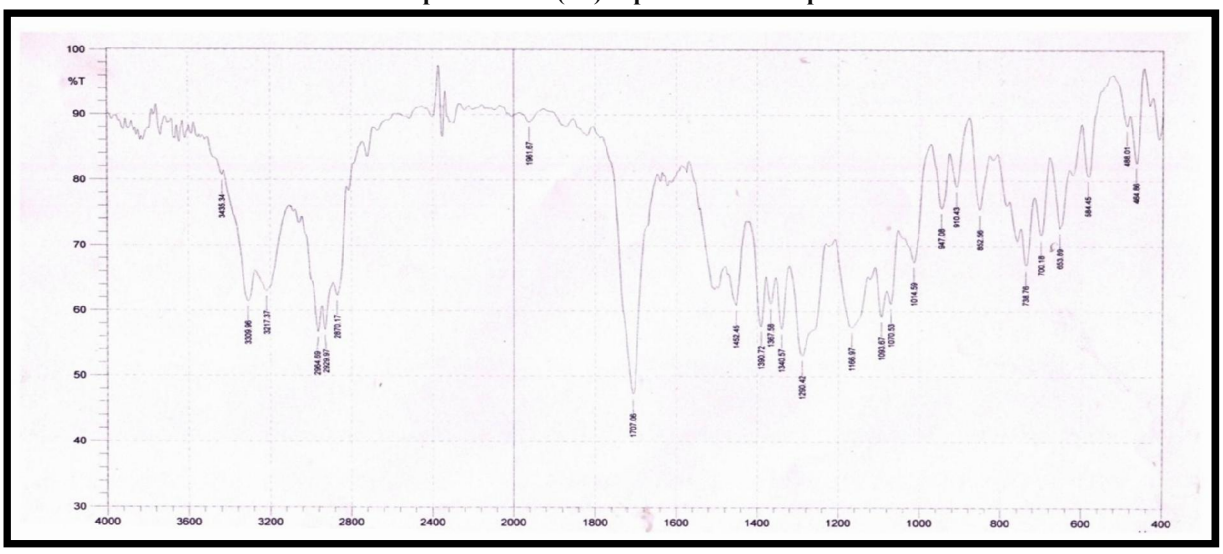
2.2 FT-IR Spectra

The IR spectral data of complexes gives good information about coordination mdes of metal ligand. The IR spectral data of synthesized coordination complexes are given in figure 1 and 2. In all the complexes, a new band is seen in the ≈ 538 - 546 cm−1 region, which is probably due to the formation of the weak band observed in the ≈ 430 - 455 cm–1 range can be attributed to ν (M-O).The IR spectral values is in 600-400 cm-1 clearly indicate that formation coordinate through oxygen atom of ligand to metal**.**

IR spectra of Rh(III) Ciprofloxacin complex



IR spectra of Ru(III) Ciprofloxacin complex



2.3 XRD data of Metal Ciprofloxacin Complexex

Powder XRD pattern of all the complexes were recorded over the 2ɵ = 05-85^0 range. Unit cell parameters were found by using trial and error method. Metal drug complexes are monoclinic and triclinic with same and different unit cell parameter respectively. The observed unit cell parameters are given in table and considering JCPDS software data (joint committee of

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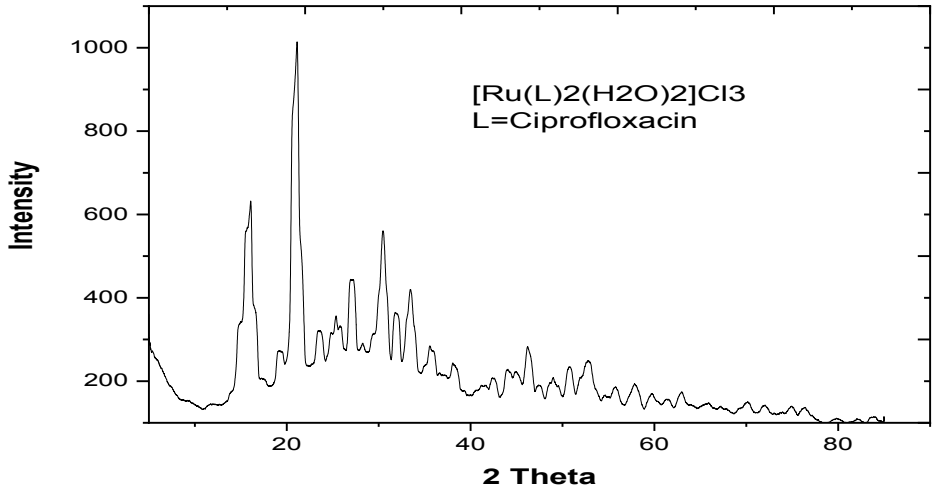
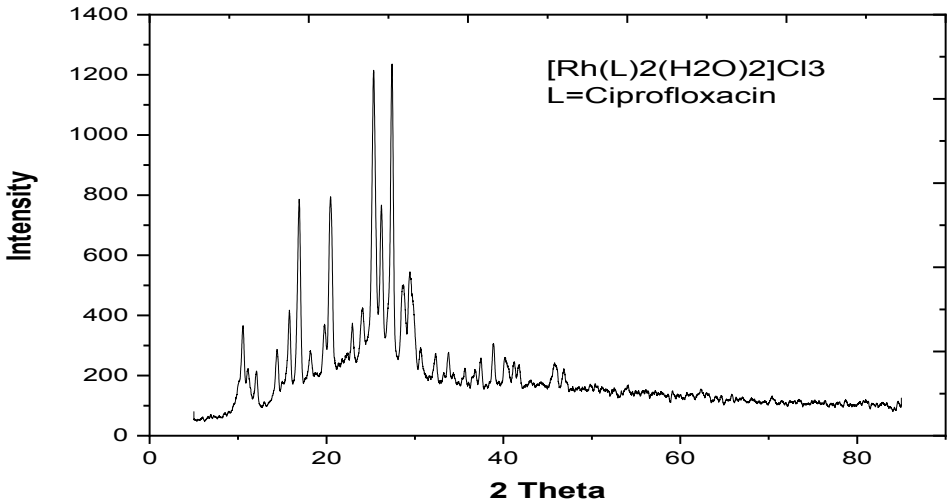
powder diffraction standard), ICDD (International center of diffraction data). The powder XRD patterns are shown in figure **13 to 18**.

Compounds	Lattice constant			α(A^0)	β(A^0)	ϒ(A^0)	2Θ	D value	Crystal system
	a(A^0)	b(A^0)	c(A^0)						
$[Ru(L)_2(H2O)_2]Cl_3$	9.31	6.4	5.8	105.4	97.7	101.9	9.32	6.67	Triclinic
$[Rh(L)_2(H2O)_2]Cl_3$	9.8	5.8	5.1	105.9	96.4	101.6	7.64	7.72	Triclinic

XRD spectra of Ru(III) Ciprofloxacin complex



XRD spectra of Rh(III) Ciprofloxacin complex



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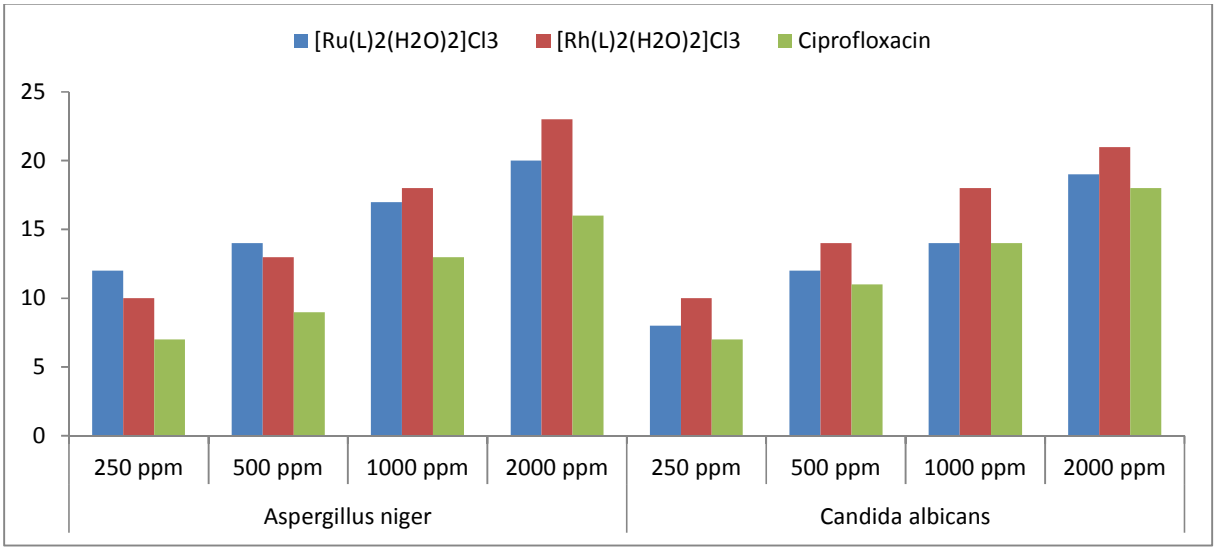
2.4 Antimicrobial Activity

The synthesized Metal ligand complexes were tested *in vitro* for their antibacterial activity against Gram(+ve) and Gram (-ve) bacteria, where antifungal against *Aspergillus niger and candida albicans* using the broth dilution method. Nutrient agar was prepared in distilled water while, pH of the solution was adjusted to neutral and sterilized in autoclaving at 15 lbpres-sure for 15 mins. The tested bacterial and fungal strains were prepared and incubated at 37°C overnight.

Anti-fungal activity ciprofloxacin with Ru(III) and Rh(III) complexes

Anti-fungal activity ciprofloxacin with Cu(II) , Ni(II), Co(II), Fe(II), Ru(III) and Rh(III) complexes given in **Table** As per data given in table shows that metal ciprofloxacin complexes are having more potent than that of the drug[11-14]. Rh(III) and Ru(III) ciprofloxacin complexes shows high zone of inhibition against *Aspergillus niger and Candida albicans.*

Complexes	*Aspergillus niger*				*Candida albicans*			
	250 ppm	500 ppm	1000 ppm	2000 ppm	250 ppm	500 ppm	1000 ppm	2000 ppm
$[Ru(L)_2(H2O)_2]Cl_3$	**12**	**14**	**17**	**20**	**8**	**12**	**14**	**19**
$[Rh(L)_2(H2O)_2]Cl_3$	**10**	**13**	**18**	**23**	**10**	**14**	**18**	**21**
Ciprofloxacin	**7**	**9**	**13**	**16**	**7**	**11**	**14**	**18**



Anti-Bacterial Activity Ciprofloxacin with Ru(III) and Rh(III) Complexes

Anti-bacterial activity ciprofloxacin with Ru(III) and Rh(III) complexes against Gram positive and Gram negative bacterial species[15-18]. All complexes shows very good zone of inhibition as compare to the standard ciprofloxacin molecule. Anti-bacterial data of ciprofloxacin metal complexes given in **table.**

Bacterial species	$[Ru(L)_2(H2O)_2]Cl_3$				$[Rh(L)_2(H2O)_2]Cl_3$				Ciprofloxacin 500 ppm
	250 ppm	500 ppm	1000 ppm	2000 ppm	250 ppm	500 ppm	1000 ppm	2000 ppm	
Pseudomonas aeruginosa	**4**	**7**	**12**	**16**	**9**	**15**	**19**	**23**	**5**
E. coli	**7**	**9**	**14**	**18**	**4**	**7**	**11**	**17**	**6**
Proteus vulgaris	**5**	**7**	**12**	**19**	**4**	**6**	**11**	**16**	**4**
Streptococcus pneumoniae	**8**	**14**	**19**	**22**	**6**	**9**	**16**	**21**	**7**
Vibrio cholerae	**10**	**13**	**18**	**22**	**9**	**14**	**19**	**23**	**8**
Salmonella typhi	**12**	**15**	**19**	**26**	**8**	**11**	**17**	**20**	**10**

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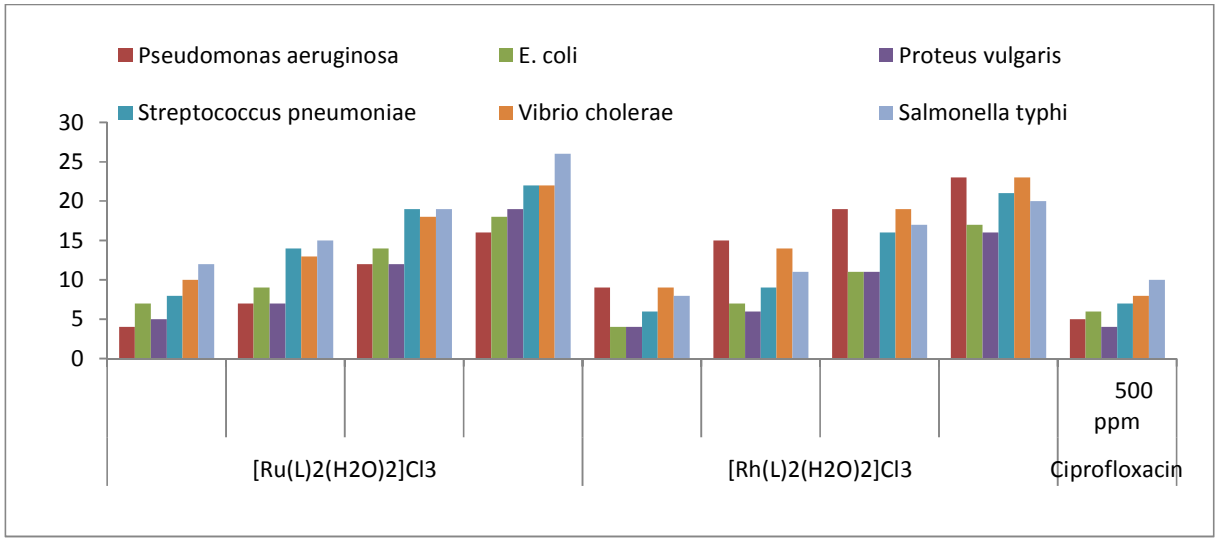

III. Conclusion

The elemental analysis of ciprofloxacin metal complexes gives idea about coordination spears of synthesized complexes in stoichiometry ratio1:2. Although all complexes obtained in good yield and their solubility is in DMSO and DMF. Ciprofloxacin metal complexes is having higher melting point than that of parent drug molecules, it indicates that metal complexes are having higher stability. In Infra Red spectra of complexes their will broad band's obtained in 500-400cm^{-1} range it gives clear idea about formation of M-O bond in complexes, decrease in C=O bond frequencies indicates that carbonyl oxygen form M-O bond. XRD data of metal ciprofloxacin complexes gives us idea about arrangement and position of atom in coordination complexes. XRD data of Ru(III), Rh(III) complexes are triclinic Crystal system.

Metal ciprofloxacin complexes shows more potency against microbial and fungal species compare to standard drug molecules, to be specify Rh(III) and Ru(III) ciprofloxacin complexes shows high zone of inhibition against *Aspergillus niger and Candida albicans a*nd All complexes shows very good zone of inhibition as compare to the standard ciprofloxacin molecule.

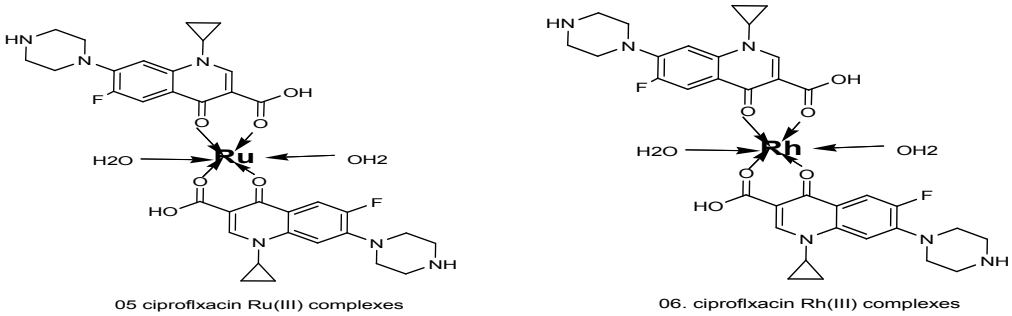

Figure: Structure of Ru(III) and Rh(III) ciprofloxacin complex

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