


# anveṣak

A bi-annual journal

2021



PRINCIPAL  
Lotnate Gopinathji Munde  
Arts, Commerce & Science College  
Mandangad Dist. Ratnagiri-415203



SARDAR PATEL INSTITUTE OF  
ECONOMIC AND SOCIAL RESEARCH

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Vol. 51, No.1(V) January - June 2021



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## X-RAY DIFFRACTION STUDY OF SYNTHESIZED Cu (II), Ni (II), Co (II) & Fe (II) ISONIAZID CO-ORDINATION COMPLEX AND THEIR ANTI-MICROBIAL ACTIVITY

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**Abstract:** X-ray diffraction studies have been undertaken using powder technique to determined lattice parameters, crystal system, crystal lattice intercept, angle etc. X-ray powder diffraction of Cu (II), Ni (II), Co (II) & Fe (II) metal complexes with isoniazid shows monoclinic and triclinic system with various unit cell parameters. The Cu (II), Ni (II), Co (II) & Fe (II) Isoanizid complexes show more intense band on spectrogram it indicates that closed pack position of atom in crystal system. Anti-bacterial activity of synthesized drug metal complexes shows considerable activity against bacteria in comparison with standard drug molecule.

**Key Words:** crystal system, complexes, X-ray diffraction, unit cell, standard drug etc.

### Introduction

A metal atom after forming complexes with drug alters the activity of drug alone. Number of metal based compounds with potential medical application has been developed and some of them approved for clinical use during the last decades. A metal drug complex gives us good platform for designing new potent drug which helps us to fight against drug resistant bacteria. The literature study reveals that drug forms stable coordination complexes with metal through hetero atom. I can choose the metal from transition series because they show variable oxidation states, variable coordination number as well as geometry of the complexes. Drug molecule some time undergoes biological redox reaction in vivo, this phenomenon help us to design potential diversified metal drug complexes. This heightened interest in inorganic medicinal chemistry is due to both prior successful drugs approved for clinical use (e.g., cisplatin, auranofin, carboplatin, nitroprusside, silver sulfadiazine) [01-02] Tuberculosis kills up to 3 million people every year in the world. Tuberculosis caused by Mycobacterium tuberculosis (MTb), is the second leading cause of death from an infectious disease and it is surpassed by the human immunodeficiency virus (HIV) [03].

Since 1950 isoniazid has used as first line drug in treatment of tuberculosis i.e. antituberculostatic and antibacterial properties [04-06]. The discovery of these properties has enabled further research on isoniazid and its derivatives [05]. WHO declared TB as a global emerges but since that time, no new drug has successfully been developed for the treatment of the disease [03]. With the global emergence of drug resistant bacteria strain, there for an urgent need to develop more potent and fast acting anti-TB drugs with new modes of action to overcome the cross-resistance with current drugs and low toxicity profiles that can be tolerated for long period of treatment [07-08]. Thus it is need to develop highly effective metal based drugs for the complete removal of TB. The researchers now a day are try to develop Isoanizid containing new metal based drug that would be more effective against TB.

Before 1912 there was still no direct techniques for the structure elucidation of crystals but in 1912 came the great turning point in crystallography i.e. the German Physicist Max Laue suggested that crystals might serve as diffraction gratings for X-rays. Friedrich and Knipping carried out an experiment to test Laue's suggestion and irradiated a crystal of  $CuSO_4 \cdot 5H_2O$  with X-rays. X-rays have the appropriate wavelength (in the A range  $\sim 10^{-8}$  cm) to be scattered by the electron cloud of an atom of comparable size. XRD scattering pattern of the molecules or atoms in the crystal of diffraction pattern which help us in the electron density reconstruction and position of atom in crystal. The detection of diffraction confirmed Laue's suggestions and launched the science of X-ray crystallography. X-ray crystallography is an experimental technique that used to check atomic arrangement in crystal by X-rays diffracted from crystals [09-12]. Literature survey shows that transition metal complexes generally crystalline as well as amorphous in nature with octahedral, tetrahedral or square planar geometry [13-14]. The X-ray diffraction is to technique used for determination of the probable structure of the metal coordination complexes. From this interference



pattern able to express mathematically the nature of the interference pattern in terms of the atomic position within the crystal [15]. In present research work paper I take powder XRD of Cu (II), Ni (II), Co (II) and Fe (II) complexes of isoniazid scanned in the range of 0-80 at wavelength 1.540598 Å.

Biological analysis of complexes was tested on microbial species like *Plasmodium falsiparum*, *Proteus vulgaris*, *E. coli*, *Streptococcus pneumonia*, *Bacillus*, *Staphylococcus aureus*, *Mycobacterium tuberculosis* species.

### Material and Methods

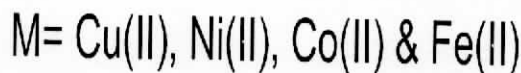
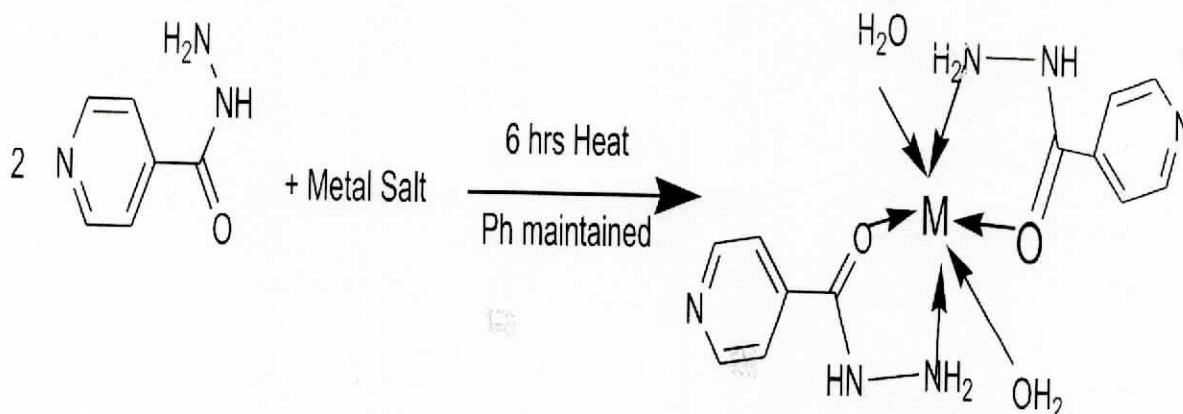
All chemicals and solvents used were of A. R. grade and used as received. The transition metal salts (Metal chlorides) obtained from Loba chem. The powder XRD was recorded on Perkin Elmer at the range of 0-80° at wavelength 1.540598 Å. The gram-positive and gram-negative strains used antimicrobial activity which was obtained from the microbiology Dept. of KDB College, Ghuhagar (Ratnagiri).

**Preparation of Cu (II) complexes:** 10 ml Ethanolic solution (1 gram) of  $\text{CuCl}_2 \cdot 5\text{H}_2\text{O}$  and About (2.5 gram) of isoniazid was weighed accurately dissolved in same ethanol solution. This mixture was refluxed for six hours in a heating mantle. After six hours solid precipitate formation takes place, precipitate dry in by evaporating solvent in desiccator containing calcium carbonate.

**Preparation of Ni (II) complexes:** Ethanolic solution (1 gram) of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  and About (2.5 gram) of isoniazid was weighed accurately dissolved in same ethanol solution. This mixture was refluxed for six hours in a heating mantle. After six hours solid precipitate formation takes place, precipitate dry in by evaporating solvent in desiccators containing calcium carbonate.

**Preparation of Co (II) complexes:** Ethanolic solution (1 gram) of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  and About (2.5 gram) of isoniazid was weighed accurately dissolved in same ethanol solution. This mixture was refluxed for six hours in a heating mantle. After six hours solid precipitate formation takes place, precipitate dry in by evaporating solvent in desiccator containing calcium carbonate.

**Preparation of Fe (II) complexes:** Ethanolic solution (1 gram) of  $\text{FeSO}_4 \cdot 6\text{H}_2\text{O}$  and About (2.5 gram) of isoniazid was weighed accurately dissolved in same ethanol solution. This mixture was refluxed for six hours in a heating mantle. After six hours solid precipitate formation takes place, precipitate dry in by evaporating solvent in desiccator containing calcium carbonate. All above complexes are insoluble in organic solvents such as acetone, chloroform but soluble in DMF and DMSO.



## Schme:-preparation of cordination complex

**Preparation of Nutrient agar plate:** Nutrient agar was prepared by adding 1 gm of pepton, 1gm meat extract, 0.8 gm sodium chloride, 1.2 gm agar-agar in 100ml distilled water in 250ml conical flask. Cotton plunge and wrapped by silver paper and sterilize this conical flask at 110° C (15lb pressure for 15 min) in Autoclave. This nutrient medium was pour on sterilized plate and solidifies it. After solidification plate was used for anti-microbial activity.

### Result and discussion:

Isoniazid complexes are colored solid and synthesized on quantitative basis. Conductivity data of synthesized complexes are indicates polar character of complexes. Higher Melting point of complexes shows that Stability of complexes. Complexes are soluble in organic solvent like DMSO. Composition of complexes was determined by elemental analysis technique. Physical/analytical parameter of metal isoniazid coordination complexes are given in

Table No.1.

Compounds	Mol. Wt.	Colour (%Yield)	M.P.	Molar Cond.oh $m^2 cm^{-2} mol^{-1}$	% Found (calculated)				
					C	H	N	O	M
Ligand (L)	137.13	White	171.4	--	(52.55)	(5.15)	(30.64)	(11.67)	-----
C1 complex	373.86	Green (76)	289	14.05	38.08 (38.55)	5.01 (4.85)	22.13 (22.48)	17.84 (17.12)	16.88 (17)
C2 complex	369.00	puple (84)	296	17.6	38.96 (39.06)	5.01 (4.92)	23.02 (22.78)	16.98 (17.34)	16.02 (15.91)
C3 complex	369.24	Orange (70)	288	19.4	39.12 (39.03)	4.81 (4.91)	22.54 (22.76)	17.43 (17.33)	16.12 (15.96)
C4 complex	366.15	red (77)	311	21.54	39.43 (39.36)	4.58 (4.96)	22.56 (22.95)	17.78 (17.48)	15.76 (15.25)

Table.No.01. Elemental analysis, molar conductance, melting point of metal complexes with commercial drug.

**XRD data:**

The X-ray diffractogram of some transition metal complexes with drug are crystalline as well as amorphous in nature. To determine the h, k, l values of reflection by using known methods. The X-ray diffraction method was used for getting evidence about the position of atom, electron density and structure of the metal complexes. The diffractogram obtained for the Cu (II), Ni (II), Co (II) and Fe (II) complexes were given in figures-a, b, c and d. The XRD patterns indicated crystalline nature of Ni (II), Co (II) complex while Fe (II) and Cu (II) complexes are amorphous in nature. The values of each peak have been determined by cell parameters and corresponding h, k, l values. The lattice constants a, b and c for each unit cell have been found out and given tables-2. The XRD patterns the major peak which showed relative intensity greater than 10% indexed by computer program Experimental density values of the complexes were determined by using specific gravity method 13.

Compounds	Lattice constant			$\alpha^{\circ}$	$\beta^{\circ}$	$\gamma^{\circ}$	$2\theta$	D value	Crystal system
	a(A)	b(A)	c(A)						
C1 complex	8.72	10.23	8.73	97	109	97	8.64	10.25	Monoclinic
C2 complex	11.23	14.15	8.45	91	104	91	7.23	6.73	monoclinic
C3 complex	7.34	12.74	8.73	107	116	94	8.43	7.34	Triclinic
C4 complex	8.24	10.27	9.42	90	101	90	14.23	8.14	monoclinic

Table No. 2 XRD data of coordination complexes

Fig. a) X-ray diffractogram of C1 complex

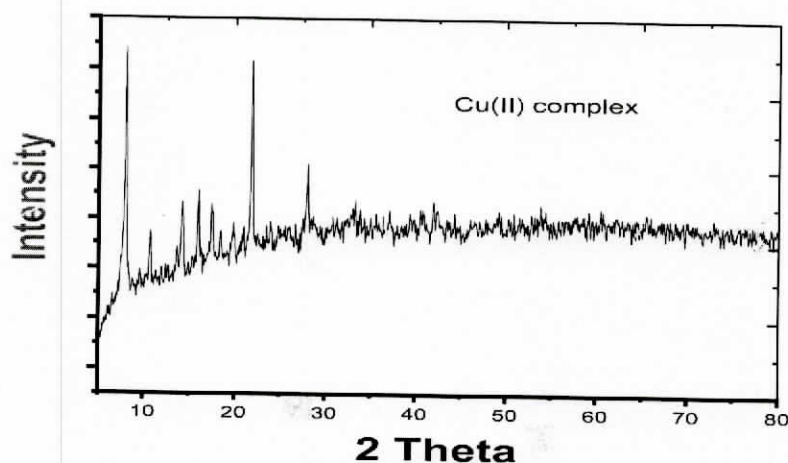


Fig. b) X-ray diffractogram of C2 complex

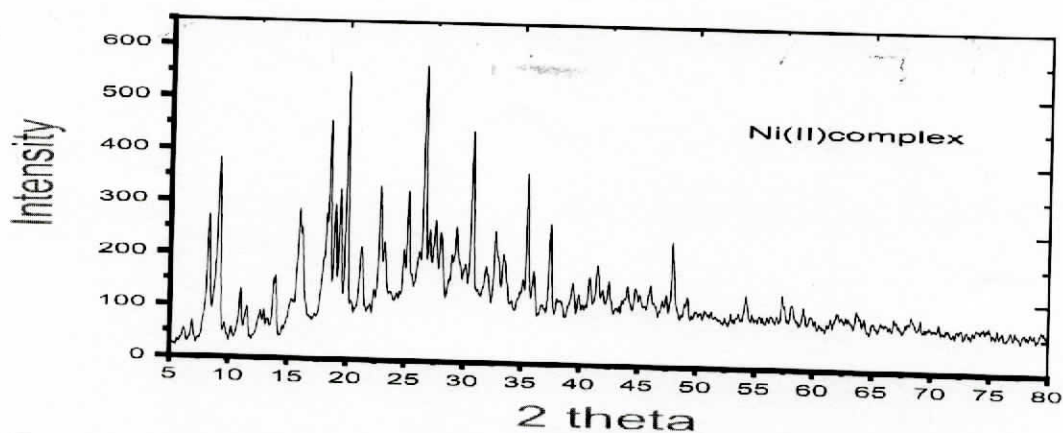


Fig. c) X-ray diffractogram of C3 complex

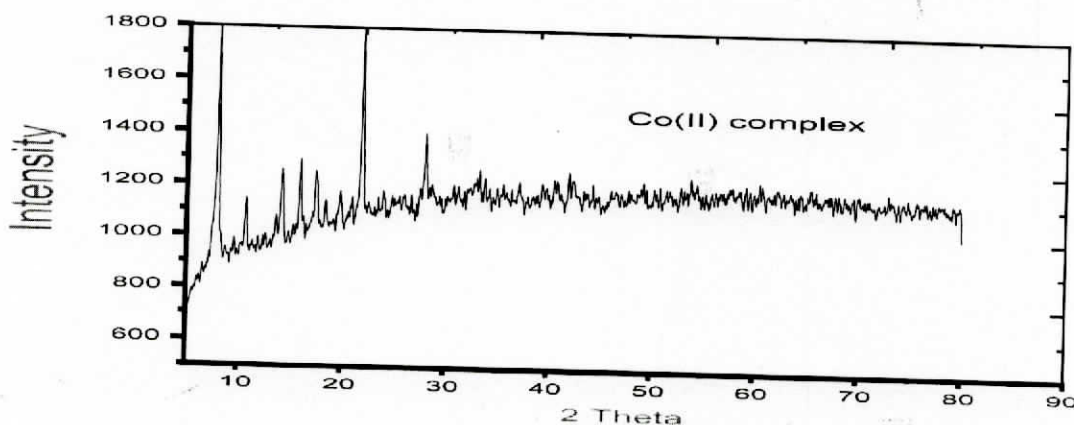
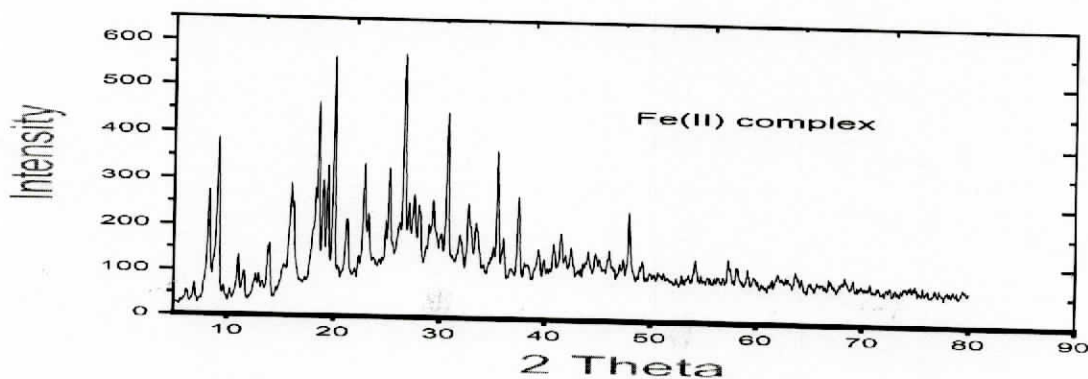


Fig. d) X-ray diffractogram of C4 complex

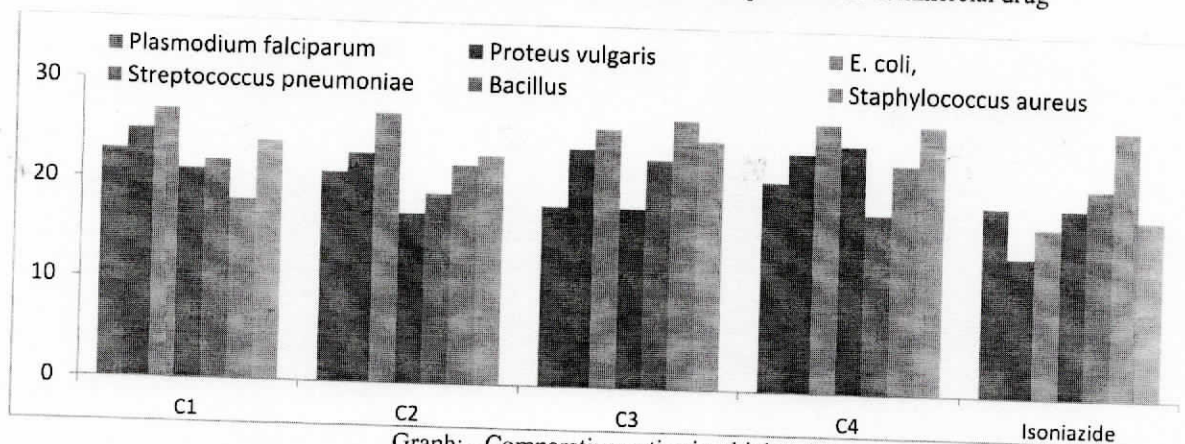


**Anti-Microbial Activity:** The ligand and corresponding metal (II) complexes were screened in vitro for their antibacterial activity against Gram (+ve) and Gram (-ve) bacteria. Recommended concentration for the test sample 1000 mg/ml (1000ppm) in DMSO was paper strip of solution introduced on nutrient plate with reference drug. The plates are incubated immediately at 37°C for 24 hours. Activity was determined by measuring the diameter of zones inhibition (mm). Metal drug Complex was shown better zone of inhibition on Petri-plates rather than commercial drugs against bacteria.

Sr.No	Name of micro-organism which is used for test	Metal complexes zone of inhibition in (mm) (Concentration of each complexes	Isoniazid drug zone of

		1000 mg/ml in DMSO)				inhibition in (mm)
		C <sub>1</sub>	C <sub>2</sub>	C <sub>3</sub>	C <sub>4</sub>	
01	<i>Plasmodium falciparum</i>	23	21	18	21	19
02	<i>Proteus vulgaris</i>	25	23	24	24	14
03	<i>E. coli,</i>	27	27	26	27	17
04	<i>Streptococcus pneumonia</i>	21	17	18	25	19
05	<i>Bacillus</i>	22	19	23	18	21
06	<i>Staphylococcus aureus</i>	18	22	27	23	27
07	<i>Mycobacterium tuberculosis</i>	24	23	25	27	18

Table. No. 03 Anti-microbial activity of metal complexes with commercial drug



**Conclusion:**

The Drug metal complexes of Cu (II) , Ni(II), Co(II), Fe(II), was synthesized and characterized on the basis of analytical and spectral data. The structural elucidation studies by XRD suggested the octahedral geometry and close packing position of atom in crystalline. Antimicrobial activity was studied by minimum inhibitory concentration (MIC) method by disc diffusion technique. Antimicrobial studies suggest that complexes give us good platform in developing a new class of antibiotics. Proposed structure of coordination complexes is as in fig.

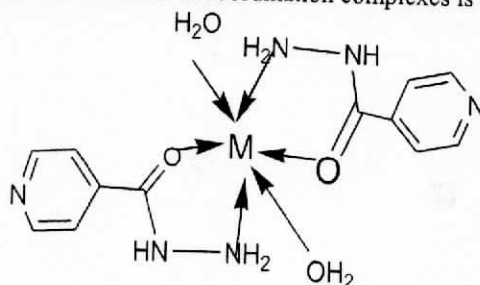


Fig. Proposed structure of metal isoniazid complex

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