

# Synthesis and Characterization of Mixed Complexes of Co (II) and Fe (II)

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**Abstract:** Mixed ligand complexes of Co (II) and Fe (II) with salicylic acid and nicotinamide were synthesized. The complexes were characterized by some standard methods. They were found to be soluble in all solvents. The following melting points were recorded; 202 °C, 320 °C, and 292 °C, for Fe (II) (sal)<sub>2</sub>, Fe (II) (Nic)<sub>2</sub> and Fe (Sal/Nic) respectively. The rest are 162 °C, 316 °C and 196 °C for Co (Sal)<sub>2</sub>, Co (Nic)<sub>2</sub> and Co (Sal/Nic) mixed ligand complex respectively. In the research work, it was found out that most of the complexes have high molar conductivity value, while some others have low molar conductivity value. The complexes with lower molar conductivity value are less soluble in solvents than the complexes with higher value. The FTIR spectra observed in all the complexes showed that the coordination of [Co (Nic)<sub>2</sub>Cl<sub>2</sub>] and [Fe (Nic)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] is between N of the amine group (NH<sub>2</sub>) and the metal of the complex. In [Co (Sal)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] and [Fe (Sal)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] complex; coordination occurred between O of C=O group and the metal M of the complex. And in [Fe (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] and [Co (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] mixed ligand complex, the coordination of the complex is between O of the salicylic acid, the metal M and N of the pyridine in nicotinamide. But in [Co (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] coordination occurred between the N of the amine group and the O from C=O of the carboxylic group in salicylic acid. .

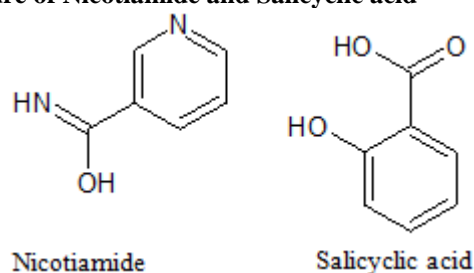
**Keywords:** Mixed Ligands; complexes, Co (II); Fe (II); nicotinamide; salicylic acid, [Co (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>]

## 1. Introduction

The number of the chemical ligand species which can be coordinated with metal ions are very large. They can coordinate with ligands of different functional groups such as C=C, C=O, C-O, C-C, C-H, O-H etc. Metal ion and ligands are therefore, in a dynamic equilibrium with one other in more or less aqueous environment in a transition metal complex which is formed in a biological system. The ligands have different vibrational spectral bands [1] when taken to FT-IR analysis. Some decades ago, many scientists and research groups operated through specialization in the direction of drug discovery by studying the simplest species that use metal ions and investigating on them as whole compound. Example, they suggested the inclusion of metal ion to antibiotics to hasten their spread throughout the body [2]. The development of resistance by some disease causing germs as well as the existence of some unwanted side effects of certain antibiotics [3, 4] had led to the search of new antimicrobial agents with the aim to discover new chemical structures which overcame the above disadvantages [5]. Zn (II) and Cu (II) are abundant transition metal ions in humans [6]. Usually, they are found at active sites as components of the structure of a number of enzymes. Cobalt is present in Vitamin B<sub>12</sub>, though Fe (II) has least anti-microbial effect. It is used in the production of blood tonics and blood boosting drugs like Raferon-12, chemiron, Ferrous, while chlorophyll in green plants contain Mg (II) complex. It is a coenzyme that plays an important role in some biochemical processes where many transition metals are effective therapeutic agents particularly when coordinated with ligands to form complexes. The list of metals containing compounds used in chemotherapy treatment of diseases are: platinum (anticancer), silver (antimicrobial), Gold (antiarthritic), Bismuth [7] (antiulcer). Antimony (antiprotozoal), Vanadium (antidiabetic) [7] and Iron (antimalaria) [8]. The study of coordination metals is called coordination chemistry [9].

complex ions, compounds or molecules are formed [10] from the principle that a central metal ion is surrounded by other molecules or ions [11]. As a result, metal ion in a solvent does not exist in isolation, but it exist in conjunction with ligands such as liquid molecules called solvent. In these complexes, the central atom or ion many a-times contain transition metal and cluster of ions or neutral molecules surrounding it [12, 13]. The central atoms or ions are bonded to the ligands which are ions or neutral molecule. The central metal atom acts as an electron pair acceptor (Lewis acid) [14] while ligands act as electron pair donors (Lewis base). The word 'ligand' originated from Latin word "ligare" which means to bind. This word ligare was first used by Alfred Stock in 1916 in reference to silicon chemistry. The word ligand could be cation, anion or neutral molecules. Ligand must contain one donor atom, with electron pair [14] used for the formation of covalent bonds with central atom. Classes of ligands include: monodentate, bidentate or tridentate. A monodentate ligand contains only one donor atom while bidentate and tridentate contains two donor atoms and three donor atoms respectively [15]. In the synthesis, characterization and antimicrobial screening of Fe (II) and Co (II) complexes by Abdulahi Mustapha et al, transition metal form bidentate and tridentate complexes with ligands [16].

## Structure of Nicotinamide and Salicylic acid



## 2. Material and Methods

In this research work Co (II) and Fe (II) complexes with salicylic acid and Nicotinamide were synthesized in addition to their mixed ligands (Note: the structure of the ligands which is salicylic acid and nicotinamide are already known). The physical properties of the complexes such as melting point, solubility and electrical conductivity were determined. The complexes are further characterized by the fourier transformed infra red spectroscopy (FT-IR) analysis and X-RD diffraction analysis.

### Preparation of Cobalt (II) Salicylic Acid

The complexes of Co (Nic)<sub>2</sub>, Fe (Nic)<sub>2</sub>, Co (Sal)<sub>2</sub>, Fe (Sal)<sub>2</sub> and their mixed ligands were prepared by the method similar to that of (Ram et al; and Lawal, et al) [17-18]. 2 g (0.0084 mol) of CoCl<sub>2</sub> was weighed using an electronic weighing scale and dissolved in a conical flask with 20 mL of distilled water, 2.32 g (0.018 mol) of salicylic acid was also weighed out and dissolved in 20 mL of ethanol. The dissolved samples were mixed together and stirred thoroughly. It was then refluxed for 3hrs at temperature of about 70°C.

### Preparation of Co (II) Nicotinamide Complex

2 g of CoCl<sub>2</sub> was weighed out and dissolved as explained above and 2.05g. (0.0168 mol) of Nicotinamide was also weighed and dissolved thoroughly then mixed together and refluxed for 3hrs at a temperature of about 70°C.

### Preparation of Fe (II) Salicylic Acid Complex

2 g. (0.01577 mol) of FeCl<sub>2</sub> was weighed, and dissolved with 4.36 g. (0.03154 mol) of salicylic acid which was thoroughly mixed together as explained then refluxed for 3hrs at the same temperature range.

### Preparation of Fe (II) Nicotinamide Complex

2 g (0.01577 mol) of FeCl<sub>2</sub> was weighed, dissolved and mixed with 3.85 g (0.03154 mol) of Nicotinamide which was also dissolved as explained above and then mixed together, and refluxed for 3 hrs [19] also at the same temperature range.

### Preparation of CO (II) Salicylic Acid / Nicotinamide Mixed Ligand Complex

The mixed ligand complexes were prepared according to (Gajendra, and Dharmendra, et al) 2 g (0.0084 mol) of (Co (II)) was mixed with 1.16 g (0.0084 mol) of salicylic acid and 1.03 g (about 0.0084 mol) of Nicotinamide. The salt was dissolved with 20 mL of distilled water and stirred thoroughly. The ligands each was dissolved in 10 mL of ethanol and the mixture stirred thoroughly. The resulting mixture was added to CoCl<sub>2</sub> solution and stirred thoroughly. It was then refluxed for 5-6 hrs. The product was poured into the beaker and allowed to cool and dry for some days leading to the formation of crystalline samples. [20]

### Preparation of Fe (II) Salicylic Acid/Nicotinamide Mixed Ligands Complex

The mixed ligand complexes were prepared according to (Gajendra, et al; Imram, M. et al; Mishra, et al) 2 g (0.01577 mol) of Fe (II) was mixed with 2.18 g (0.81577 mol) of salicylic acid and 1.93 g (0.01577 mol) of Nicotinamide. The separate mixtures of the ligands were dissolved in 10

mL of ethanol, while the salt Fe (II) was dissolved in 20 mL of distilled water, the mixture was stirred thoroughly. Then the resulting mixture of salt and ligand was refluxed for 6 hrs allowed to cool leading to the formation of crystalline samples. [20-23]

## 3. Results and Discussion

**Physical characteristics of the metal complexes;** all the complexes can dissolve slightly in distilled water, ethanol and methanol. All the complexes are crystalline in nature. All the complexes melt above 190°C except Co (Sal)<sub>2</sub> which melt at 162°C. [Fe (Sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] and [Co (Sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] have temperature of 202°C and 162°C respectively, which is higher than that of neutral salicylic acid. The increase in temperature is attributed to the increase in mass of the formed complexes. [Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] and [Co (Nic)<sub>2</sub>Cl<sub>2</sub>] have temperature of 320°C and 316°C respectively, which is also higher than that of the neutral nicotinamide. The increase in temperature is attributed to increase in the mass of the complexes formed. The complexes with temperature range of 200°C and above, have high temperature and are more stable than complexes with less than 200°C temperature range. The melting point of [Fe (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] and [Co (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] is higher than that of neutral salicylic acid with 148.8°C and nicotinamide with 131°C. [Fe (Sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] have high molar conductivity of 2872 (□s/cm). [Co (Sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] have 214 (□s/cm) molar conductivity. [Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] have 14 (□s/cm) molar conductivity. [Co (Nic)<sub>2</sub>Cl<sub>2</sub>] have molar conductivity of 12 (□s/cm). [Fe (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] have molar conductivity of 344 (□s/cm), while [Co (Sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] have molar conductivity of 244 (□s/cm). Complexes with high molar conductivity value are strong electrolytes, while complexes with lower conductivity are weak electrolyte. The complexes with lower molar conductivity are less soluble than the complexes with higher molar conductivity.

### FT-IR Analysis

#### [Co (Nic)<sub>2</sub> (Cl<sub>2</sub>) and (Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>) complexes

The infra red spectra of nicotinamide was compared with that of its prepared complexes. The absorption band at 3711.17cm<sup>-1</sup> due to NH<sub>2</sub> vibration in the spectrum of neutral nicotinamide was shifted in the spectra of the prepared complexes. This shows that the coordination of [Co (Nic)<sub>2</sub>Cl<sub>2</sub>] and [Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] is between N of the amine group (NH<sub>2</sub>) and the metal of the complex. The spectra band of the C=O present in nicotinamide shifted down from 1681.98 to 1666.56 cm<sup>-1</sup>, showing that coordination of the complex is between the metal M of the complex and the O of the C=O and in [Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] the C=O shifted from 1681.98 cm<sup>-1</sup> to 1612.54 cm<sup>-1</sup> showing that coordination of the complex is between the central metal and the C=O. The spectra band at 1141.90cm<sup>-1</sup> due to N-H deformation vibration position in the metal complexes also shows that no coordination took place through it. The C=N vibrations which occurs at 1404.22 cm<sup>-1</sup> in the neutral nicotinamide was not shifted in the metal complex of [Co (Nic)<sub>2</sub>Cl<sub>2</sub>], but was slightly shifted in [Fe (Nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] complex showing that no effective coordination took place through the N of the pyridine ring. The spectrum band of 609.53cm<sup>-1</sup> and 524.66 cm<sup>-1</sup> which is not sighted in the neutral Nicotinamide is temporarily assigned to ν (M-O) stretching. Also the

spectrum band at  $416.60\text{ cm}^{-1}$  and  $462.93\text{ cm}^{-1}$  in the infra red of the metal complex which could not be sighted in that of neutral nicotinamide is temporarily assigned to  $\nu$  (M-N) stretching band. The occurrence of the spectra at  $3410.26\text{ cm}^{-1}$  in virtually all the metal complexes except [Co (Nic)

$2\text{Cl}_2$ ] suggests the involvement of OH from  $\text{H}_2\text{O}$  in the coordination of the metal complexes, though the shift from the band is slight from  $3417.98$  to  $3410.26\text{ cm}^{-1}$ .

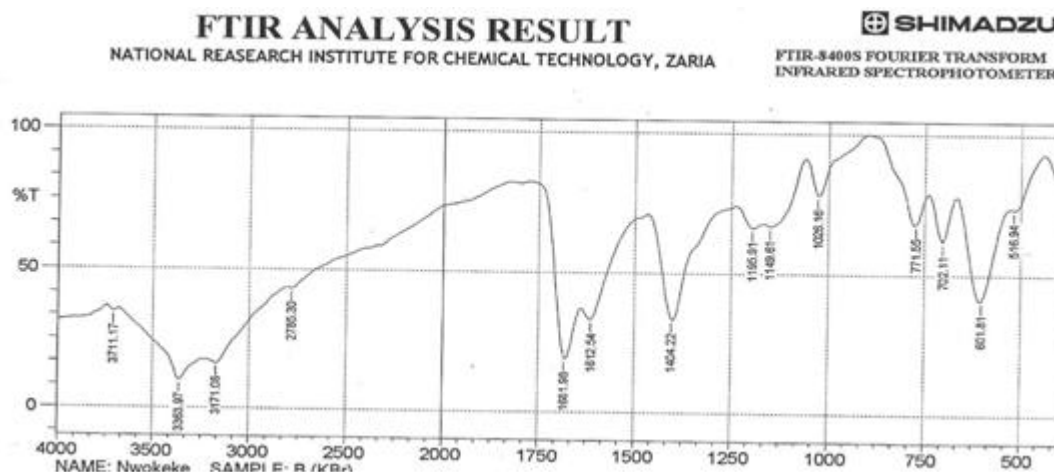
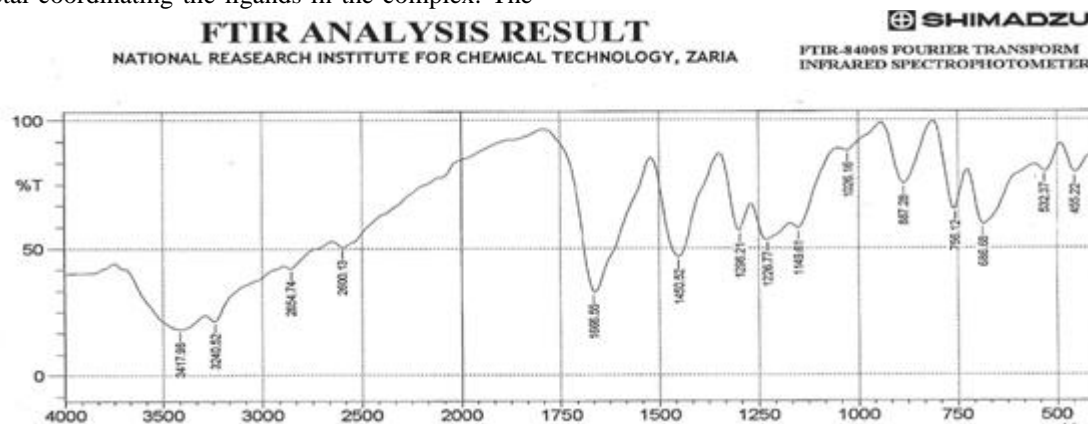


Figure 1: FTIR Result of nicotinamide ligand

#### [Co (Sal) $_2$ (H $_2$ O) $_2$ ] and [Fe (Sal) $_2$ (H $_2$ O) $_2$ ] complexes

The spectra band of the C=O shifted from  $1666.55\text{ cm}^{-1}$  of the neutral salicylic acid to  $1543.10\text{ cm}^{-1}$  and  $1465.95\text{ cm}^{-1}$  respectively in the metal complexes which supports that coordination occurred between O of the carboxylic group and the metal coordinating the ligands in the complex. The

spectra band of  $\nu$  (M-O) in the complex of [Co (Sal)  $_2$  (H $_2$ O)  $_2$ ] and [Fe (Sal)  $_2$  (H $_2$ O)  $_2$ ] showed at  $601.81$  and  $617.24\text{ cm}^{-1}$  respectively, supporting that coordination occurred between O of C=O group and the metal M of the complex.



[Fe (Sal/Nic) (H $_2$ O)  $_2$ ] and [Co (Sal/Nic) (H $_2$ O)  $_2$ ] mixed ligand complex;

In [Fe (Sal/Nic) (H $_2$ O)  $_2$ ] mixed ligand complex,  $\text{NH}_2$  spectra band of neutral nicotinamide remained unchanged at  $3711.17\text{ cm}^{-1}$  showing that it is not taking part in the coordination. The spectra band of C=O of the carboxylic group present in neutral salicylic acid dropped from  $1666.55\text{ cm}^{-1}$  to  $1635.69\text{ cm}^{-1}$ , while C=N dropped from  $1404.22\text{ cm}^{-1}$  to  $1234.48\text{ cm}^{-1}$  in the nicotinamide, showing that the coordination of the complex is between O of the salicylic acid and the metal M, and N of the pyridine in

nicotinamide. But in [Co (Sal/Nic) (H $_2$ O)  $_2$ ] complex, the  $\text{NH}_2$  spectra band dropped from  $3711.17\text{ cm}^{-1}$  in the neutral nicotinamide to  $2592.41\text{ cm}^{-1}$ . This supports that coordination occurred between the N of the amine group and the O from C=O of the carboxylic group in salicylic acid which dropped slightly from  $1681.81\text{ cm}^{-1}$  to  $1651.12\text{ cm}^{-1}$ . Also from the FT-IR result, the mixed ligand complexes coordinated to the central metal ion with two water molecules to form an octahedral structure.

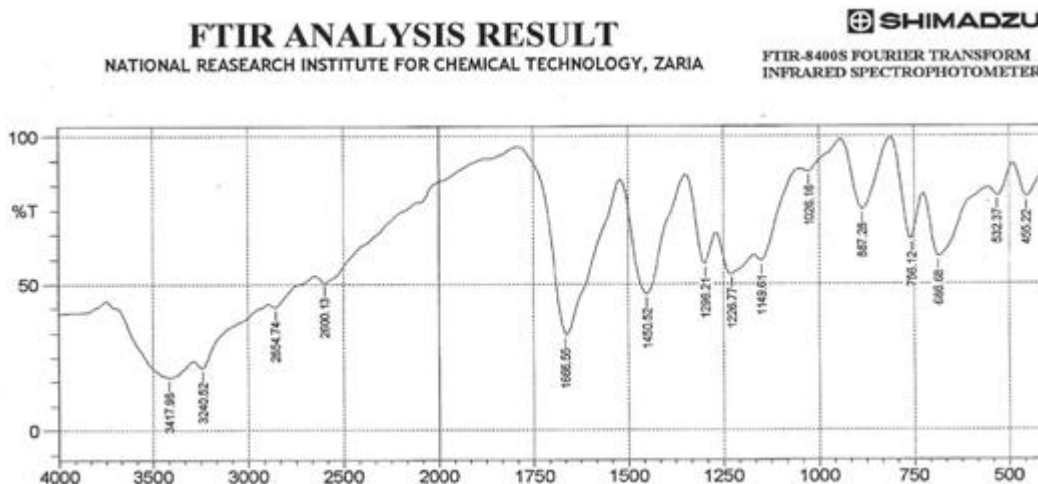


Figure 2: FTIR result of Salicylic acid ligand

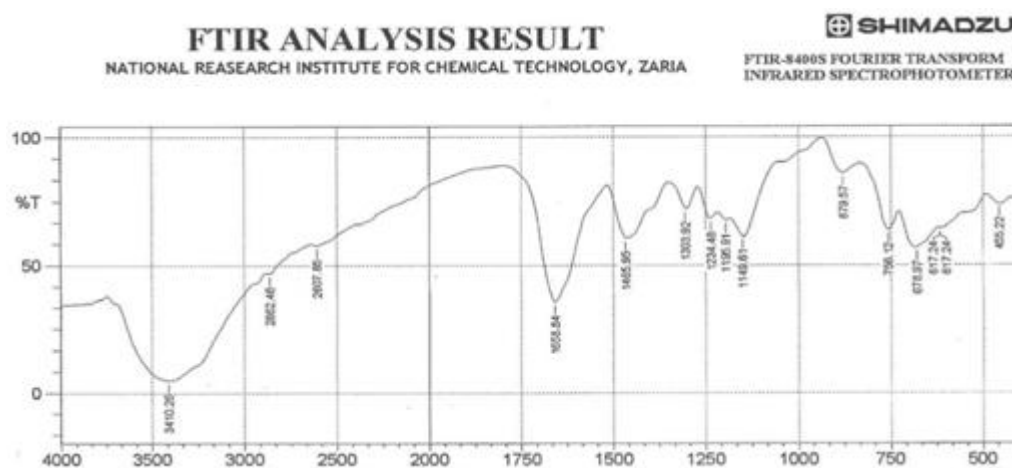


Figure 3: FTIR result of Fe (II) Salicylic acid

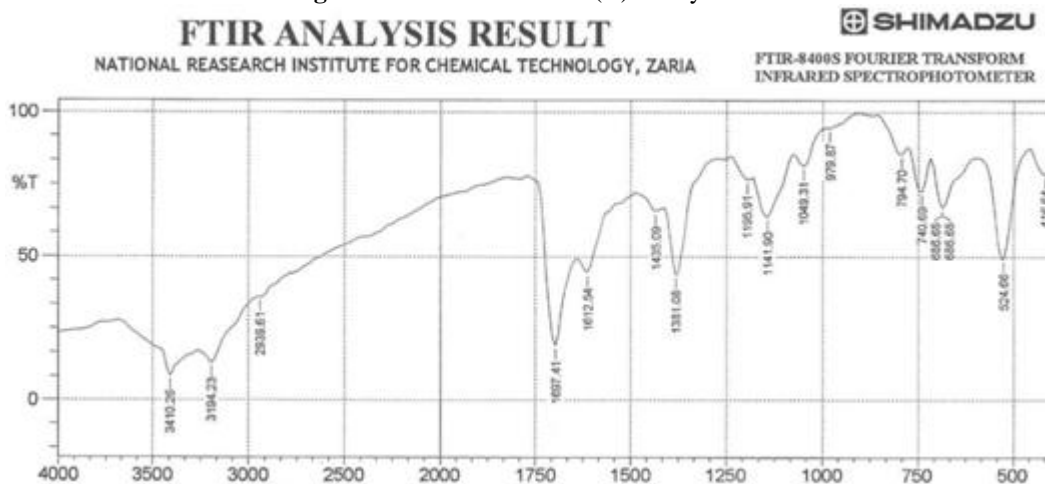


Figure 4: FTIR result of Fe (II) Nicotinamide Complex

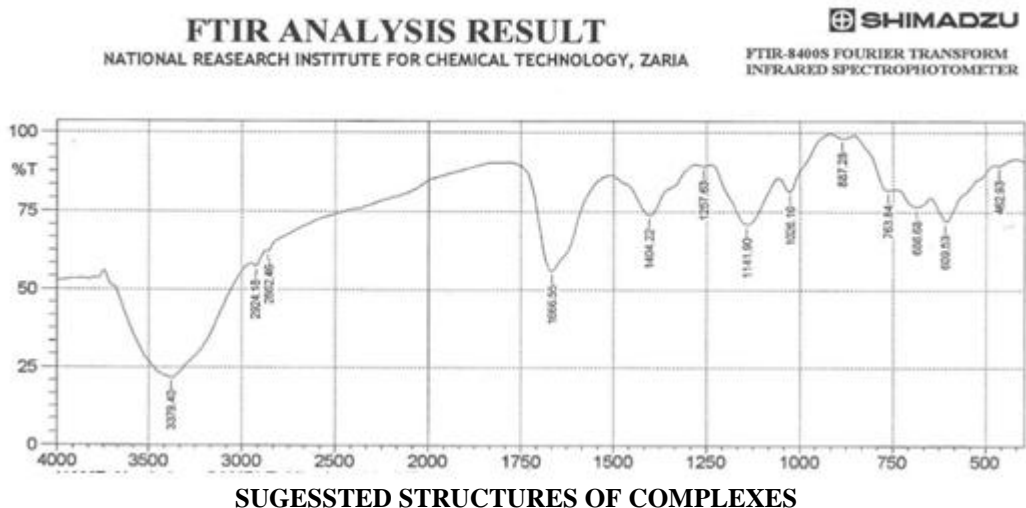


Figure 5: Co (II) nicotinamide Complex

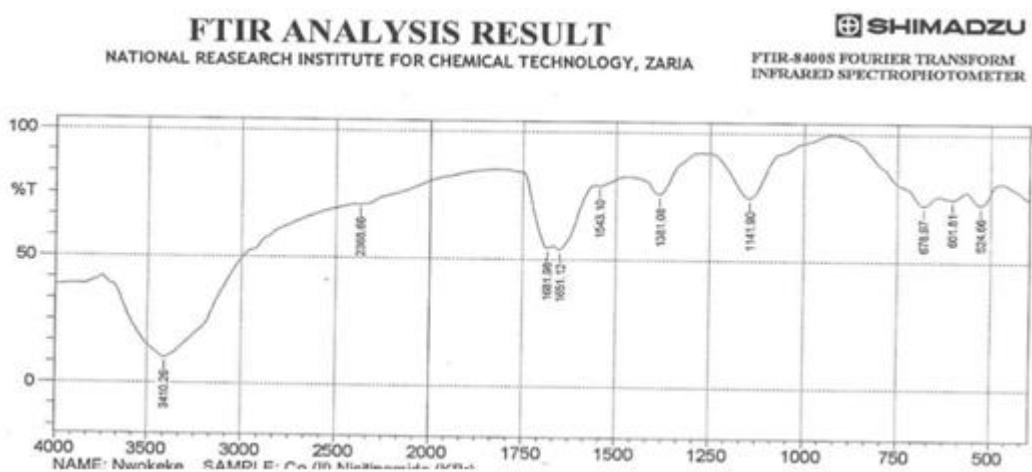


Figure 6: Co (II) salicylic acid Complex

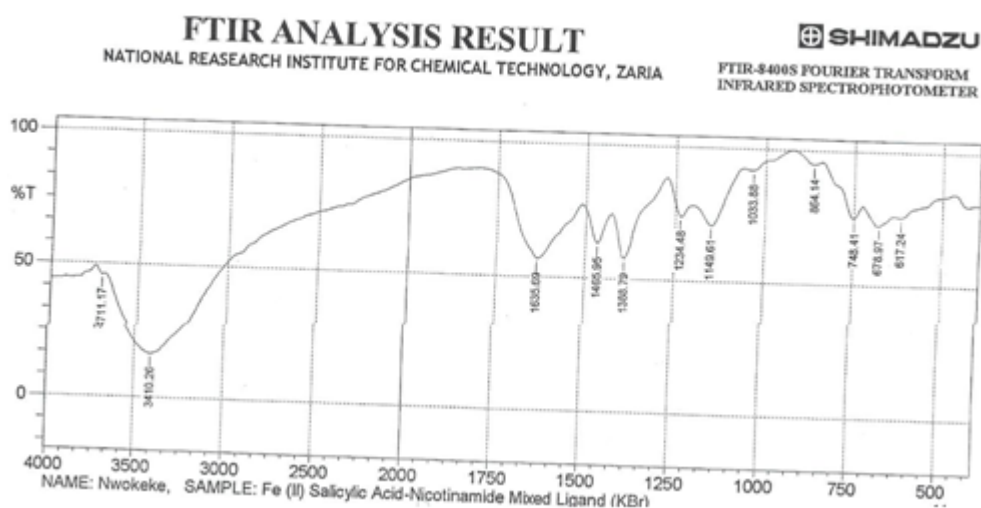


Figure 7: Fe (II) Salicylic Acid/Nicotinamide mixed ligand complex

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SHIMADZU  
FTIR-8400S FOURIER TRANSFORM  
INFRARED SPECTROPHOTOMETER

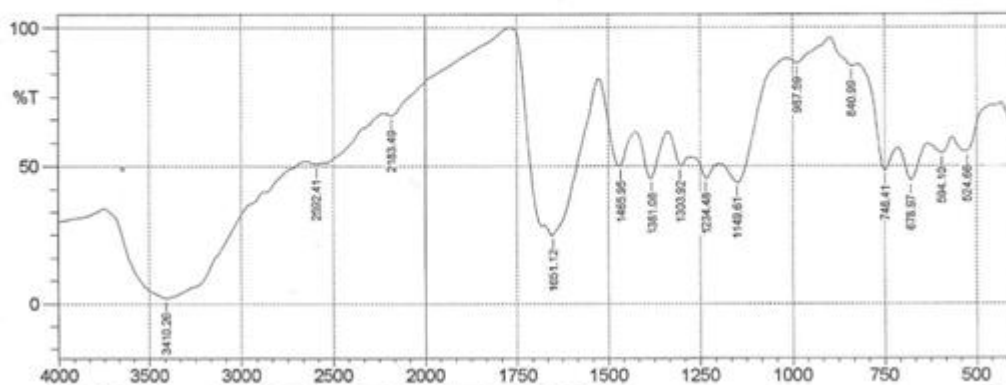


Figure 8: Co (II) Salicylic Acid/Nicotinamide Mixed ligand complex

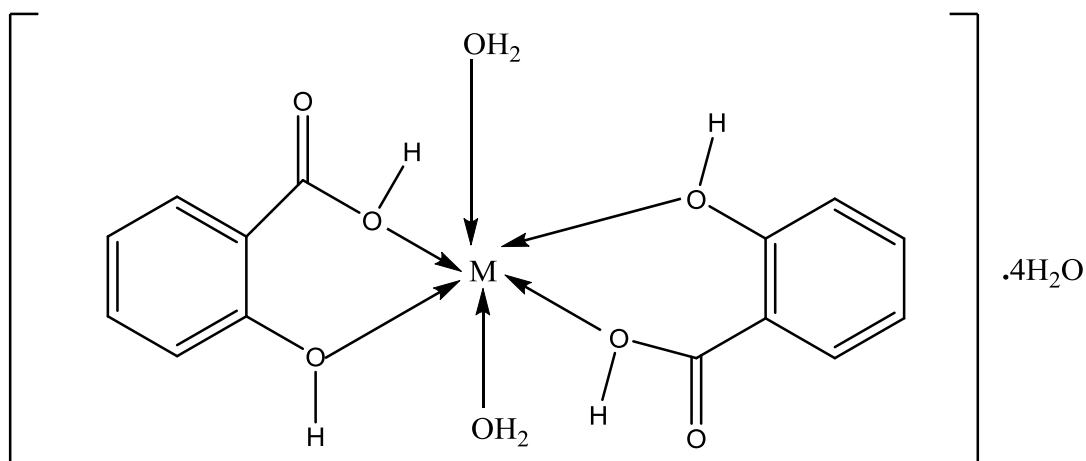


Figure 9:  $[Fe(sal)_2(H_2O)_2]$  complex

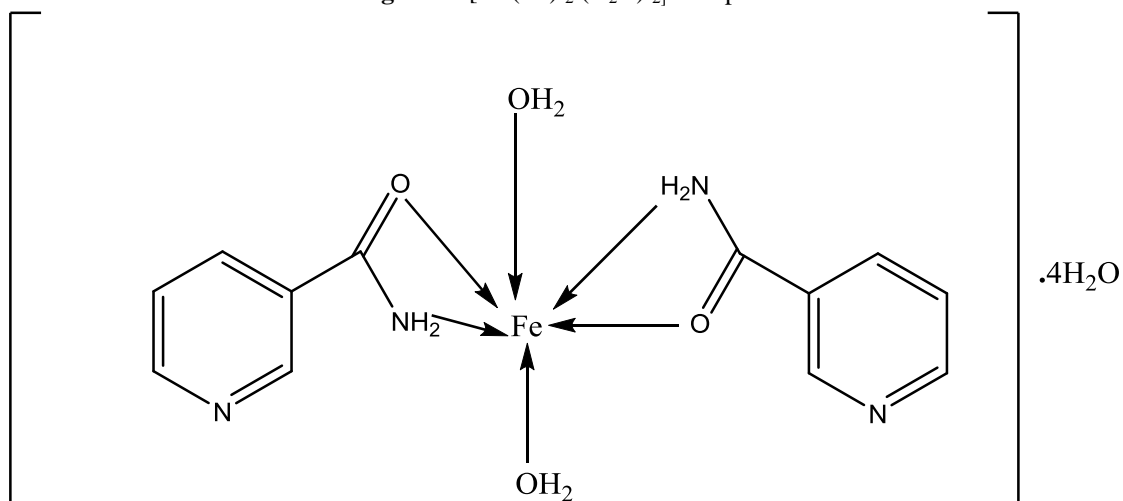


Figure 10:  $[Fe(Nic)_2(H_2O)_2]$  complex

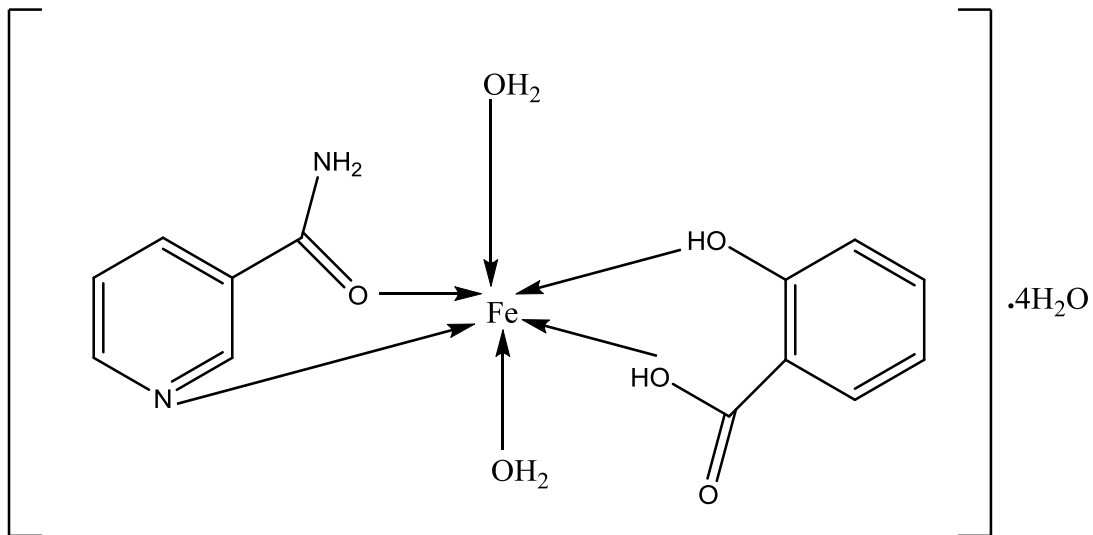


Figure 11:  $[Fe (sal/Nic)_2 (H_2O)_2]$  mixed ligand complex

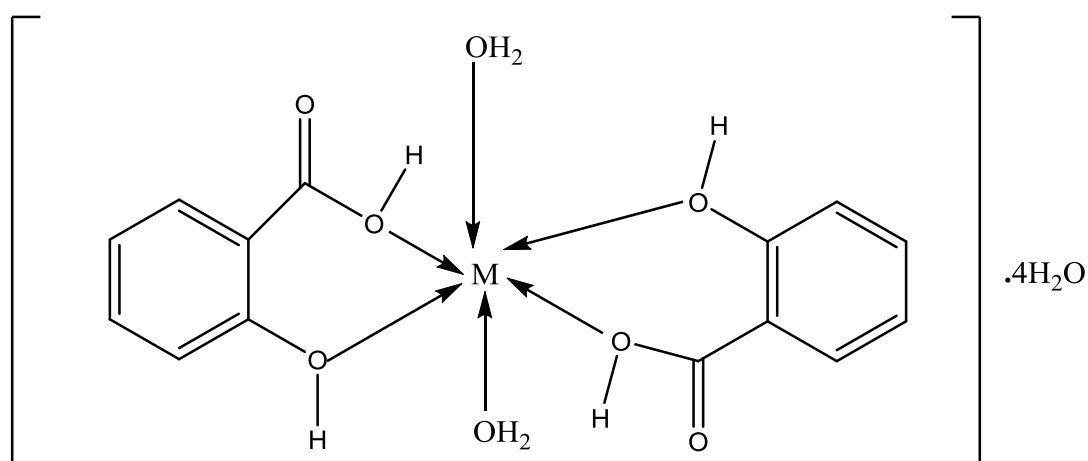


Figure 12:  $[Co (sal)_2 (H_2O)_2]$  complex

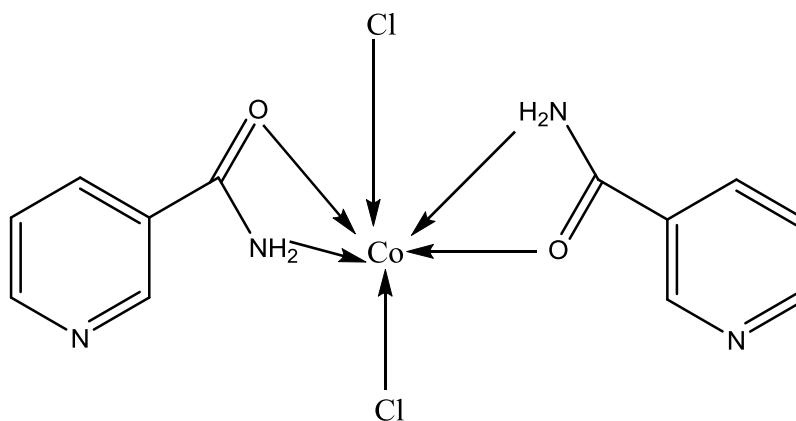


Figure 13:  $[Co (Nic)_2 Cl_2]$  complex

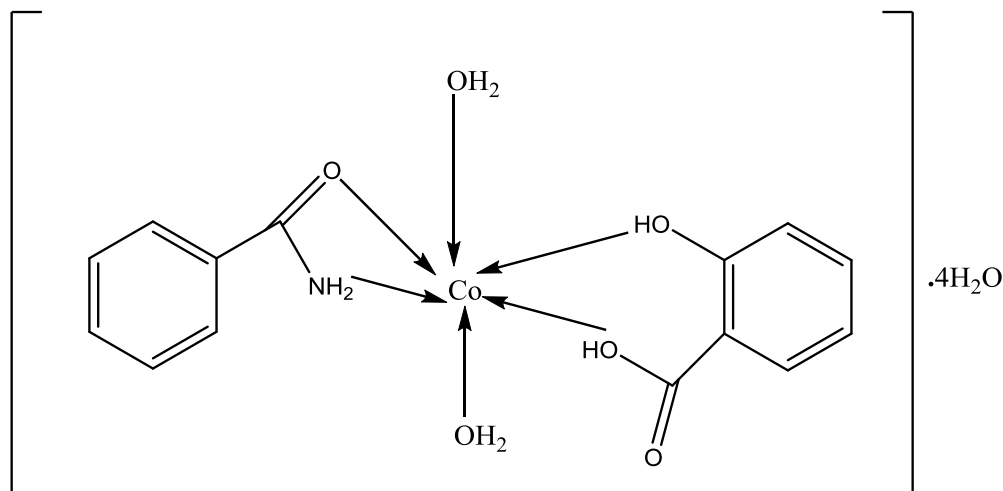


Figure 14: [Co (sal/Nic) (H<sub>2</sub>O)<sub>2</sub>] mixed ligand complex

**X-RD GRAPH / ANALYSIS**

Shown below is the X-RD graph of the complexes prepared and their analysis.

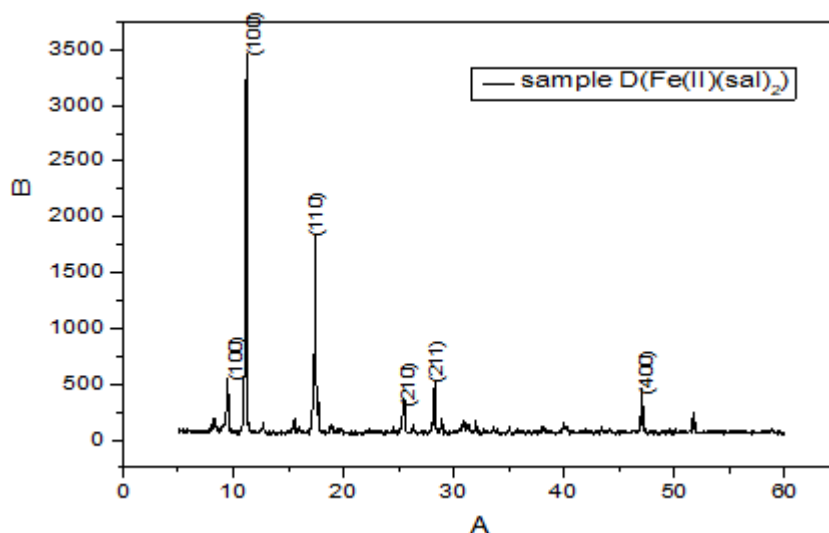


Figure 15: X-RD graph of (Fe (II) (Sal)<sub>2</sub>)

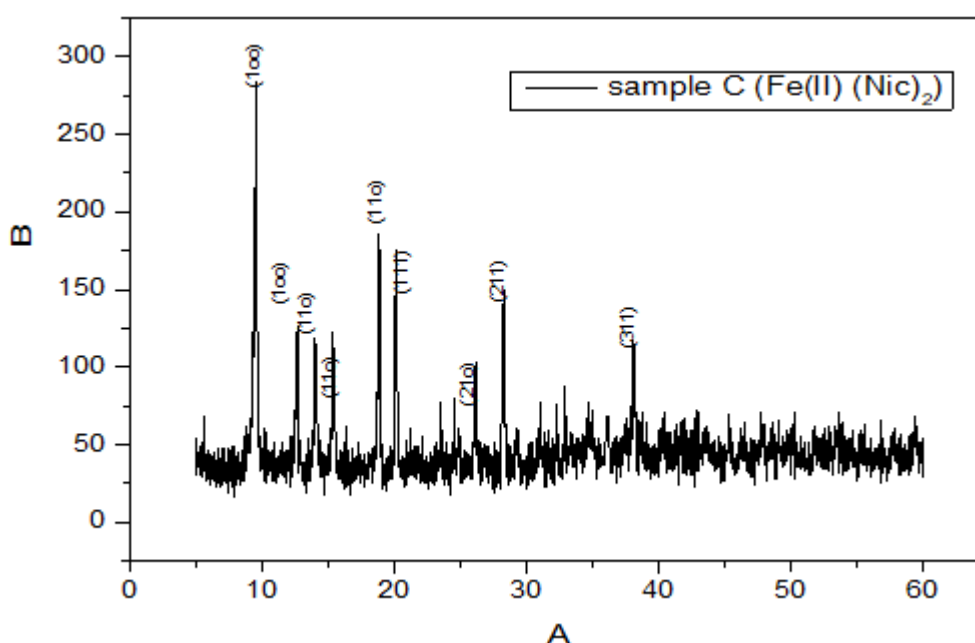


Figure 16: X-RD graph of (Fe (II) (Nic)<sub>2</sub>)



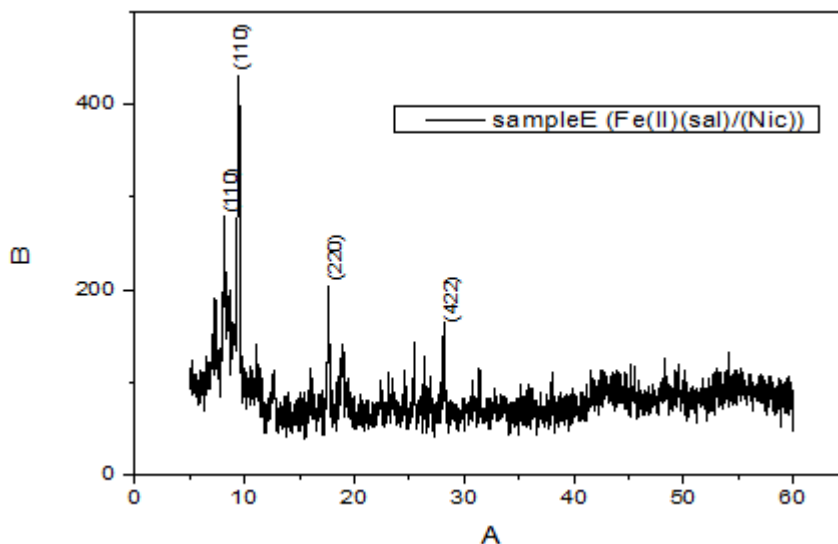


Figure 17: X-RD graph of (Fe (II) (Sal) / (Nic))

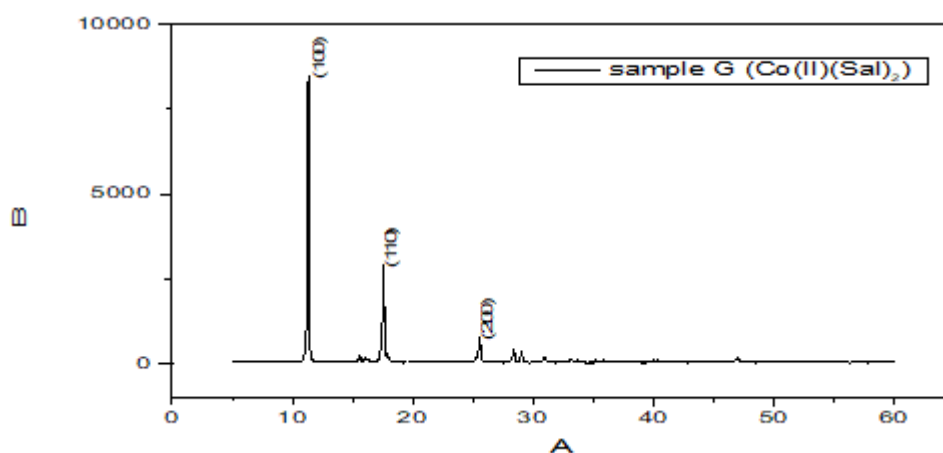


Figure 18: X-RD graph of (Co (II) (Sal) <sub>2</sub>)

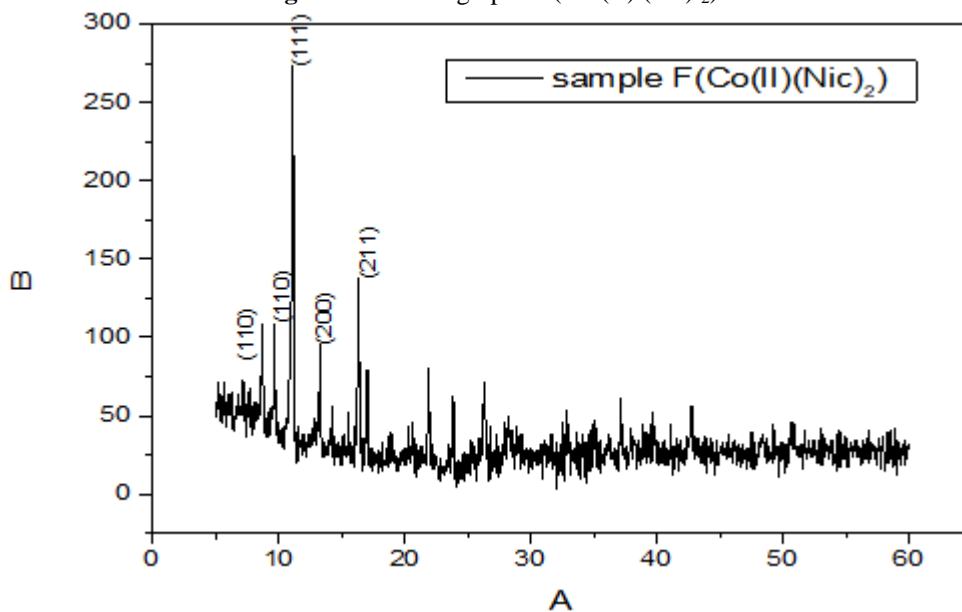


Figure 19: X-RD graph of (Co (II) (Nic) <sub>2</sub>)

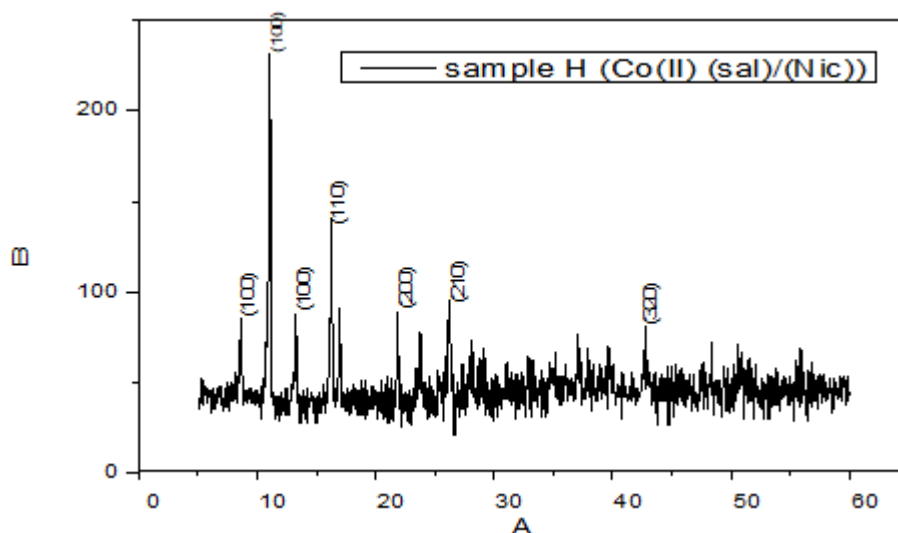


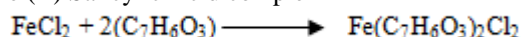
Figure 20: X-RD graph of (Co (II) (Sal) / (Nic))

Table 1: X-RD graph analysis table

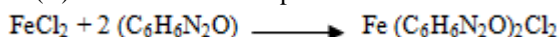
Sample D in $2\theta$	Sample C in $2\theta$	Sample E in $2\theta$	Sample G in $2\theta$	Sample F in $\theta$	Sample H in $\theta$
Fe (II)-9.5 Fe (II)-11.0 (Sal) <sub>2</sub> -18.0 (Sal) <sub>2</sub> -26.0	Fe (II) 10.0 (Nic) <sub>2</sub> -15.5 (Nic) <sub>2</sub> -19.5 (Nic) <sub>2</sub> -27.0 (Nic) <sub>2</sub> -28.0	Fe (II)-9.0 Fe (II)-10.0 (Sal)-18.0 / (Nic)-29.0	Co (II)-11.5 (Sal) <sub>2</sub> -17.5 (Sal) <sub>2</sub> -25.5	Co (II)-8.5 Co (II)-9.5 Co (II)-11.0 (Nic) <sub>2</sub> -13.0 (Nic) <sub>2</sub> -16.5	Co (II)-8.5 Co (II)-11.0 Co (II)-13.5 (Sal)-17.0 / (Nic)-21.0

#### Equation of the reactions for the complexes prepared

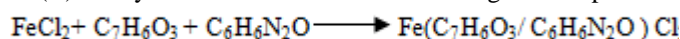
Fe (II) Salicylic Acid complex



Fe (II) Nicotinamide Complex



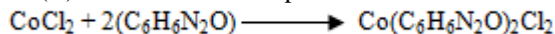
Fe (II) Salicylic Acid/Nicotinamide mixed ligand complex



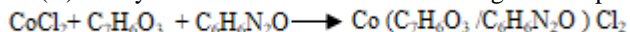
Co (II) Salicylic Acid Complex



Co (II) Nicotinamide Complex



Co (II) Salicylic Acid/Nicotinamide Mixed ligand complex



#### 4. Conclusion

Complexes of salicylic acid and nicotinamide with Co (II) and Fe (II) and their mixed ligands were synthesized. In the complexes two molecules of water and two molecules of the bidentate ligand coordinated to the central metal ion. Example, [Fe (nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>], [Fe (sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>], [Co (sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>], [Fe (sal) / (nic) (H<sub>2</sub>O)<sub>2</sub>] and [Co (sal/nic) (H<sub>2</sub>O)<sub>2</sub>]. The synthesized complexes were characterized by determining their melting point, solubility, molar conductivity, FT-IR spectroscopy and X-RD diffraction. In the research work, it was found out that most of the complexes have high molar conductivity value, while some

others have low molar conductivity value. The complexes with lower molar conductivity value are less soluble in solvents than the complexes with higher value. The results of the physical and spectroscopic data of this research work both confirm that nicotinamide and salicylic acid are chelating ligands. In [Fe (nic)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] and [Co (nic)<sub>2</sub>Cl<sub>2</sub>] complexes, nicotinamide coordinated to the metal ion through the nitrogen of the amine group (NH<sub>2</sub>). While in [Fe (sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] and [Co (sal)<sub>2</sub> (H<sub>2</sub>O)<sub>2</sub>] complexes, salicylic acid coordinated through the oxygen of the C=O group to the metal ion. But in [Fe (sal) / (nic) (H<sub>2</sub>O)<sub>2</sub>] mixed ligand complex, nicotinamide coordinated to the metal ion through the nitrogen of the pyridine ring while salicylic acid coordinated to the metal ion through the oxygen of the C=O group. In [Co (sal/nic) (H<sub>2</sub>O)<sub>2</sub>] mixed ligand complex, nicotinamide coordinated to the metal ion through the nitrogen of the amine group (NH<sub>2</sub>), while salicylic acid coordinated to the metal ion through the oxygen of the C=O group. From the X-RD, the graph of the complexes showed sharp peaks consolidating the crystallinity of the complexes prepared.

#### References

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