Journal of Basics and Applied Sciences Research (JOBASR)
ISSN (print): 3026-9091, ISSN (online): 1597-9962
Volume 3(2) March 2025
DOI: https://dx.doi.org/10.4314/jobasr.v3i2.7

Synthesis, Characterization and Antimicrobial Activities of a Schiff Base Derived from Acetyl Acetone and 2-aminopyridine and its Cobalt (II) and Nickel (II) Complexes

Abdullahi Musa[1*], Sani Ahmad Ibrahim[2] and Abdullahi Aminu Garba[3]
[1, 3]Department of Chemistry, Federal University Dutsin-Ma, Katsina State of Nigeria.
[2]Department of Chemistry, Sa'adatu Rimi Universityof Education Kunbotso, Kano, Nigeria.
Corresponding Author Email: [1]mabdullahi3@fudutsinma.edu.ng ; ORCID: https://orcid.org/0000-0002-9227-8506
[2] deedatdawaki.asi@gmail.com ; ORCID ID: https://orcid.org/0009-0006-9727-6995
[3] aagarba1@fudutsinma.edu.ng ; ORCID ID: https://orcid.org/0009-0006-1965-1729

ABSTRACT

A Schiff base derived from condensation reaction of from acetyl acetone and 2-aminopyridine was synthesized. It was refluxed with Co (II) and Ni (II) chlorides which results in the formation of the corresponding metal (II) complexes in good yield.The Schiff base and metal (II) complexes were characterized by using FTIR, Solubility test, Melting point and decomposition temperature, molar conductance and Gravimetric analysis. The Schiff bases were insoluble in H_2O, CCl_4, but slightly soluble in methanol, ethanol, acetone, nitrobenzene, and petroleum ether and completely soluble indimethylsulphoxide (DMSO) and dimethylformamide (DMF). The Co (II) and Ni (II) metals Complexes were soluble in DMSO and DMF and insoluble in water, CCl4, and nitrobenzene and petroleum ether. The IR spectral data revealed azomethine peak of the Schiff base at 1600 cm^{-1} while for the Co (II) and Ni (II) metals complexes, the peak was found within 1611cm^{-1}– 1605cm^{-1}supporting coordination of Schiff base/ligands. Magnetic moment values of the synthesized Co (II) and Ni (II) complexes obtained were in the range of 4.30 – 3.38 BM which suggested the complexesare paramagnetic. Molar conductance values were found to be within the range of 5.2– 6.05 $\Omega^{-1}cm^2mol^{-1}$revealing that all the complexes are non-electrolytic in nature. The compound obtained were tested for antimicrobial activities against some pathogenic bacteria and fungi: *Staphylococcus aureus, Streptococcus pyogens, and Salmonella typhi, Aspergillusformigatus, Rhizopus spp. and Mucor spp.* respectively, using paper disc diffusion method.The metal Schiff base complexes exhibit higher antimicrobial activity than the free Schiff base.

Keywords:
Acetyl Acetone,
2-Aminopyridine
Schiff's base,
Characterization,
Antimicrobial
activity.

INTRODUCTION

Schiff bases is a chemical compound containing carbon and nitrogen double bond (-HC=N-) (Ghosh*et al.*, 2020, da-silva *et al.*, 2011). It is formed by condensation of an aldehyde or ketone with a primary amine. The oxygen atom in the carbonyl group (>C=O) is replaced by a nitrogen atom to give an imine group (>C=NR where R≠ H). A Schiff base acts as a ligand because it usually contains –N and –O donor atoms (Cotton and Wilkinson, 1972). Schiff bases of both natural and non-natural origin, have exhibited varied range of applications including antibacterial, antitubercular, antifungal, antiparasitic, antiviral, antioxidant, anticancer, analgesic, catalytic and anti-inflammatory properties (Ghosh *et al.*, 2020, da-silva*et al.*, 2011, Noor *et al.*, 2020). Transition metal Schiff base complexes play very vital roles as they are known to possess biological activities such as anticonvulsant, antibacterial, antiviral and antidiabetic (Mimose et al., 1991; Okpechi *et al*. 2024). Schiff base complexes could be used as corrosion inhibitors as well as antifungal and antifouling agents (Bhatia et al., 1993).Schiff bases derived from acetylacetone and p-methoxyaniline showed great activity against some bacteria like *Staphylococusaureus*, *Bacillus sutilis*, *Escheriachia coli* and fungi, *Aspergillusniger* (Ramon et al., 2003)

MATERIALS AND METHODS

Reagents of analytical grade were used without further purification. All glasses as used were well washed with detergent, rinsed with distilled water and dried in an oven at 110°C.The weighing was carried out on

How to cite this article: Abdullahi M., Sani A. I. and Abdullahi A. G. (2025). Synthesis, Characterization and Antimicrobial Activities of a Schiff Base Derived from Acetyl Acetone and 2-Aminopyridine and its Cobalt (II) and Nickel (II) Complexes. *Journal of Basics and Applied Sciences Research*, *3*(2), 62-69. https://dx.doi.org/10.4314/jobasr.v3i2.7

electrical Metler balance Toledo B154, melting point and decomposition temperature were determined using Gallen Kamp melting point apparatus. Molar conductivity was determined using Jen way 4010 model. FT-IR Spectral analysis was recorded on a Fourier Transformed spectrophotometer 8400S model.

Preparation of Schiff base

Equimolarmethanolic solution of 2-aminopyridine (0.941g,0.01 mol) was mixed with Acetyl acetone (1.027cm^3 0.01mol). The resulting mixture was refluxed for 3 hours and the solid product formed after cooling in ice was filtered, purified by recrystallization from methanol and finally dried in desiccator over P_2O_5 for 18 hours (Ashraf *et al.*, 2011).

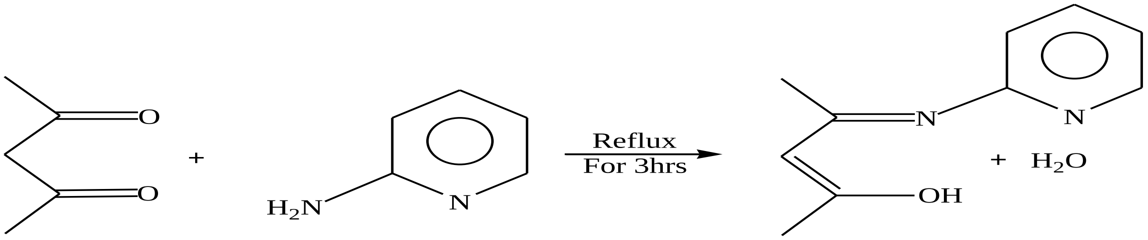

Figure 1: Preparation of Schiff base

Preparation of Metal (II) Complexes

The metal complexes were synthesized by mixingmethanolic solution (50cm^3) of the Schiff base (0.01mol) with anmethanolic solution (50cm^3) of the Manganese chloride (0.01mol). The resulting mixtures were refluxed for three hours. On cooling, complex that precipitated out was filtered, washed with methanol and ether and dried over P_2O_5 for 18 hours in desiccators (Aliyu and Al-Hakim, 2012).

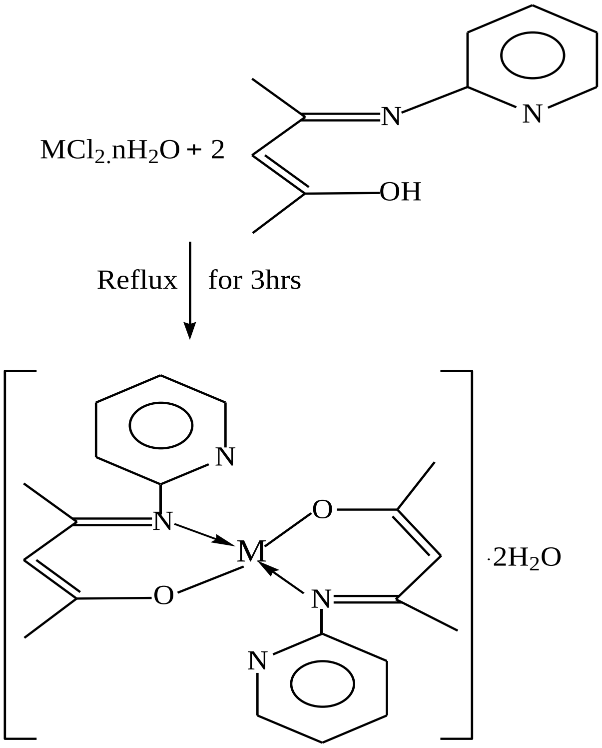

Figure 2: Preparation of Metal (II) Complexes

Antibacterial Activity

The *invitro* antibacterial activity of the Schiff base and its Metal (II) complexes was assayed by ager disc diffusion method using cultures of *Staphyllococcusaureus*, *Staphylococcus pyogen*and*Salmonellatyphi*. The Schiff base and its complexes were separately dissolved in DMSO to have concentration of 60, 30 and 15 µg/cm^3 respectively. Amoxicilin was used as reference drug. (Yusha'u and Salisu, 2011).

Antifungal Activity

The *invitro* antifungal activity of the Schiff base and its corresponding metal (II) complexes was assayed by ager dics diffusion method using cultures of *Aspergillusformigatus*, *RhizopusSpp.* and*Mucor Spp.* Using various concentration of 60, 30 and 15 µg/cm^3 respectively. Ketoconazole was used as reference drug. (Yusha'u and Salisu, 2011).

RESULTS AND DISCUSSION

Result

Results of several analyses of the synthesis acetyl acetone and 2-aminopyridine and its Metal (II) complexes are as follows: Table 1. Physical Properties of the Schiff base and its Metals (II) Complexes; table 2:Solubility test of the Schiff base and its Metals (II) Complexes;table 3: Molar Conductance of the Metals(II)Complexesin DMSO Solution; Table 4: Infrared spectral data of the Schiff base and its Metals(II) Complexes; Table 5: Magnetic Susceptibility Value of the Co(II) and Ni(II) Metals Complexes; table 6: Average percentage composition by weight of metal ion in the Complexes; table 7: Determination of water of crystallization in the Complexes; table 8:Sensitivity Test of the Schiff base and its Co(II) and Ni(II) MetalsComplexesagainstselected fungal isolates, table 9: Sensitivity Test of Schiff base and its Co(II) and Ni(II) MetalsComplexesagainst some bacterial isolates.

Table 1: Physical Properties of the Schiff base and its Metals (II) Complexes

Compounds	**Formular Weight**(g/mol)	**Colour**	**Percentage Yield**	**Melting Point (˚C)**	**Decomposition Temperature (˚C)**
Ligand	176	Brown	75.90	120	–
$[CoL_2].2H_2O$	447	Pale blue	81.82	–	198
$[NiL_2].2H_2O$	446	Light green	78.25	–	207

Ligand = $C_{10}H_{12}N_2O$

Table 2: Solubility test of the Schiff base and its Metals (II) Complexes

Compounds	H_2O	CH_3OH	C_2H_5OH	CCl_4	C_3H_6O	$C_6H_5NO_2$	C_6H_{14}	**DMF**	**DMSO**
Ligand	IS	SS	SS	IS	SS	SS	SS	S	S
$[CoL_2].2H_2O$	IS	SS	SS	IS	SS	IS	IS	S	S
$[NiL_2].2H_2O$	IS	SS	SS	IS	SS	IS	IS	S	S

Ligand (L)= $C_{10}H_{12}N_2O$ S =Soluble, IS = Insoluble, SS = Slightly Soluble, DMF=Dimethylformamide, DMSO =dimethylsulphoxide

Table 3: Molar Conductance of the Metals(II)Complexesin DMSO Solution

Compounds	**Concentration ($moldm^{-3}$) × 10^{-3}**	**Specific Conductance ($ohm^{-1}cm^{-1}$) × 10^{-6}**	**Molar Conductance ($ohm^{-1}cm^{-1}mol$)**
$[CoL_2].2H_2O$	1.00	5.20	5.20
$[NiL_2].2H_2O$	1.00	6.05	6.05

Table 4: Infrared spectral data of the Schiff base and its Metals(II) Complexes

Complexes	**ν(C=N)cm^{-1}**	**ν(M–N)cm^{-1}**	**ν(MO)cm^{-1}**	**ν(OH)cm^{-1}**
Ligand	1600	–	–	3332
$[CoL_2].2H_2O$	1611	724	491	3319
$[NiL_2].2H_2O$	1605	720	454	3362

L= Ligand

Table 5: Magnetic Susceptibility Value of the Co(II) and Ni(II) Metals Complexes

Compounds	**Gram magnetic susceptibility (X_g) (g^{-1})**	**Molar Magnetic Susceptibility (X_g) (mol^{-1})**	**μeff (B.M)**
$[CoL_2].2H_2O$	3.907×10^{-5}	7.935×10^{-3}	4.30
$[NiL_2].2H_2O$	6.68×10^{-6}	3.36×10^{-5}	3.38

L= ligand

Table 6: Average percentage composition by weight of metal ion in the Complexes

Complex	**Weight of ppt. (g)**	**Average weight of metal in the complexes = G.F× W.ppt (g)**	**Percentage composition of metal in the complex**
			Calculated
$[CoL_2].2H_2O$	0.1320	0.023364	11.68
$[CuL_2].2H_2O$	0.1397	0.0285	14.25

L= ligand, G.F = Gravimetric factor, Wppt = Weight of precipitate

Table 7: Determination of water of crystallization in the Complexes

Complexes	**Initial mass (g)**	**Final mass (g)**	**Loss in mass**	**% of water**
$[CoL_2].2H_2O$	0.2	0.187	0.013	6.5
$[NiL_2].2H_2O$	0.2	0.185	0.015	7.5

L= ligand

Table 8: Sensitivity Test of the Schiff base and its Co(II) and Ni(II) MetalsComplexesagainstselected fungal isolates

Test organism	**Compound**	**Zone of inhibition conc. (µg/disc)**		
		60	**30**	**15**
Aspergillusformigatus	Ligand(L)	12	13	10
	$[CoL_2].2H_2O$	16	15	10
	$[NiL_2].2H_2O$	14	12	08
	Standard	**32**	**26**	**15**
*Rhizopus*spp.	Ligand (L)	10	10	08
	$[CoL_2].2H_2O$	16	14	06
	$[NiL_2].2H_2O$	15	10	06
	Standard	**31**	**19**	**14**
*Mucor*spp.	Ligand (L)	12	10	06
	$[CoL_2].2H_2O$	14	06	06
	$[NiL_2].2H_2O$	14	08	06
	Standard	**32**	**18**	**12**

Standard = Ketoconozole (200mg)

Table 9: Sensitivity Test of Schiff base and its Co(II) and Ni(II) MetalsComplexesagainst some bacterial isolates

Test organism	**Compound**	**Zone of inhibition conc. (µg/disc)**		
		60	**30**	**15**
Staphylococcus aureus	Ligand (L)	16	15	12
	$[CoL_2].2H_2O$	16	14	10
	$[NiL_2].2H_2O$	17	16	14
	Standard	**27**	**19**	**18**
Staphylococcus pyogens	Ligand (L)	17	14	12
	$[CoL_2].2H_2O$	20	16	15
	$[NiL_2].2H_2O$	20	16	14
	Standard	**30**	**23**	**18**
Salmonella typhi	Ligand (L)	16	15	08
	$[CoL_2].2H_2O$	15	13	06
	$[NiL_2].2H_2O$	18	14	13
	Standard	**25**	**23**	**21**

Standard = Amoxicillin (500mg)

Discussion

The Schiff base was synthesed by condensing reaction of acetyl acetone and 2-aminco pyridine (Scheme 1) The Schiff base was obtained as light brown crystals with a melting point of 120°Cand percentage yields of 75%.Complexation of Schiff base and corresponding Metal(II) salts produced stable complexes of Co(II) and Ni(II) obtained to be pale blue and Light green colours with percentage yields of 81.82% and 78.25% for Co(II) and Ni(II) complexes respectively. The complexes decomposed at a temperature range of 198 – 207°C thus confirming stability as affirmed by the work of (Osowole*et al.*, 2015). The high decomposition temperature suggested a good thermal stability of the complex as shown in (Table 1).The solubility test of the TheSchiff base and itsMatel (II) complex were soluble in dimethyl isulphoxide (DMSO), dimethyl formamide (DMF), this may be attributed to their high polarity but insoluble in carbon tetrachloride (CCl_4), nitrobenzene and petroleum ether as show in (Table 2). Determination of metal-ligand ratio was determined using Job’s method of continuous variation (UV-visible). (Renny*et al.*, 2013), this is an agreement with report of (Tomasevich*et al.*, 2013; Bharti*et al.*, 2015). The molar conductance values (Table 3) of the synthesized complex obtained was found to be in range 5.20 – 6.05 $ohm^{-1}mol^{-1}cm^{-3}$, these low values suggested that the complexes are non electrolytes, similar results were reported in the literature by (Eman *et al.*, 2015 and Osuwole*et al.*, 2015). The magnetic moment of the complex determined at room temperature were found to be in range 4.30 – 3.38BM this positive value indicated that the complex is paramagnetic in nature. The percentage composition of the metal ions, were found gravimetrically. The result obtained was in good agreement with calculated values as can be seen in (Table 6) the same procedure was adopted by Yoder (2015). In determining similar metal ions. The percentage of water crystallization in the complexes was determined and the result from table 7 confirmed the presence of water of water crystallization in the complex with percentage 8.0%, the same procedure was adopted by Aliyu and Abdullahi (2009). The percentage composition of the ligand was determined by difference and the results obtained were within the calculated value. The empirical formulae of the Metal (II) complex was determined from the percentage compositions of the Metal(II) ion, the ligand and the water of crystallization. The results obtained suggested the general formula of $[MnL_2]2H_2O$. The FT-IR spectra were recorded in KBr pellet using Agilent Technology FT-IR spectrometer (4000–400) cm^{-1}. The significant IR band for the ligand as well as its complexes are compiled and presented in (Table 4) in the IR spectrum of the Schiff base ligand a sharp band observed at 1600cm^{-1} is assigned to the ν(–C=N–H) mode of azomethine group, this band is shifted to the of 1605 cm^{-1} in the spectra of the complex indicating coordination of the azomethine nitrogen atom to the central metal ion and is supported by the appearance of new peaks in the range of 763cm^{-1} in the spectra which was due to metal to nitrogen bond (M – N) (Fugu*et al* 2013). A band at the range of (3293)cm^{-1} is due to ν(C–OH) phenolic group was observed in the ligand which is formed as a result of tautomerism.The disappearance of phenolic ν(OH) band in the complex suggested that coordination by phenolic oxygen after deprotonation with metal ion. This is further supported by shifting of ν(C–O) phenolic band to higher frequency in all the complexes. The appearance of band at the range of 491cm^{-1} is due to ν(M–O) suggestedbydeprotonation of OH of the ligand. In the IR spectra of the complexes there is bands at the range of 3362cm^{-1} is observed suggesting water crystallization in the complexes (Aliyu and Abdullahi, 2009) (Table 7). This indicated the presence of water crystallization in the complexes. The ligand and its synthesized metal (II) complex was screened for antibacterial and antifungal activities against three bacterial isolates (*Staphylococcus aureus*, *Streptococcus pyogens* and salmonella typhi) and three fungal isolates (*Aspergillusfungitus*,*Rhizopus*spp. and *Mucor*spp) using disc diffusion method in DMSO respectively. The Schiff base and its Metal (II) complexshowed good activity against all the tested organisms, how ever the activity is more at higher concentration in al cases. Comparative studies show that the Metal (II) complex show significant activity than the ligand but lower than the ligand but lower than the reference drugs as can be seen in (Table 8 and 9) The result obtained are similar to that of Prakash and Adakhari (2011).

CONCLUSION

The Schiff base ligand derived from condensation of 2-aminopyridine with acetyl acetone and its Metal(II) complexes has been synthesized and characterized by solubility test, melting point, decomposition temperature, molar conductance, magnetic susceptibility, FTIR analysis, UV visible, gravimetric analysis. The antimicrobial activity of the ligand as well as the Metal(II) complex were tested. The FTIR anslysis revealed that the ligand is dentate. The molar conductance values of the complexes show low value which demonstrated the nonelectrolyticnature of the complexes. The melting point and decomposition temperature of the ligand and complex showed good thermal stability. The UV-visible confirmed that the metal: ligand ratio is 1:2, and the gravimetric method of analysis confirmed the empirical formulae of the complexes as $[M(C_{10}H_{12}N_2O)_2]2H_2O$. The magnetic susceptibility measurement values of the complexes revealed that all the complexes are paramagnetic. The result of the antimicrobial test revealed that the biological activity of the ligand is enhanced when its presented in the form of a Metal (II) complexes.

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SUPPLEMENTED DATA

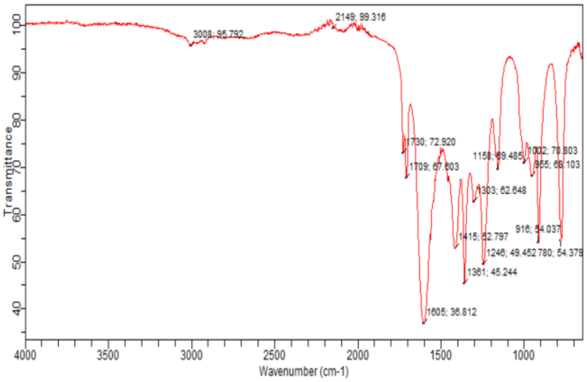


Fig. 1: Acetyl acetone (ACAC)

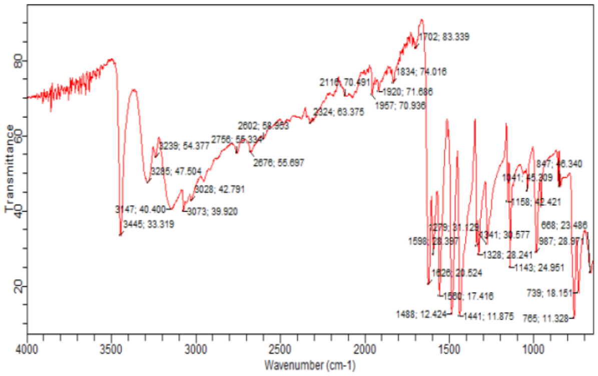


Fig. 2: 2-amino pyridine

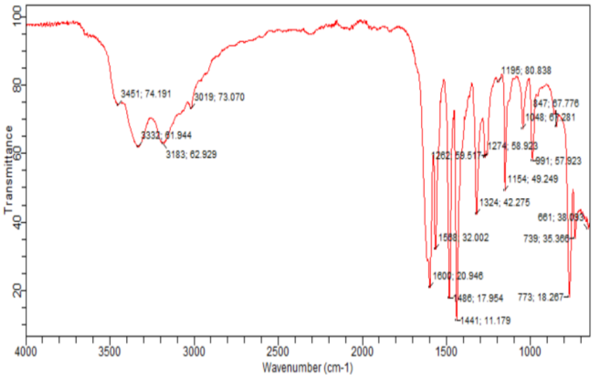


Fig. 3: Schiff base

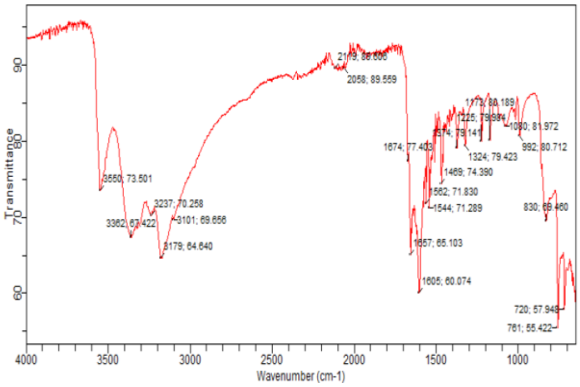


Fig. 4: Co (II) Complex

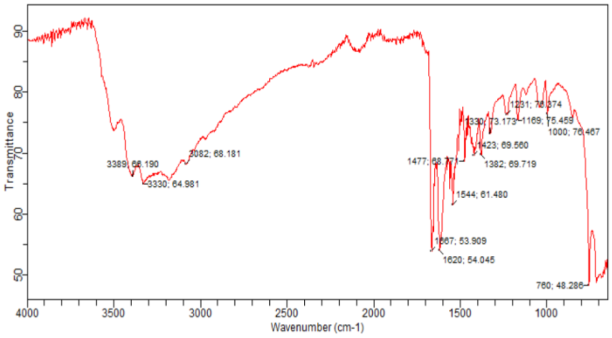
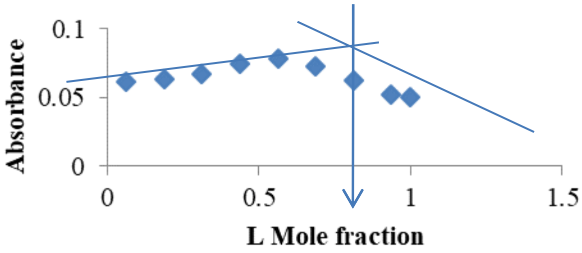
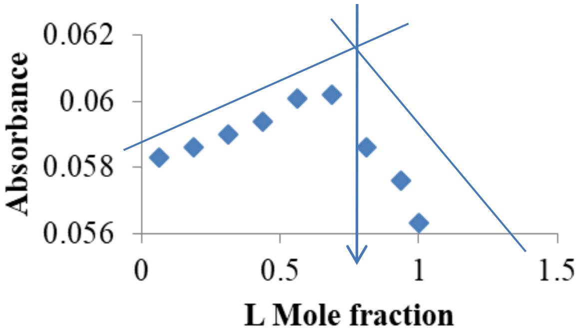


Fig. 5: Ni (II) complex



Plot of absorbance against ligand mole fraction at λ_{max}620nm for Co(II) complex.



Plot of absorbance against ligand mole fraction at λ_{max}700nm for Ni(II) complex