

Synthesis and Characterization Complex of Group 12 Iminopyridin

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ABSTRACT

In this work, Complexes of the group 12 iminopyridin is synthesized along with the spectrometry using the ligand. Vibration spectrums are provided by the use of the BUCKER apparatus. Also, the ¹HNMR and ¹³CNMR spectrums are provided using the Avance BURKER-300MHZ. Therefore, resulted spectrums are an indication of the formation of the complex

KEYWORDS: Schiff base, Complex, Ligand, 2,3 Dimethyl Aniline.

INTRODUCTION

The chemistry of the carbon-nitrogen double bond plays a vital role in the progresses of chemistry science [1]. Schiff bases have often been used as chelating ligands in the field of coordination chemistry and their metal complexes are of great interest for many years [2].

The Schiff base ligands with sulphur and nitrogen donor atoms in their structures act as good chelating agents for the transition and non-transition metal ions [3]. Schiff bases [4] were still regarded as one of the most potential group of chelators for facile preparations of metallo-organic hybrid materials. The interest in Schiff base compounds as analytical reagents is increasing since they enable simple and unexpensive determinations of different organic and inorganic substances [5].

Schiff base metal complexes have been known since the nineteenth century. Investigation on metal-organic complexes represents one of the most active areas of material science and chemical research [6] Schiff bases form a class of compounds with azomethin group, which are usually synthesized from the condensation of primary amines and active carbonyl groups by the elimination of water molecule.

2. Experimental

2.1. Complex Syntheses by HgCl₂

For the preparation of the title compound, a solution of 2-[(2,3-dimethylphenyl) iminomethyl] pyridine (0.210 g, 1.00 mmol) in acetonitril (10 ml) was added slowly to a solution of HgCl₂ (0.271 g, 1.00 mmol) in acetonitril (10 ml) and the resulting yellow solution was stirred for 45 min at room temperature. Then the yellow precipitate was filtered and dissolved in acetonitril and left to evaporate slowly at -18°C. After a few days, yellow crystals of the title compound were isolated (yield: 0.272 g, 56.5%; m.p. 489 K).

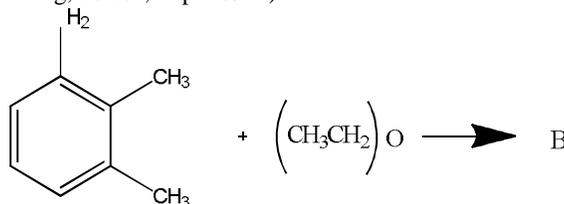


Figure 1. Solution of the 2,3 Dimethyl Aniline in the ether (B)

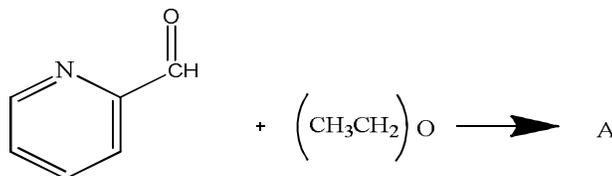


Figure 2. Solution of the pyridin carbaldehyd in the ether (A)

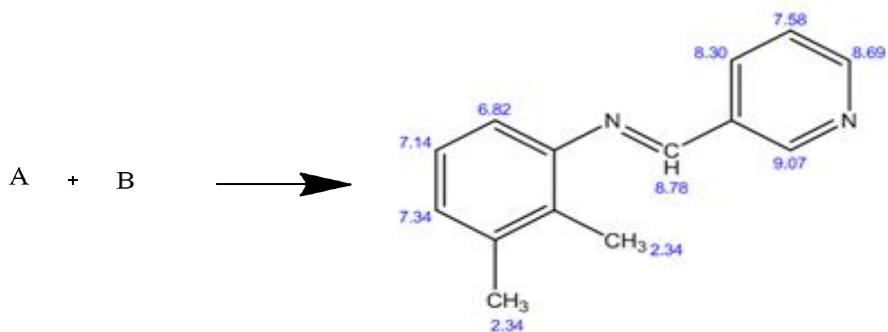


Figure 3. Mixing A and B for forming the Ligand

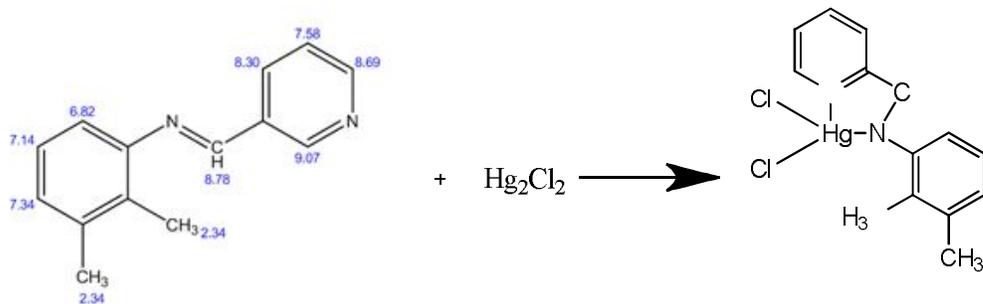


Figure 4. Formation of the complex using the additional of the metal to the ligand

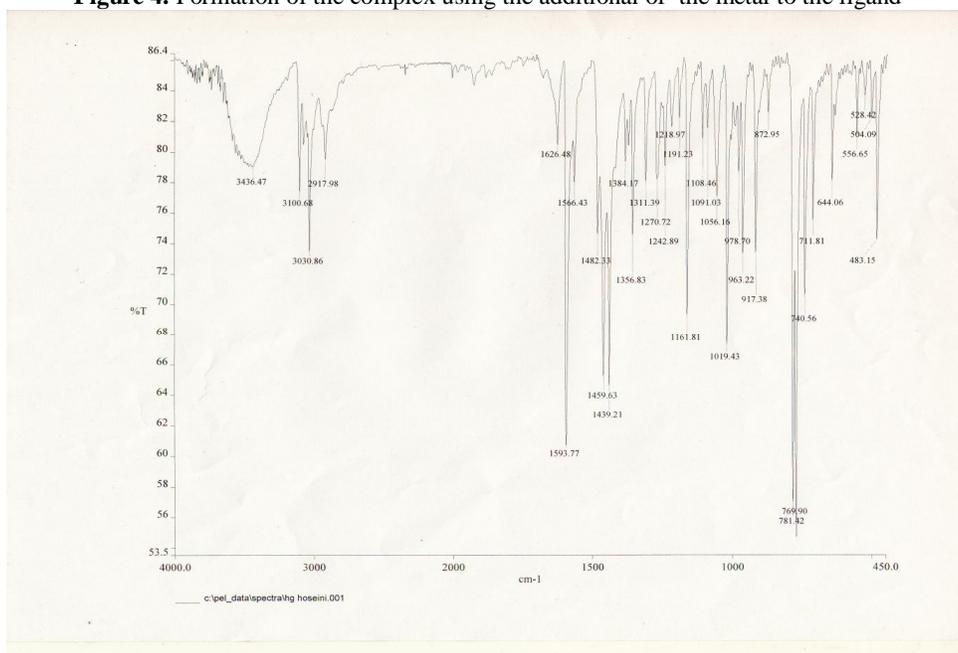


Figure 5. Spectrum of the IR for the Complex

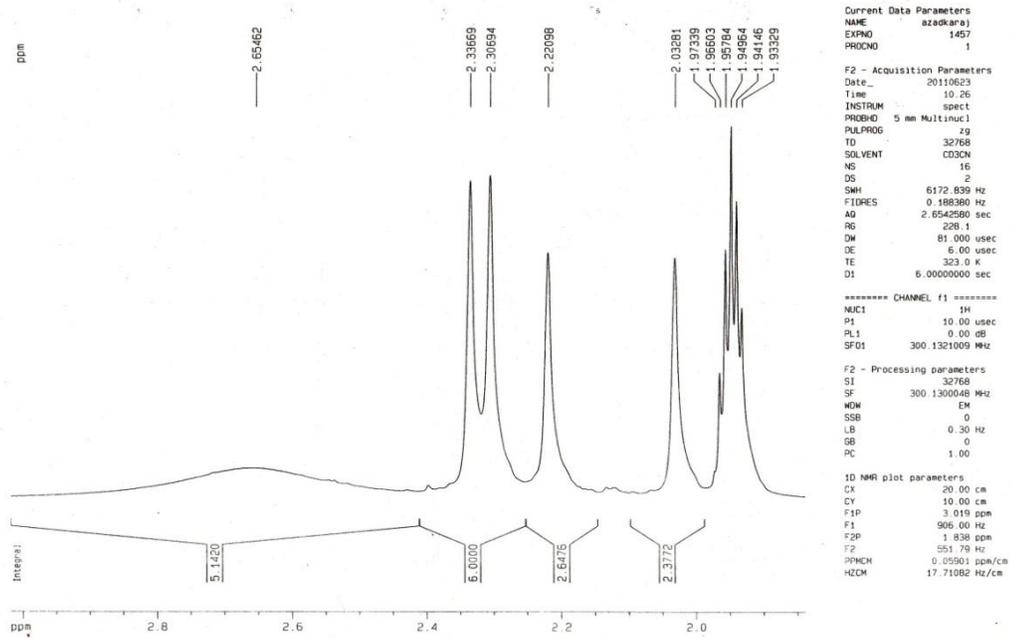


Figure 6. Spectrum of the NMR for the Complex

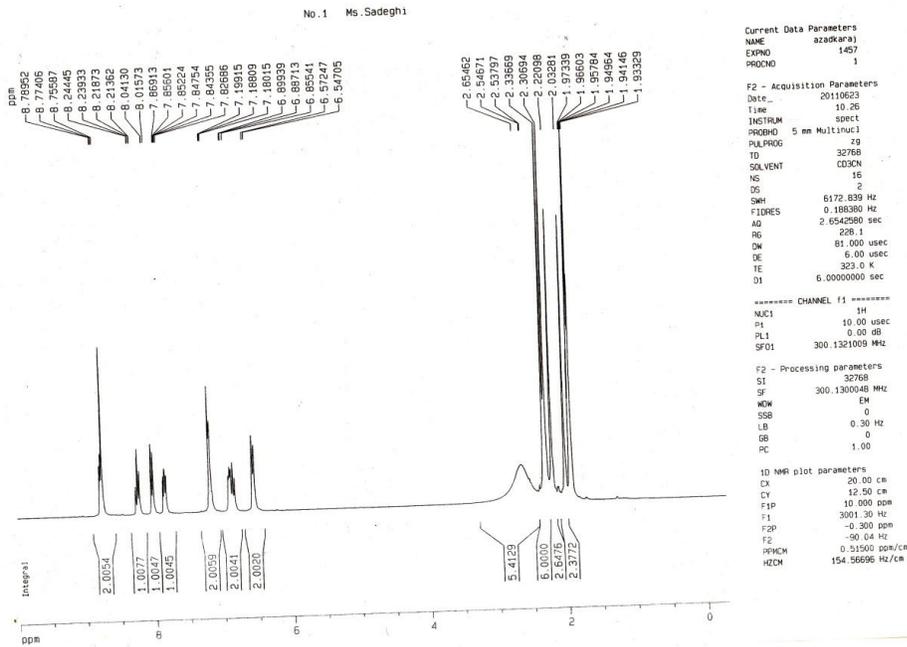


Figure 7. Spectrum of the ¹H NMR for the Complex

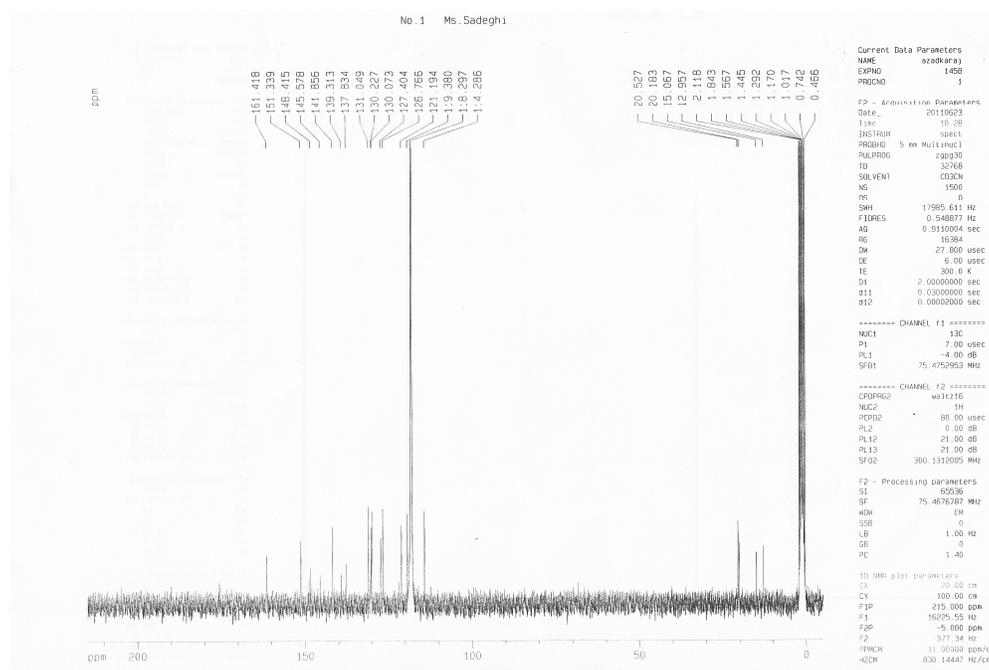


Figure 8. Spectrum of the ^{13}C NMR for the Complex

According to Fig. 5, the band of 3436 cm^{-1} is related to frequency of C-H Aromatic. The band of 1593 cm^{-1} is respected to C=N which is verified the imin. The bands of $1356\text{--}1566\text{ cm}^{-1}$ is related to the C=C benzen. The strong bands of $740\text{--}978\text{ cm}^{-1}$ is related to the bending frequency of Aromatic.

In accordance with Figs. 6 and 7, the region of 8.75ppm indicates the hydrogen imin. the regions of 7.15-7.19ppm indicates the hydrogens Aromatic. the regions of 1.94-1.96ppm indicates the hydrogens methyl.

Based on Fig. 8, the region of 15.52ppm indicates the carbon methyl. the region of 20.52ppm indicates the carbon methylen. the region of 161ppm indicates the carbon imin. the regions of 114-121ppm indicates the carbons Aromatic. the regions of 137-151ppm indicates the carbons pyridin.

2.2. Complex Syntheses by ZnCl_2

For the preparation of the title compound, a solution of 2-[(2,3-dimethylphenyl) iminomethyl] pyridine (0.210 g, 1.00 mmol) in acetonitril (10 ml) was added slowly to a solution of ZnCl_2 (0.014 g, 1.00 mmol) in methanol (10 ml) and the resulting yellow solution was stirred for 45 min at room temperature. Then the yellow precipiate was filtered and dissolved in acetonitril

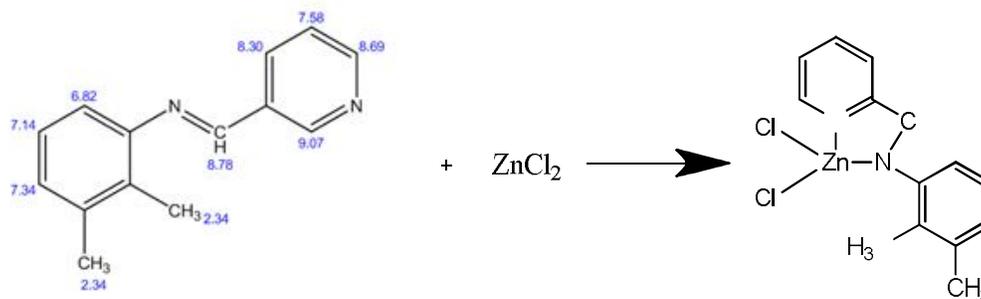


Figure 9. Formation of the complex using the additional of the metal to the ligand

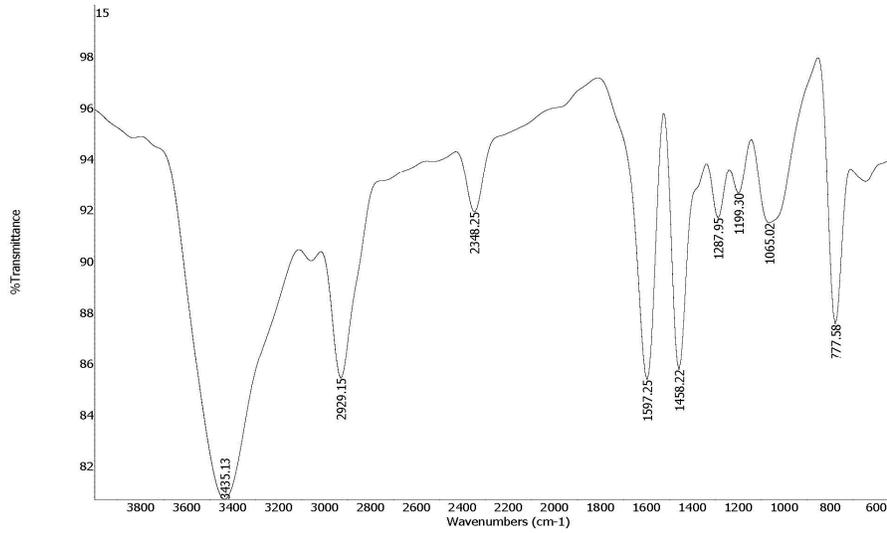
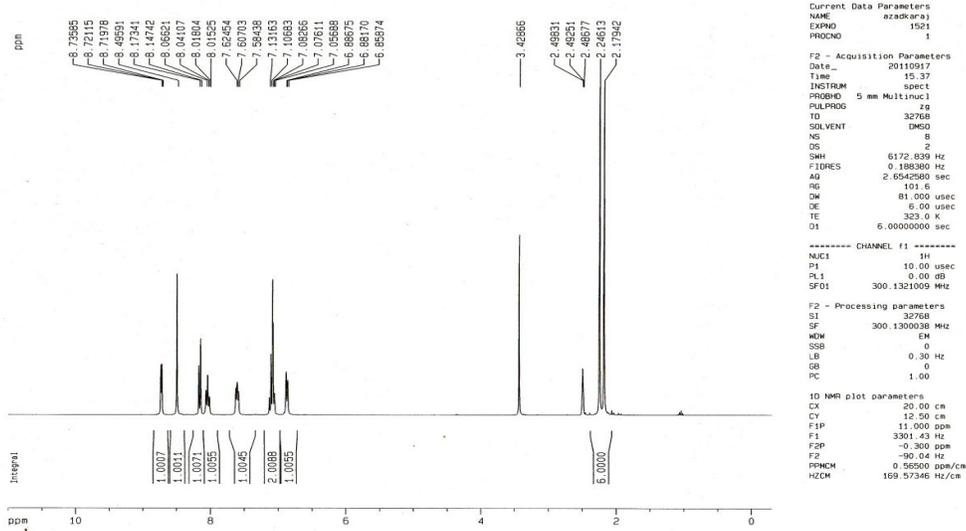


Figure 10. Spectrum of the IR for the Complex



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PROCNO 1

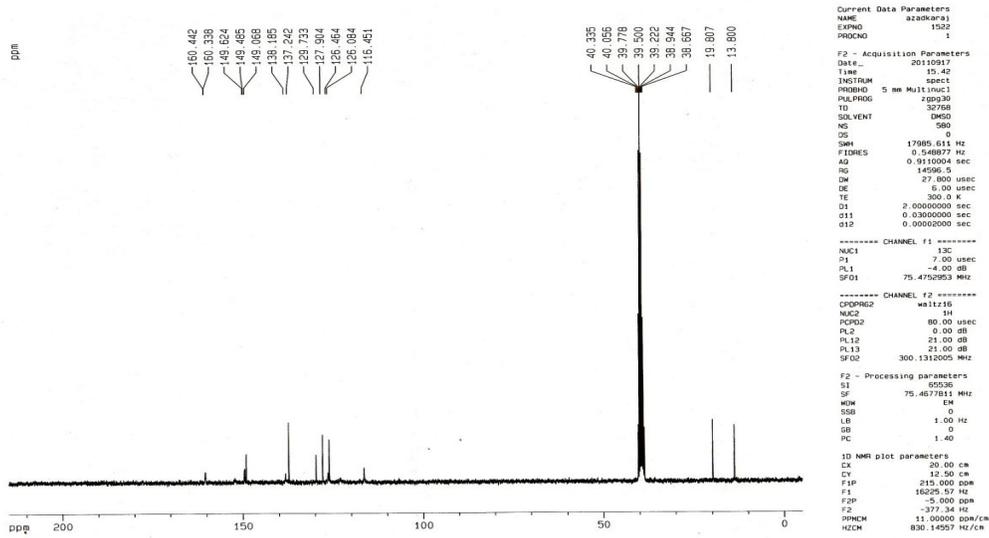
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Date_ 20110917
Time 15.37
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.188360 Hz
AQ 2.6542980 sec
RG 101.6
DM 81.000 usec
DE 6.00 usec
TE 293.0 K
DQ1 6.00000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300638 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPHM 0.56500 ppm/cm
HZCM 169.57346 Hz/cm
    
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Figure 11. Spectrum of the ¹H NMR for the Complex



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Current Data Parameters
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EXPNO 1532
PROCNO 1

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PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110084 sec
RG 14596.5
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
DQ1 2.00000000 sec
DQ2 0.03000000 sec
DQ3 0.00000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SFO1 75.4752953 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677511 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 213.000 ppm
F1 16205.97 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPHM 11.00000 ppm/cm
HZCM 830.14957 Hz/cm
    
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Figure 12. Spectrum of the ¹³C NMR for the Complex

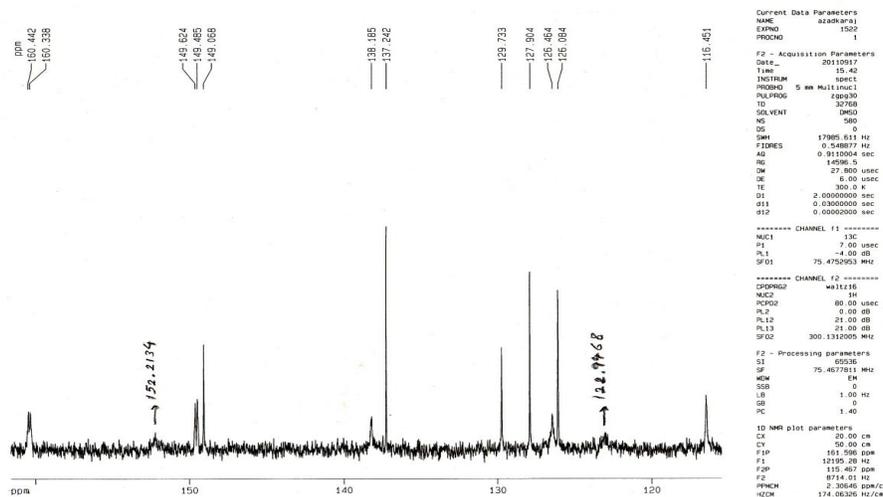


Figure 13. Spectrum of the ^{13}C NMR for the Complex

According to Fig. 10, the band of 3620 cm^{-1} is related to frequency of C-H Aromatic. The band of 1597 cm^{-1} is respected to C=N which is verified the imin. The bands of $1065\text{--}1287\text{ cm}^{-1}$ is related to the C=C benzen. The strong band of 777.58 cm^{-1} is related to the bending frequency of Aromatic.

In accordance with Figs. 11, the region of 8.49ppm indicates the hydrogen imin. the regions of 7.58-8.17ppm indicates the hydrogens Aromatic. the region of 2.24ppm indicates the hydrogens methyl.

Based on Fig. 12 and 13, the region of 13.8ppm indicates the carbon methyl. the region of 19.8ppm indicates the carbon methylen. the region of 149.62ppm indicates the carbon imin. the regions of 116-129ppm indicates the carbons Aromatic. the regions of 149.06-149.62ppm indicates the carbons pyridine.

2.3. Complex Syntheses by CdCl_2

For the preparation of the title compound, a solution of 2-[(2,3-dimethylphenyl) iminomethyl] pyridine (0.210 g, 1.00 mmol) in acetonitril (10 ml) was added slowly to a solution of CdCl_2 (0.182 g, 1.00 mmol) in H_2O (10 ml) and the resulting yellow solution was stirred for 45 min at room temperature. Then the yellow precipitate was filtered and dissolved in acetonitrile.

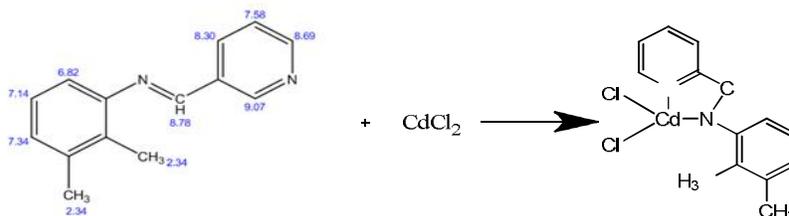


Figure 14. Formation of the complex using the additional of the metal to the ligand

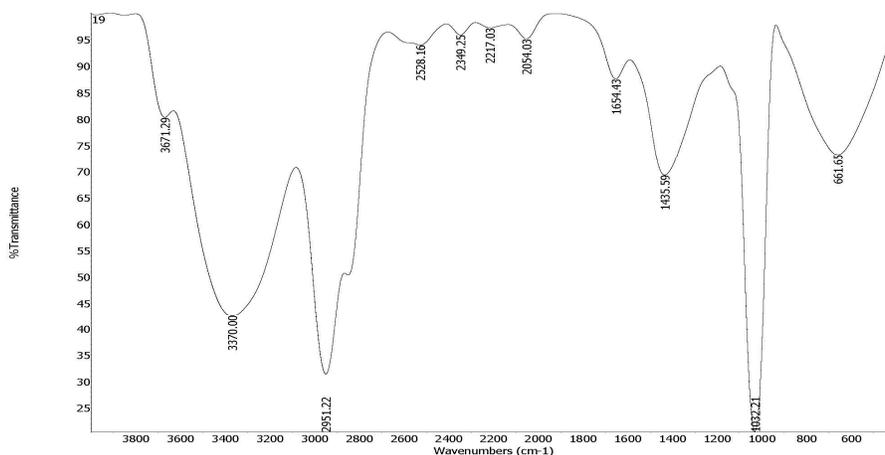


Figure 15. Spectrum of the IR for the Complex

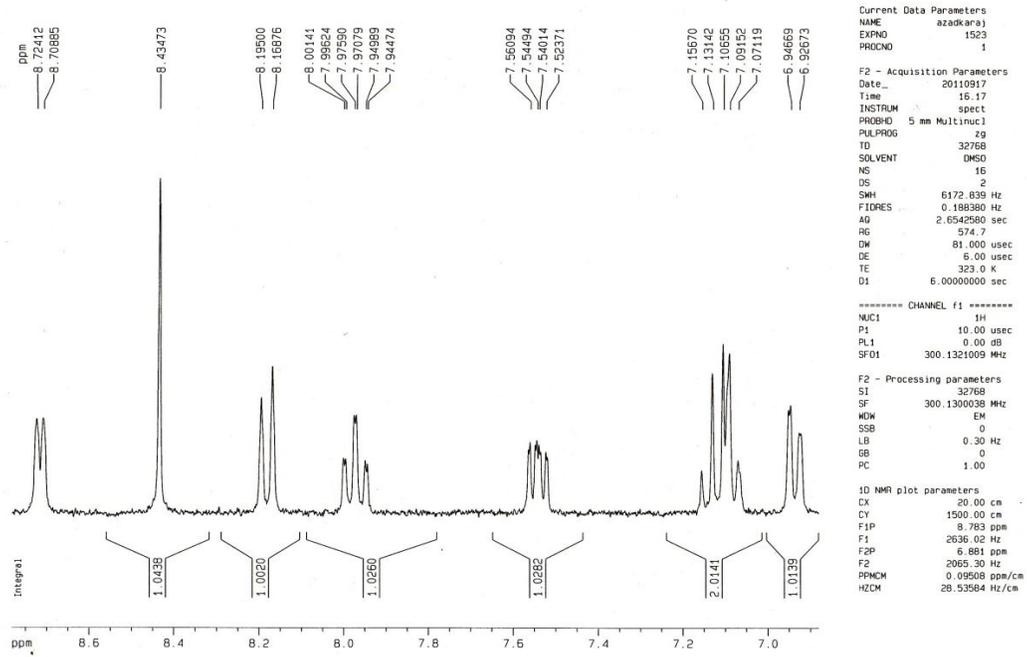


Figure 16. Spectrum of the ¹H NMR for the Complex

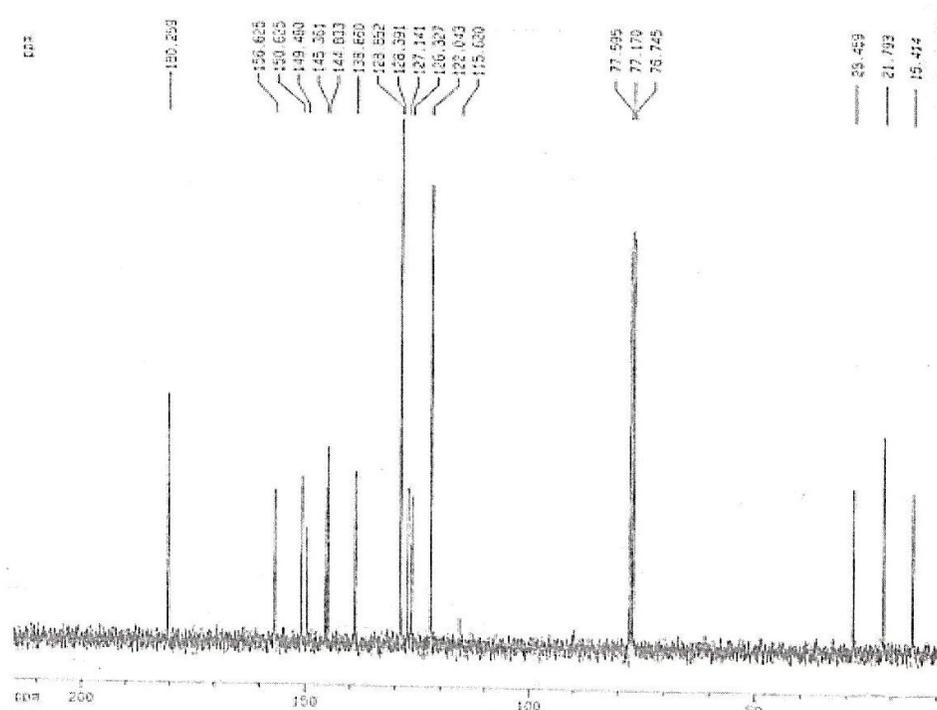


Figure 17. Spectrum of the ¹³C NMR for the Complex

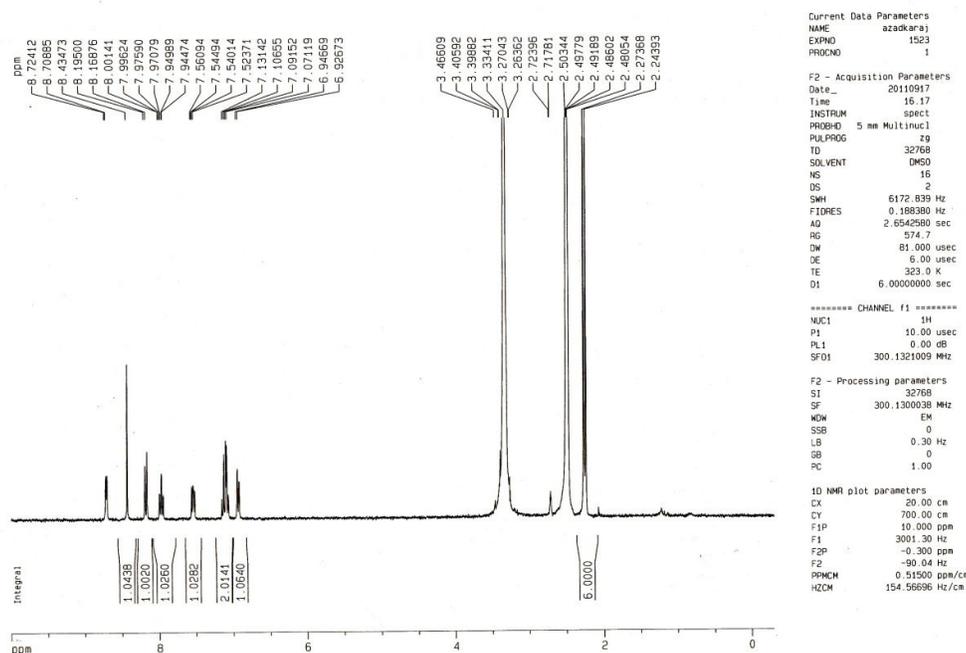


Figure 18. Spectrum of the ^1H NMR for the Complex

According to Fig. 15, the band of 3348 cm^{-1} is related to frequency of C-H Aromatic. The band of 1612 cm^{-1} is respected to C=N which is verified the imin. The bands of $1400\text{-}1600\text{ cm}^{-1}$ is related to the C=C benzen. The strong bands of $742\text{-}776\text{ cm}^{-1}$ is related to the bending frequency of Aromatic.

In accordance with Figs. 16 and 18, the region of 8.19ppm indicates the hydrogen imin. the regions of 7.52-8.16ppm indicates the hydrogens Aromatic. the region of 3.33ppm indicates the hydrogens methyl.

Based on Fig. 17, the region of 15ppm indicates the carbon methyl. the region of 28ppm indicates the carbon methylen. the region of 156ppm indicates the carbon imin. the regions of 115-128ppm indicates the carbons Aromatic. the regions of 137-151ppm indicates the carbons pyridin.

3. Conclusion

In this paper, we synthesizes the Complexes of the group 12 iminopyridin experimentally along with the spectrometry of them using the ligand. the BUCKER apparatus provides the vibration spectrums and the Avance BURKER-300MHz provides the ^1H NMR and ^{13}C NMR. Consequently, the obtained spectrums verifies the complex formation.

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