# Microwave-assisted synthesis of Cd (II) complex of 4-pyridinecarboxaldehyde thiosemicarbazone

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**Abstract**

In this work, dichloro 4-pyridinecarboxaldehyde thiosemicarbazone cadmium (II) was synthesized by an efficient and facile microwave heating technique. The reaction was performed using $CdCl_2.H_2O$ as metal source and 4-pyridinecarboxaldehyde thiosemicarbazone as ligand in 1:1molar ratio in EtOH under microwave irradiation with the power of 100 W for 30 sec. The progress of the reaction was monitored by TLC. The product was characterized by Fourier transform infrared (FT-IR), $^{1}H$-, $^{13}C$- NMR spectroscopy and elemental analysis.

**Keywords:** Microwave, Thiosemicarbazide, 4-Pyridinecarbaldehyde, Cadmium.

## 1. Introduction

Thiosemicarbazones were obtained by condensation of thiosemicarbazide with suitable aldehydes or ketones, act as ligand in the preparation of their complexes. They bind to the metal through N and S atoms. Thiosemicarbazones and their metal complexes may have biological properties such as anticancer, fungicides, antibacterial, antiviral, antifungal, antiHIV, antitumour and so on [1-5]. Microwave irradiation nowadays is an accepted tool for accelerating the

organic and inorganic reactions[6]. In the present work, we report the synthesis of Cd(II) complex of 4-pyridinecarboxaldehyde thiosemicarbazone under microwave irradiation. The product was characterized by FT-IR, $^{1}$H-NMR, $^{13}$C-NMR and elemental analysis.

## 2. Experimental

All the chemicals were purchased from Merck Co. and were used as received.

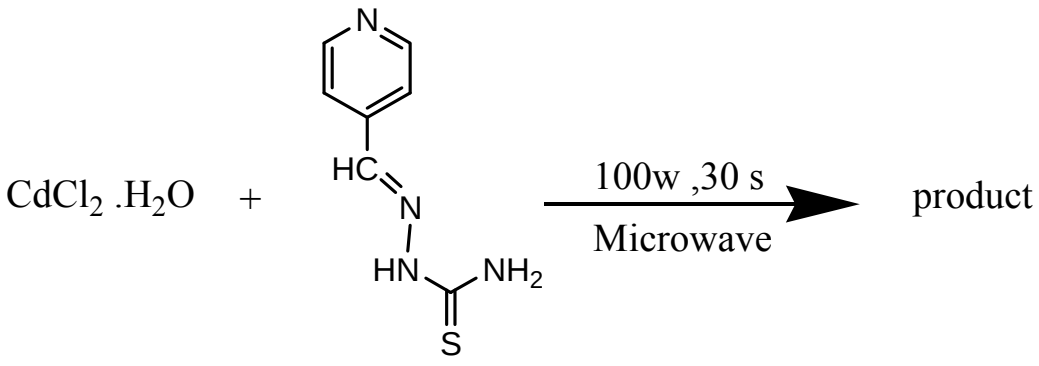
### 2.1. Synthesis of di chloro 4-pyridinecarboxaldehyde thiosemicarbazone cadmium (II)

The reaction was performed using $CdCl_2.H_2O$ as metal source and 4-pyridinecarboxaldehyde thiosemicarbazone as ligand in 1:1 molar ratio in EtOH under microwave irradiation with the power of 100 W for 30 sec. The progress of the reaction was monitored by TLC. After the completion of the reaction, a solid white compound was formed (Scheme 1). Then it was washed with ethanol and dried. Found: C, 24.29; H, 2.18; N, 15.27 Calc. for $C_7H_8N_4SCdCl_2$: C, 23.14; H, 2.20; N, 15.42 %.

IR (KBr, $cm^{-1}$): 3440(m), 3317(m), 3172(m), 1541(s), 1326(s), 823(m), 655(m), 530(m).

$^{1}$HNMR (DMSO-$d_6$, *ppm*): δH: 7.78(s, 2H), 8.21(s, 2H), 8.39 (s, 1H), 8.57 (d, 2H), 11.69(s, 1H).

$^{13}$CNMR (DMSO-$d_6$, *ppm*): δC: 121.88, 139.93, 142.66, 150.30, 178.68.




Scheme 1. The synthesis of $C_7H_8N_4SCdCl_2$

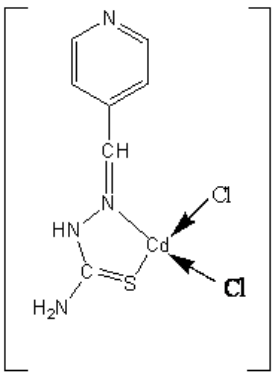
## 3. Results and discussion

In the FT-IR spectrum, the appeared band at 3440 $cm^{-1}$ can be attributed to the asymmetric (N–H) vibrations of $NH_2$ group. The other bands at 3317 and 3172 $cm^{-1}$ may be due to the symmetric (N–H) vibrations of the imino and amino groups. The band at 1541 $cm^{-1}$ is related to ν(C=N). The ν(C=S) stretching frequency is observed at 823 $cm^{-1}$. These results indicate that the 4-pyridinecarboxaldehyde thiosemicarbazone as a ligand was coordinated to the Cd(II) ion through the N azomethane and sulfur atoms. Thus, it has been concluded that ligand acts as a bidentate agent. The coordination of the metal ions via both of nitrogen and sulfur atoms is confirmed by the presence of band at 655 $cm^{-1}$due to ν(M–N) assignment [7-8].

In $^{1}$H-NMR spectrum, the singlet resonances are observed at δ = 11.69 and 8.39 ppm, which are assigned to aldehyde CH and hydrazide NH groups, respectively. The observed peaks at δ = 7.78 and 8.57 ppm belong to pyridine protons.

There are five signals in $^{13}$C-NMR spectrum. The appeared peak at δ = 178.68 is related to C=S.

According to spectropic data and elemental analysis, the structure of the Cd (II) complex of 4-pyridinecarboxaldehyde thiosemicarbazone can be proposed as shown in Scheme 2.




Scheme 2. The suggested structure of the complex, $[Cd(C_7H_8N_4S)Cl_2]$

The synthesis of Cd(II) complex of 4-pyridinecarboxaldehyde thiosemicarbazone, di chloro 4-pyridinecarboxaldehyde thiosemicarbazone cadmium (II), under microwave irradiation has been done during 30 sec. This investigation showed that the used method has several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures in comparison with conventional processes [9-10].

**Acknowledgements**

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