# Preparation of 2-pyridinecarbaldehyde thiosemicarbazone by Microwave irradiation

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**Abstract:** A fast and efficient method has been developed for the synthesis of 2-pyridinecarbaldehyde thiosemicarbazone under microwave irradiation. 2-pyridinecarbaldehyde thiosemicarbazone has been synthesized by the reaction of 2-pyridinecarbaldehyde and thiosemicarbazide using microwave irradiation.
This compound has been characterized by FT-IR, $^{1}$H-NMR, $^{13}$C-NMR.

**Keywords:** thiosemicarbazide; microwave; 2-pyridinecarbaldehyde

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## 1. Introduction

Thiosemicarbanzones have received considerable attention because of their pharmacological activities. They have numerous biological activities, e.g. anticarcinogenic, antibacterial, anti-HIV, anticancer, fungicides, antiviral, antifungal, antitumour, etc[1]. These compounds containing thione (C =S) and thiole (C-S) groups occupy an important position among organic reagents as potentialdonor ligands for transition metal ions. Thiosemicarbazones usually act as chelating ligands with transition metal ion, bonding through the sulfur and hydrazine nitrogen atoms[2]. Chandra and co-workers have been reported the synthesis of title compound by the direct reaction 2- pyridine-carbaldehyde and thiosemicarbazide by reflux[3].

The application of microwave techniques for organic synthesis has attracted considerable interest in recent years. Microwave assisted organic synthesis has proven to be a valuable technique for reducing reaction times, giving cleaner reactions, improving yields, simplifying work-up and designing energy-saving protocols[4-9].

In the present work, we report the synthesis of 2-pyridinecarbaldehyde thiosemicarbazone under microwave irradiation. This compound was characterized by FT-IR, $^{1}$H-NMR, $^{13}$C-NMR.

## 2. Experimental

Hot ethanolic solution of thiosemicarbazide (1.1375g, 0.0125mol) and 2-pyridinecarboxaldehyde (1.1875 mL, 0.0125mol) were mixed slowly with constant stirring and then irradiated at 900W for 90sec. The progress of the reaction was monitored by TLC. After the completion of the reaction, a solid compound was formed. Then it was washed with ethanol and dried.

(mp. 207-208 °C)

**IR (KBr, *cm*$^{-1}$):** 3434(m), 3261(m), 3161(s), 1608(s), 742(m), 619(m).

**$^{1}$HNMR (DMSO, *ppm*) δH:** 7.35(t, 1H), 7.80(t, 1H), 8.20 (d, 1H), 8.23 (d, 1H), 8.35 (s, 1H), 8.53(d, 1H), 11.64 (s, 1H).

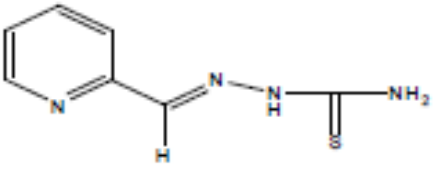
**$^{13}$CNMR (DMSO, *ppm*) δC:** 120.82, 124.68, 137.13, 142.97, 149.79 ($C_5H_4$ N ring), 153.68 (C=N), 178.74 (C=S).

## Results and Discussion

The structure of title compound has been assigned by spectroscopic data. In the IR spectrum, the highest frequency band of the 2-pyridinecarboxaldehyde

thiosemicarbazone at 3434 $cm^{-1}$can be assigned to the asymmetric ⁻(N–H) vibration of the terminal $NH_2$ group. The other bands at 3261 and 3161 $cm^{-1}$ may be due to the symmetric⁻(N–H) vibrations of the imino and amino groups. The band ν(C=N) appeares at 1608 $cm^{-1}$. The ν(C=S) stretching frequency is observed at 742 $cm^{-1}$.

The $^{1}H$ NMR spectrum displayed singlet peaks at δ = 11.64 and 8.35 *ppm* are assigned to hydrazide NH and aldehyde CH groups, respectively. The observed doublet peaks at δ = 8.23 and 8.20 *ppm* are related to $NH_2$ groups. The other appeared signals belong to pyridine protons. There are seven signals in $^{13}$CNMR spectrum. The observed peak at δ = 178.74 *ppm* could be assigned to to C=S. Based on the presented $^{1}$HNMR, $^{13}$CNMR and FT-IR spectroscopy for this compound, structure can be proposed as shown in scheme 1.




Scheme 1. The structure of 2-pyridinecarbaldehyde thiosemicarbazone

The synthesis of 2-pyridinecarbaldehyde thiosemicarbazone under microwave irradiation has been done during 90 sec when compared with conventional processes, which need to a long reaction time. This investigation showed that the used method has several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures when compared with conventional processes.

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