

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, ¹H-NMR, ¹³C-NMR.

Keywords: thiosemicarbazide; microwave; 2-pyridinecarbaldehyde

1. Introduction

Thiosemicarbazones 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 potential donor 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].

In the present work, we report the synthesis of 2-pyridinecarbaldehyde thiosemicarbazone under microwave irradiation. The preparation of title compound by this way has several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures[4,. This investigation showed that the used method and the results when compared with conventional processes were found to be inexpensive, more friendly and high yielding.

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).

1H NMR (DMSO, ppm) δ H: 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).

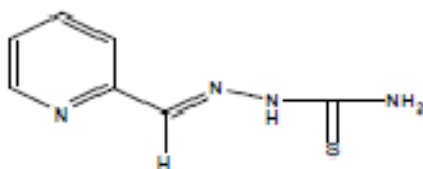
^{13}C NMR (DMSO, ppm) δ C: 120.82, 124.68, 137.13, 142.97, 149.79 (C_5H_4 N ring), 153.68 (C=N), 178.74 (C=S).

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 NH₂ 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 $\nu(\text{C}=\text{N})$ appears at 1608 cm⁻¹. The $\nu(\text{C}=\text{S})$ stretching frequency is observed at 742 cm⁻¹.

The ¹H NMR spectrum displayed singlet peaks at $\delta = 11.64$ and 8.35 ppm are assigned to hydrazide NH and aldehyde CH groups, respectively. The observed doublet peaks at $\delta = 8.23$ and 8.20 ppm are related to NH₂ groups. The other appeared signals belong to pyridine protons. There are seven signals in ¹³CNMR spectrum. The observed peak at $\delta = 178.74$ ppm could be assigned to C=S. Based on the presented ¹HNMR, ¹³CNMR and FT-IR spectroscopy for this compound, structure can be proposed as shown in scheme 1.



Scheme 1. The structure of 2-pyridinecarbaldehyde thiosemicarbazone

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