Synthesis and characterization of 4-pyridinecarboxaldehyde thiosemicarbazone

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Abstract

In this work, 4-pyridinecarboxaldehyde thiosemicarbazone was synthesized by two methods, reflux and microwave and got the same results. The progress of the reaction was monitored by TLC. After the completion of the reaction, a yellow compound was formed, which was characterized by Fourier transform infrared (FT-IR), ^{1}H-, ^{13}C- NMR spectroscopy and elemental analysis.

Keywords: Thiosemicarbazide, 4-Pyridinecarbaldehyde, Spectroscopy.

1. Introduction

Thiosemicarbazones belong to a large group of thiourea derivatives, which obtained by condensation of thiosemicarbazide with suitable aldehydes or ketones. They have biological activities, such as antitumour, antiviral, anticancer, antifungal, antibacterial and antimalarial [1-7]. In this work, the title compound was synthesized by two methods, reflux and microwave. This compound, 4-pyridinecarboxaldehyde thiosemicarbazone, was characterized by elemental analysis, FT-IR, ^{1}H-NMR, ^{13}C-NMR.

2. Experimental

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

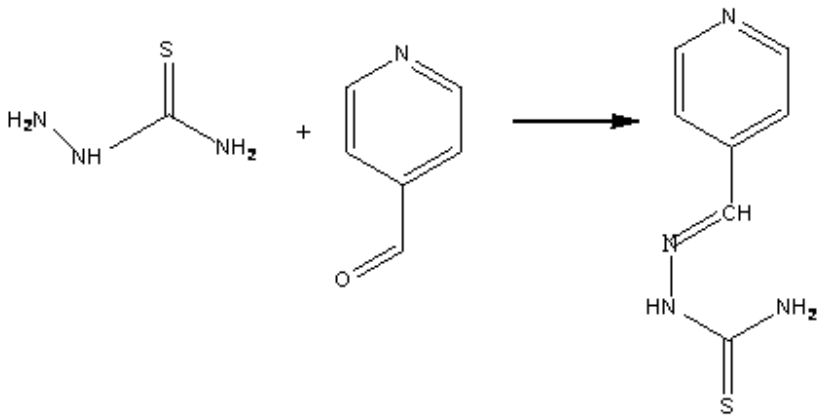
Thiosemicarbazide and 4-pyridinecarboxaldehyde in 1:1 molar ratio in ethanol were refluxed at for 2 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, yellow crystals were obtained (Scheme 1). Then it was washed with ethanol and dried (mp 218-220°C). The same reaction, thiosemicarbazide and 4-pyridinecarboxaldehyde in 1:1 molar ratio in ethanol, was performed using microwave irradiation with the power of 300 W for 50 sec.

Found: C, 46.70; H, 4.31; N, 31.30. Calc. for $C_7H_8N_4S$: C, 46.60; H, 4.43; N, 31.07 (%).

IR (KBr, cm^{-1}): 3425(s), 3267(s), 3159(s), 1598(s), 1108(s), 829(m), 619(m), 522(m).

^{1}HNMR (DMSO-d_6, *ppm*): δH: 7.74(s, 2H), 7.97 (s, 1H), 8.19 (s, 1H), 8. 36 (s, 1H), 8.56 (s, 2H), 11.67(s, 1H)

^{13}CNMR (DMSO-d_6, *ppm*): δC: 122.72, 141.13, 143.07, 151.57, 181.00



Scheme 1. Synthesis of 4-pyridinecarbaldehyde thiosemicarbazone

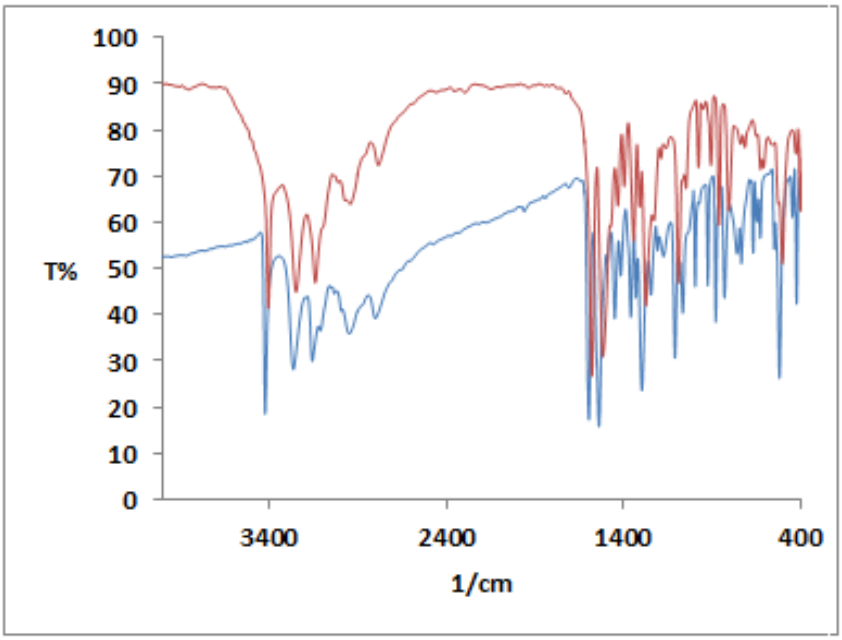


Fig. 1 FT-IR spectra of 4-pyridinecarboxaldehyde thiosemicarbazone

red line: reflux and blue line: Microwave.

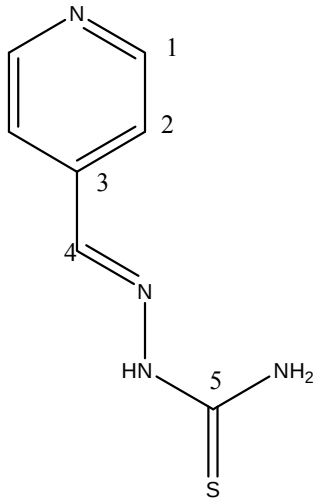
3. Results and Discussion

In the FT-IR spectra, the appeared band at 3425 cm^{-1} at 3425 cm^{-1} can be assigned to the asymmetric (N–H) vibrations of NH_2 group. The other bands at 3267 and 3159 cm^{-1} may be due to the symmetric (N–H) vibrations of the imino and amino groups. The band at 1598 cm^{-1} is related to ν(C=N). The ν(C=S) stretching frequency is observed at 829 cm^{-1} (Fig. 1)[7, 8].

In ^{1}H-NMR spectrum, the singlet resonances are observed at δ = 11.67 and 7.978 ppm, which are assigned to hydrazide NH and aldehyde CH groups, respectively. The observed peaks at δ = 7.748 and 8.562 ppm belong to pyridine protons.

There are five signals in ^{13}C-NMR spectrum. The appeared peak at δ = 180.00(5) is related to C=S.

According to resulting data and elemental analysis, the structure of the title compound can be proposed as shown in scheme 2.



Scheme 2. The structure of 4-pyridinecarbaldehyde thiosemicarbazone

Conclusion

4-pyridinecarboxaldehyde thiosemicarbazone was synthesized by two methods, reflux and microwave. According to resulting data and elemental analysis, we got the same results. This investigation showed that the microwave method has several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures in compare of reflux.

Acknowledgements

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