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[a026]

Solvent-free synthesis of Thiocarbamic acid [(furan-2-yl) ethylidene] hydrazide under ball-milling conditions

Azadeh Tadjarodi [1,*], Saeedeh Eslami Nezhad[1]

1 Chemistry Faculty, Iran University of Science and Technology, Narmak, Tehran, Iran
E-mail: tajarodi@iust.ac.ir

Abstract: Thiocarbamic acid [(furan-2-yl) ethylidene] hydrazide has been synthesized by the reaction between 2-acetylfuran and thiosemicarbazide in 1:1 molar ratio under ball milling conditions. The title compound has been characterized by FT-IR and elemental analysis.

Keywords: Solvent free; Thiosemicarbazide; 2-acetyl furan; Ball milling.

Introduction

Thiosemicarbanzones have received considerable attention because of having numerous biological activities such as anticarcinogenic, antibacterial, anti-HIV, anticancer, fungicides, antiviral, antifungal, antitumour, etc[1].

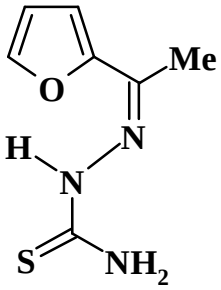
Solvent-free organic synthesis are gathering increasing interest from the viewpoints of green chemistry [2-6]. In any solvent-free reactions, interaction between dissimilar species becomes sensitive since solvation and associated shielding by solvent molecules are absent. Specific interactions between dissimilar solid organic species, among other hydrogen bonding and/or π interaction may, therefore, play a more significant role than those in a solution state, provided that the distance between the solid particles is shorter than the critical distance, through which electrons or protons can move across [7]. Herein, we wish to report solvent-free synthesis of thiocarbamic acid [(furan-2-yl) ethylidene] hydrazide, [TFEH], under ball-milling conditions**.**

Results and Discussion

In the IR spectrum, the absorption bands at 3380 and 3175 cm^{-1}, which may be assigned to –NH_2 and –NH group, respectively. The band ν(C=N) appeared at 1598 cm^{-1}. The stretching

vibration at 1371 cm^{-1} are attributed to ν(CH) vibrations of CH_3 group. Also, ν(C=S) stretching frequency is observed in 1107 cm^{-1}.

Based on the presented FT-IR spectroscopic data and elemental analysis for this compound, structure can be proposed as shown in scheme 1.



Scheme 1. The structure of *[TFEH]*

Experimental Section

A mixture of 2-acetyl furan (0.005 mol, 0.55 g) and thiosemicarbazide (0.005 mol, 0.48 g) and 1g silica gel was mixed and placed in to a stainless-steel jar. The reactants were milled vigorously at a rate of 1200-1500 rpm (20-25 Hz) at room temperature for 5 h. The progress of the reaction was monitored by TLC. After this time, the result dark yellow powder was produced (scheme 1). mp. 107-109 °C.

Elemental analysis found % C 46.15, H 4.75, N 21.73 calculated for $C_7H_9N_3OS$ % C 45.9, H 4.9, N 22.00**.**

IR (KBr, cm^{-1}): 3380(m), 3175(m), 1598(s), 1502(s), 1371(m), 1294(m), 1107(m), 835(m).

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