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Preparation of 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione under microwave ir-radiations

Hiteshi Gahlawat[1], Dr. Ravinder Singh[2]

[1]B.Sc. Chem. Hons. 1st Year, 0411/2022, Miranda House, University of Delhi, Delhi
E.mail: gahlawat.hiteshi@gmail.com
[2]Associate Professor (Chemistry), Pt. N.R.S. Govt. College, Rohtak
E.mail: gahlawat.ravinder@gmail.com

Abstract: The 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione having different biological activities was prepared in high yield using Mont.K-10, KSF under microwave conditions which causes no pollution, reduces the reaction time, provide uniform heating of reaction material and becomes a part of green chemistry by counteracting against the conventional heating methods in Brown chemistry.
Key Words: Triazole, Microwave, Heterocyclic, Biological activity.

Introduction:

The triazoles, exhibit potent antineoplastic agent[1], bactericide and a fungicide[2], insecticidal and acaricidal activities[3]. The triazoles are previously prepared by ordinary heating using Bunsen burner which causes pollution and takes very long time for reaction completion and also have hectic workup process.[4-18] The organic reaction supported by Microwave conditions causes no pollution, reduces the reaction time, causes uniform heating of reaction material.[19-28]
Our research work deals with the synthesis of 5-Mercapto-3-(2-thienyl)-s-triazole[3,4-b][1,3,4]thiadiazole-6(5H)-thione having different biological activities in high yield using microwave conditions which becomes a part of green chemistry due its non-polluting nature. (Figure 1).

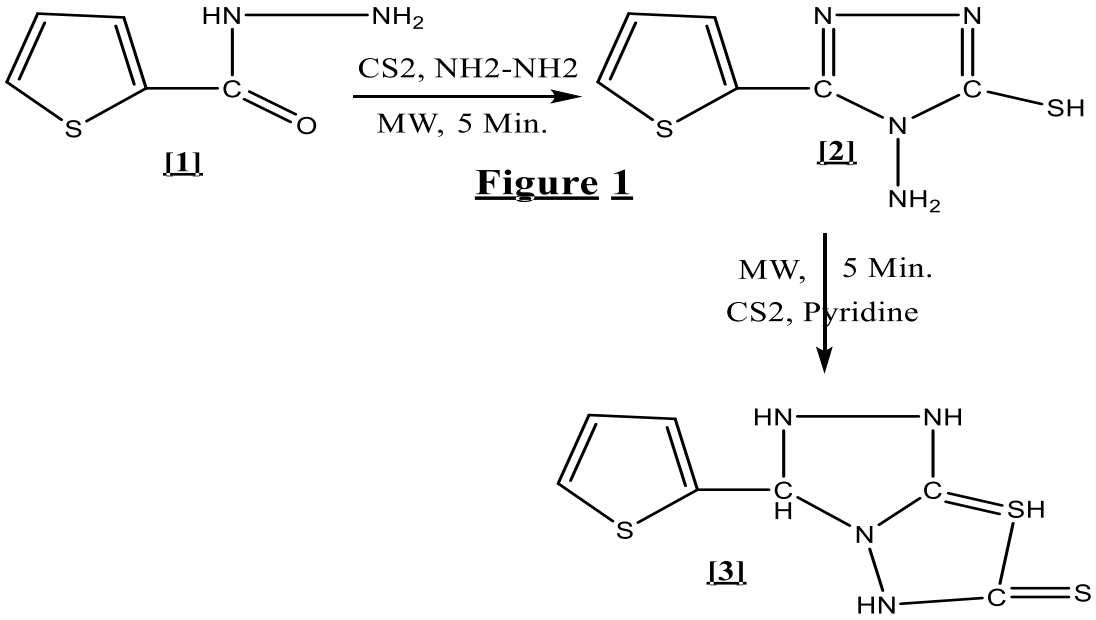

Figure 1

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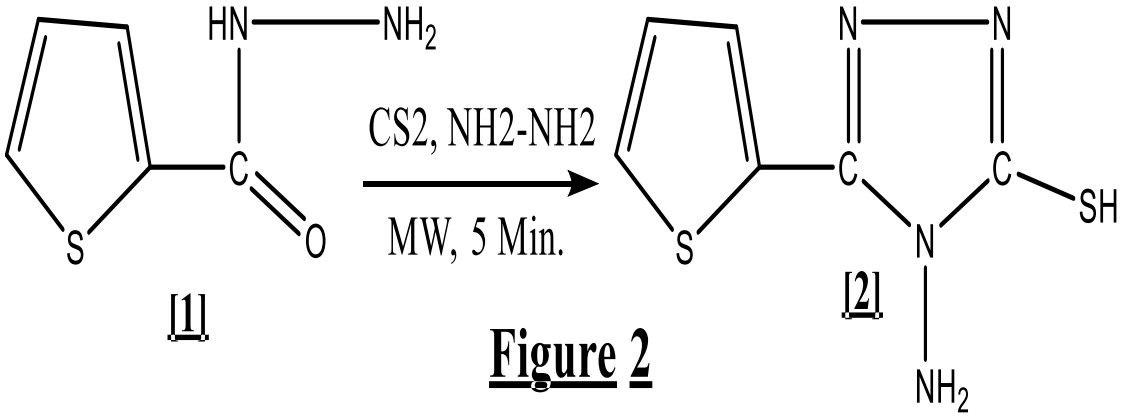
Our research study was started by Traditional Heating methods by reacting 2-thienylhydrazide [1] with CS2, hydrazine under basic condition using KOH give 4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2] in 85% yield after refluxing for 4 hours. The compound [2] solution in pyridine followed by heating in oil bath for 6 hours. The Pyridine was removed under vacuum followed by addition of small of benzene give brown crystals of 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione [3] in 47% yield. All compounds [2], [3], are characterized by their IR, NMR data & Elemental analysis.

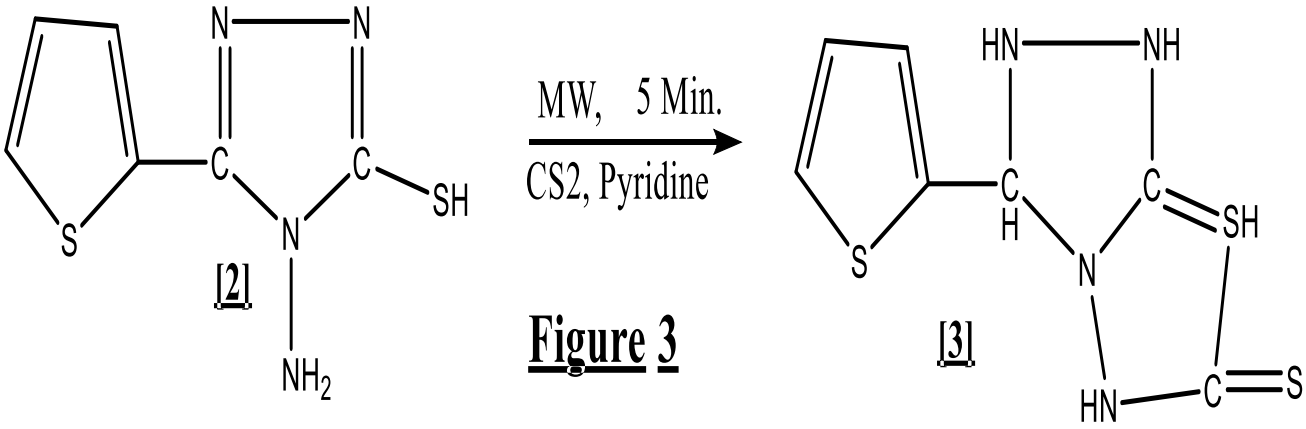
Further, Traditional heating methods are found to be very tedious, time consuming, hectic, and produces product in low yield due to non-uniform heating of reaction mixture. Hence, we elaborated our work by synthesis of 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione [3] by green technique using Microwave irradiations. 2-thienylhydrazide [1]) reacts with hydrazine using Mont. K-10/KSF clay under MW irradiation to give 4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2]. The formation of 4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2] is identified by TLC.



[1] [2]

Figure 2

4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2] simultaneously undergo intramolecular condensation under MW irradiations to give give brown crystals of 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione [3]. The formation of All compounds [2], [3], were analyzed by TLC and they are further characterized by their IR, NMR data & Elemental analysis.



[2] [3]

Figure 3

4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2]

A mixture of 2-thienylhydrazide [1] (1.42g, 0.01 mol), CS2, hydrazine under basic condition

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using KOH, Mont. K-10 clay(0.5g) was irradiated under microwave conditions at optimum condition of 560W for 5-minutes. The resulting mixture was cooled and extracted using water and then crystallized using ethanol-DMF furnishing colorless shining flakes of 4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2]. m.p. 225^{0}C, yield 95%; [$C_6H_6N_4S_2$ Anal. Found N 28.00%, S 32.51%, Requires: N 28.28%, S 32.32%].

3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione [3]

A mixture of 4-amino-5-mercapto-3-(2-thienyl)-s-triazole [2] (0.98g, 0.005 mol) in pyridine (10 ml.), CS2(0.5 ml.) was irradiated under microwave irradiation at 560W for 5-minutes. The Pyridine was removed under vaccum followed by addition of small of benzene give brown crystals of 3-(2-thienyl)-s-triazolo[3,4-b][1,3,4]thiadiazole-6(5H)-thione [3]. m.p. 210^{0}C, yield 87%; IR: 700, 840, 1200, 1310, 1345, 1400, 1470, 1500, 1520, 1620, 3100, 3180 cm^{-1} ; cm^{-1} H NMR(DMSO-d6): 5.84 (1H, bs, N-H), 7.22 [1H, dd (J4,5 = 5.0 Hz; J3,4 = 3.8 Hz)]; 7.79 [1H, d (J3,4 = 3.8 Hz), C-3-H]; 7.99 [d (J4,5 = 5.0 Hz), C-5-H]; [$C_7H_4N_4S_3$ Anal. Found N 23.58%, S 40.27%, Requires: N 23.33%, S 40.0%].

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