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Preparation of 5-Mercapto-3-(2-thienyl)-s-triazole under microwave ir-radiations

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Abstract: The 5-Mercapto-3-(2-thienyl)-s-triazole having different biological activities were 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 conventional 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 having different biological activities in high yield using microwave conditions which becomes a part of green chemistry due to its non-polluting nature. (Figure 1).

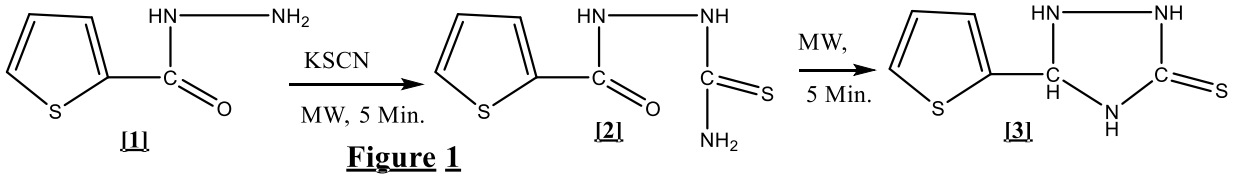

Figure 1

Our research study was started by Traditional Heating methods by reacting 2-thienylhydrazide [1] with potassium thiocyanate under acidic condition to give 2-Thienoylthiosemicarbazide [2] in 60% yield which on refluxing with 8% NaOH for 4 hours followed by cooling and acidification with dil. Acetic acid, washing with water, crystallization with ethanol gives colorless crystals of 5-Mercapto-3-(2-thienyl)-s-triazole [3] in 28.1% 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 5-Mercapto-3-(2-thienyl)-s-triazole [3] by green technique using Microwave irradiations. 2-thienylhydrazide [1]) reacts with potassium thiocyanate using

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Mont. K-10/KSF clay under MW irradiation to give 2-Thienoylthiosemicarbazide [2]. The formation of 2-Thienoylthiosemicarbazide [2] is identified by TLC.

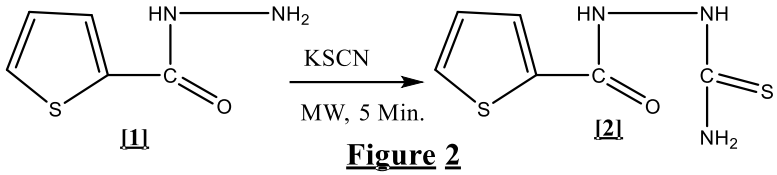

Figure 2

2-Thienoylthiosemicarbazide [2] simultaneously undergo intramolecular condensation under MW irradiations to give colorless crystals of 5-Mercapto-3-(2-thienyl)-s-triazole [3]. The formation of All compounds [2], [3] were analyzed by TLC and they are further characterized by their IR, NMR data & Elemental analysis.

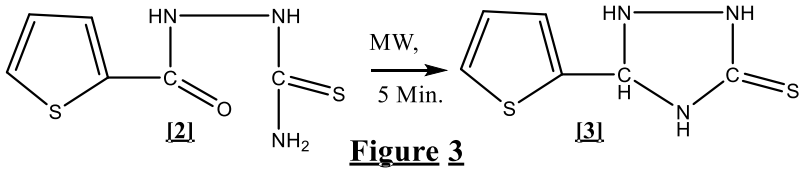

Figure 3

Synthesis of 2-Thienoylthiosemicarbazide [2]

A mixture of 2-thienylhydrazide [1] (1.42g, 0.01 mol), potassium thiocyanate (0.97g, 0.01mol), 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 2-Thienoylthiosemicarbazide [2]. m.p. 215^0C, yield 90%; IR: 700, 840, 1120, 1240, 1390, 1420, 1520, 1590, 1600, 1670, 3220, 3320 cm^{-1} [$C_6H_7N_3S_2O$ Anal. Found N 21.18%, S 31.61%, Requires: N 20.89%, S 31.84%].

Synthesis of 5-Mercapto-3-(2-thienyl)-s-triazole [3]

A mixture of 2-Thienoylthiosemicarbazide [2] (2.01g, 0.01 mol) in 8% NaOH solution (300 ml) was irradiated under microwave irradiation at 560W for 5-minutes. The resulting mixture was cooled and then small amount of dil. acetic acid is added. The compound was further filtered, and then crystallized using ethanol to give colorless crystal 5-Mercapto-3-(2-thienyl)-s-triazole [3]. m.p. 240^0C, yield 97%; IR: 680, 835, 1240, 1380, 1400, 1520, 1590, 1620, 2590, 3040, 3100 cm^{-1} [$C_6H_5N_3S_2$ Anal. Found N 22.64%, S 35.25%, Requires: N 22.95%, S 34.97%].

References:

[1] Kano, S; Noguchi T, Japan Pat., 1971, 836, 71 37; Chem. Abstr., 1972, 76, 25295g.
[2] Hoffmann H; Hammann I, Ger. Offen., 1972, 32, 2, 173 (1972); Chem. Abstr., 1972, 76, 72525s.
[3] Deshmukh A.A., Mody M.K., Ramalingam T. and Sattur P.B., Indian J. Chem, 1984, 23B, 793.
[4] Rogness D. C, Larock, R.C. Tetrahedron Lett, 2009, 50, 4003.
[5] Lin, Z; Larock, R.C., J. Org. Chem. , 2006, 71, 3198.

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[6] Peddibhotla, S., Curr. Bioact. Compd., 2009, 5, 20.
[7] Saburo K., Zasshi Y, 1972, 92(8), 935; Chem. Abstr., 1972, 77, 126492V.
[8] Potts K.T. and Husain S., J. Org. Chem., 1971, 36, 10.
[9] Mohan J, Indian J. Chem., 1982, 21B, 243.
[10] Mohan J and Anjaneyulu G.S.R., Polish J. Chem., 1987, 61, 547.
[11] Hoggarth E., J. Chem. Soc., 1952, 4811.
[12] George T., Thilramani R. and Dabholkar D.A., Indian J. Chem., 1969, 7, 959.
[13] Reid J.R. and Heindel N.D., J. Heterocycl. Chem., 1976, 13, 925.
[14] Hantzsch and Weber H.J., Berdt Chem. Ges., 1987, 20, 3118.
[15] Dodson R.M., King L.C., J. Am. Chem. Soc., 1945, 87, 2242.
[16] King L.C. and Hlavacek R.J., J. Am. Chem. Soc., 1950, 72, 3722.
[17] Pattanayak B.K., Rout D.N. and Mahapatra G.N., Indian J. Chem., 1978, 16B, 1030.
[18] Chadha V.K. and Pujari H.K., Can. J. Chem., 1979, 47, 2843.
[19] Singh R, Chemical Science Review and Letters, 4, 15(2015), 835-837.
[20] Singh R, Rajeev Kumar, Chemical Science Review and Letters, 4, 16(2015), 937-940.
[21] Dewan S K, Singh R, Arkivoc, 2006, Vol (ii), 41-44.
[22] Dewan S K; Singh R; Anil Kumar, Synthetic Communications; 2004, 34(11), 2025-2029.
[23] Dewan S K, Singh R Synthetic Communications; 2003, 33(17), 3085-3088.
[24] Dewan S K, Singh R, Synthetic Communications; 2003, 33(17), 3081-3084.
[25] Dewan S K, Singh R J. Indian council Chemists; 2003, 20(1), 1-3.
[26] Dewan S K, Singh R, Indian J. Heterocyclic Chemistry; 2003, 12(1), 287-288.
[27] Dewan S K, Singh R, Indian J. Heterocyclic Chemistry; 2002, 12(1), 173-174.
[28]Dewan S K, Singh R, Kanwar M, oriental J. of chemistry, 2002, 18(3), 555-558.