Research Article

A multistep preparation of 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole under microwave ir-radiations

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Abstract

The 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole having different biological activities are prepared in high yield using microwave conditions which becomes a part of green chemistry due its non pollution nature

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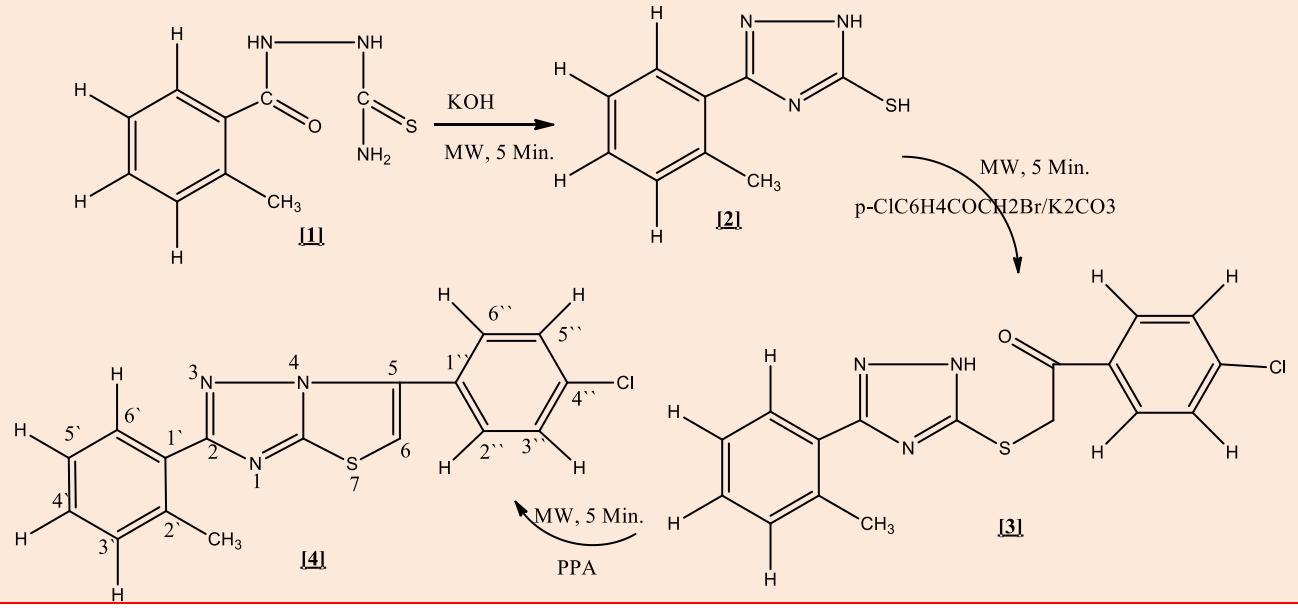

Introduction

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 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole having different biological activities in high yield using microwave conditions which becomes a part of green chemistry due its non pollution nature (**Figure 1**).

Our research study started by reacting 4-o-methylbenzoylthiosemicarbazide[1]) with 8% potassium hydroxide solution using microwave conditions at optimum condition of 560W for 5-minutes leads to formation of 5-Mercapto-3-o-tolyl-s-triazole[2] (**Figure 2**). The 4-o-methylbenzoylthiosemicarbazide[1] was in turn obtained by reacting o-methylbenzoylhydrazide with potassium thiocynate, conc. HCl and water using microwave conditions at optimum condition of 560W for 5-minutes.

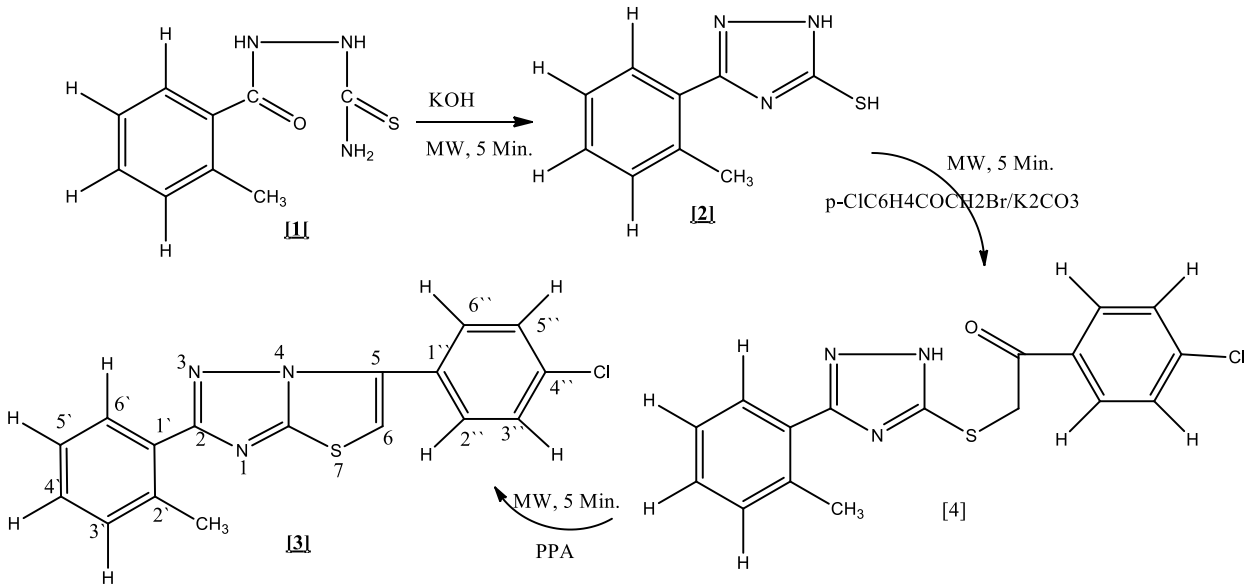

Figure 1

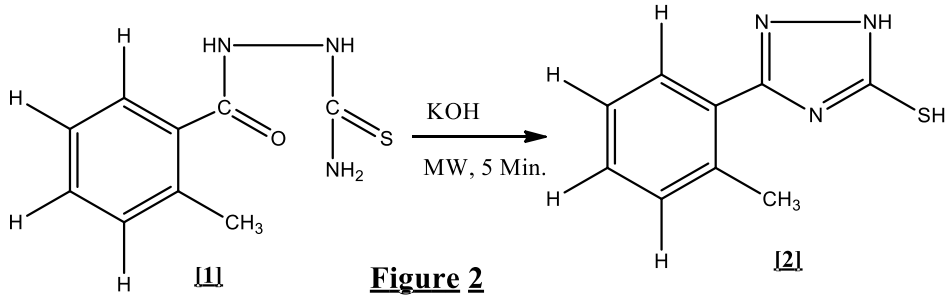

Figure 2

Further, 5-Mercapto-3-o-tolyl-s-triazole[2] reacts with p-chlorophenacy bromide in dry methanol using microwave conditions at optimum condition of 560W for 5-minutes to afforded white compound 5-p-Chlorobenzoylmethylmercapto-3-o-tolyl-s-triazole[3] (**Figure3**).

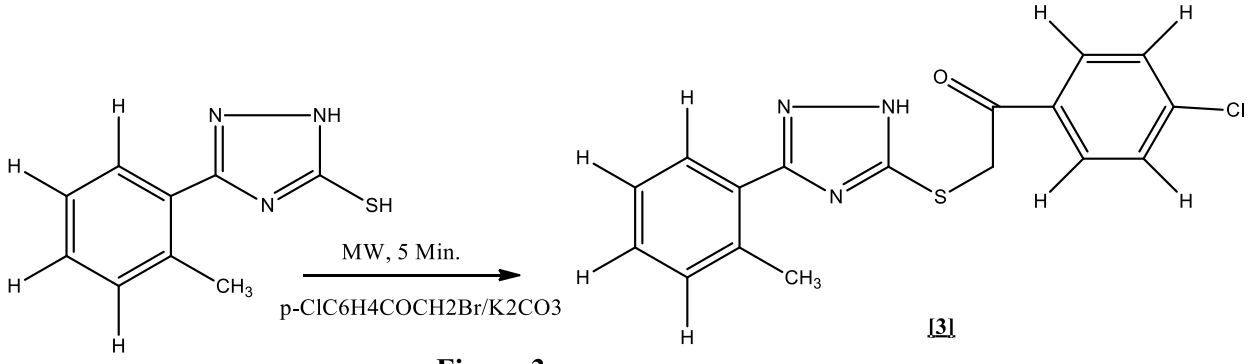

Figure 3

We further explored our work by reacting 5-p-Chlorobenzoylmethylmercapto-3-o-tolyl-s-triazole[3] (R_1 = p-$C1C_6H_7$, 1g) with PPA/P_2O_5 using microwave conditions at optimum condition of 560W for 5-minutes which leads to the formation of 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole[4] (**Figure 4**).

Synthesis of 4-o-methylbenzoylthiosemicarbazide[1]

A mixture of o-methylbenzoylhydrazide (1.50g, 0.1 mol), potassium thiocynate (19.4g, 0.2 mol), conc. HCl (8 ml) and water (150 ml) was irradiated using microwave conditions at optimum condition of 560W for 5-minutes. The resulting mixture was cooled and filtered to get resultant compound. The compound was further filtered, washed

using water and then crystallized using ethanol-DMF furnishing colourless shining flakes of 4-o-methylbenzoylthiosemicarbazide[1]. m.p. 185^{0}C, yield 89% [Found : S, 15.03.$C_9H_{11}N_3OS$ requires S, 15.31%].

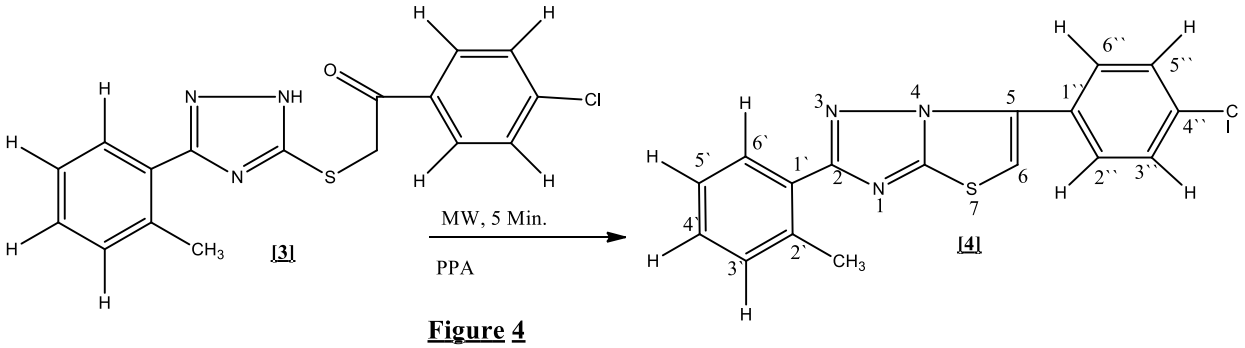

Figure 4

Synthesis of 5-Mercapto-3-o-tolyl-s-triazole[2]

A mixture of 4-o-methylbenzoylthiosemicarbazide(I) (20.9 g, 0.1 mol) in 8% potassium hydroxide 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 white solid 5-Mercapto-3-o-tolyl-s-triazole[2]. m.p. 255^{0}C (Lit^{254} m.p. 255^{0}C), yield 77% [Found :S, 16.92.$C_9H_9N_3S$ requires S, 16.75%].

Synthesis of 5-p-Chlorobenzoylmethylmercapto-3-o-tolyl-s-triazole[3]

A mixture of 5-Mercapto-3-o-tolyl-s-triazole[2] (1.91g, 0.01 mol) and p-chlorophenacy bromide (2.34g, 0.01 mol) in dry methanol (100 ml) was treated using microwave conditions under optimum condition at 560W for 5-minutes. The resultant mixture was cooled and then small amount of aq. K_2CO_3 is added for neutralization. The resultant compound was filtered, and crystallized using methanol to give a white solid 5-p-Chlorobenzoylmethylmercapto-3-o-tolyl-s-triazole[3]. m.p. 220^{0}C, yield 65%. IR : 755, 830 (1, 2 & 1, 4-dsubstituted benzene rings) 1485 (C-N stretching), 1590 (C=N), 1690 (C=0), 3400 (NH) [Found: N, 12.33; S, 9.11.$C_{17}H_{13}N_3OSCI$ requires N, 12.26; S, 9.34%].

Synthesis of 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole[4]

A mixture of 5-p-Chlorobenzoylmethylmercapto-3-o-tolyl-s-triazole[3] (R_1 = p-C1C_6H_7, 1g), P_2O_5 (4.0g) and H_3PO_4 (3 ml) was treated using microwave conditions under optimum condition at 560W for 5-minutes. The resultant mixture was cooled and then small amount of aq. K_2CO_3 is added for neutralization. The resultant compound was filtered, and crystallized using ethanol to give colourless needles of 2-o-Tolyl-5-p-chlorophenyl thiazolo [3, 2-b]-s-triazole[4]. Yield: 72%, m.p. > 250^{0}C. IR : 740, 830 (1, 2 & 1, 4 – disubstituted benzene), 1500 (C-N), 1580, 1600 (C = C & C =N), 3040 (C-H, Ar); H^1-NMR ($CDC1_3$) : δ 2.30 (3H, s, CH_3), 6.70 (1H, s, C_6-H) 6.60 – 7.40 (8H, m, Ar-H). [Found :C, 62.78; H, 3.46; S. 9.68; N, 13.14.$C_{17}H_{12}N_3SC1$ requires C, 62.86; H, 3.39; S, 9.86; N, 12.94%].

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