IJFANS INTERNATIONAL JOURNAL OF FOOD AND NUTRITIONAL SCIENCES

ISSN PRINT 2319- 1775 Online 2320 -7876

Research paper
 UGC CARE Listed (Group -I) Journal Volume 11, Iss 10, Oct 2022

Preparation of 5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole from 5-Mercapto-3-(2-thienyl)-s-triazole [3] under microwave ir-radiations

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Abstract: The 5-p-Bromophenyl thiazolo[3,2-b]-2-(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 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-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole having different biological activities in high yield using microwave conditions which becomes a part of green chemistry due its non-polluting nature. (Figure 1).

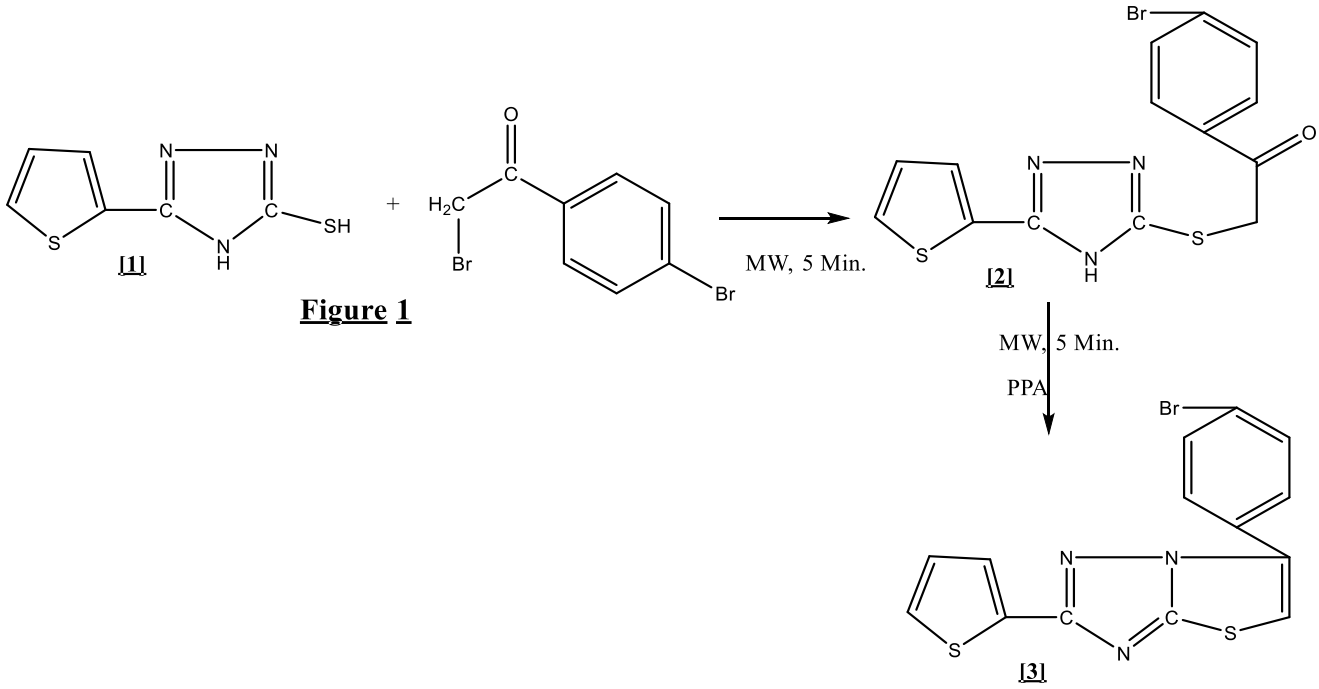

Figure 1

Our research study started with traditional heating by refluxing 5-Mercapto-3-(2-thienyl)-s-

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triazole [1] on steam bath with p-bromophenylacyl bromide in anhyd. ethanol followed by cooling, washing with water, crystallization with ethanol gives brown crystals of 5-p-Bromobenzoylmethylmercapto-3-(2-thienyl)-s-triazole hydrobromide [2]. The compound [2] on heating with P_2O_5 and H_3PO_4 followed by cooling, addition of water, neutralization with aq. K_2CO_3 solution, crystallization with ethanol gives orange granules of 5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole [3]. All compounds [2], [3] were characterized by their IR & Elemental analysis.

Our study was further elaborated by carrying out synthesis of 5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole [3] by green technique using Microwave irradiations. 5-Mercapto-3-(2-thienyl)-s-triazole [1] reacts with p-bromophenylacyl bromide under MW radiations to give brown crystals of 5-p-Bromobenzoylmethylmercapto-3-(2-thienyl)-s-triazole hydrobromide [2].

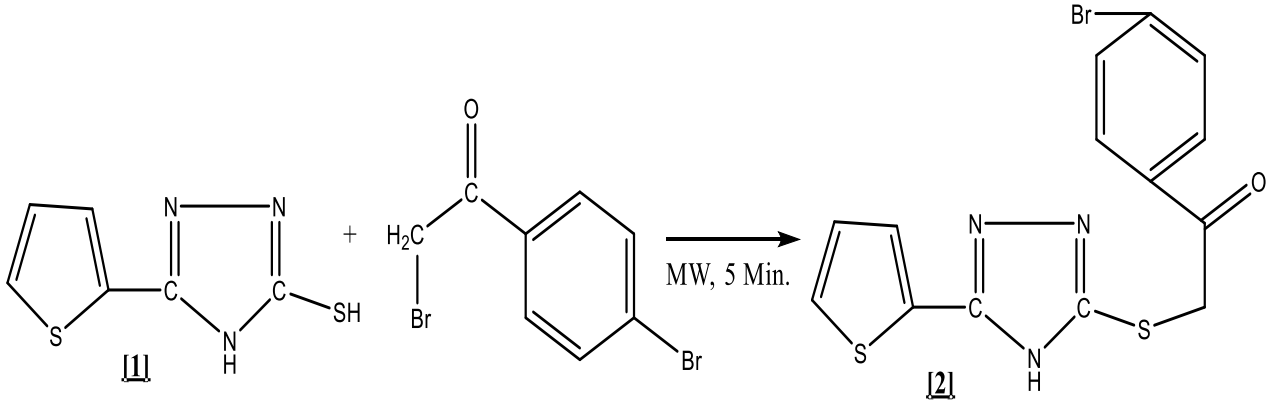
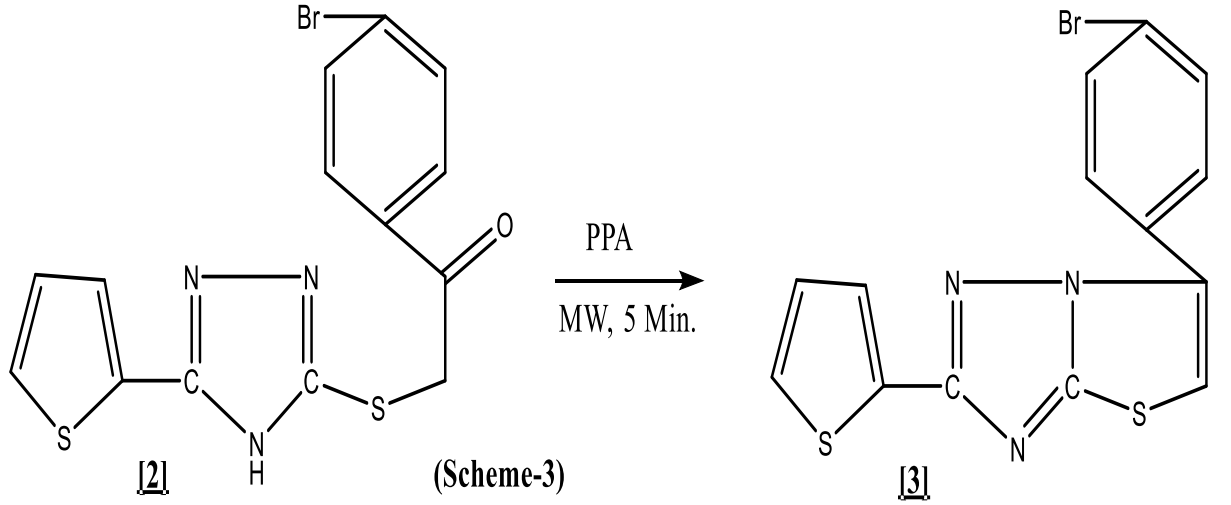


Figure 2

The compound [2] simultaneously undergo intramolecular condensation under MW irradiations to give orange granules of 5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole [3]. The formation of All compounds [2], [3], was analysed by TLC and they are further characterized by their IR & Elemental analysis.



(Scheme-3)

Synthesis of 5-p-bromobenzoylmethylmercapto-3-(-2-thienyl)-s-triazole hydrobromide[2]

A mixture of 5-Mercapto-3-(2-thienyl)-s-triazole (1.91g, 0.005 mol), p-bromophenacyl bromide

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(1.39g, 0.005 mol), was irradiated using microwave conditions at optimum condition of 560W for 5-minutes. The resulting mixture was cooled, washed using water and then crystallized using ethanol-DMF furnishing brown crystals of Synthesis of 5-p-bromobenzoylmethylmercapto-3-(-2-thienyl)-s-triazole hydrobromide [2]. m.p. 165^0C, yield 92%; IR: 700, 830, 840, 1240, 1380, 1420, 1525, 1695, 3100, 3420 cm^{-1} [$C_{14}H_{11}Br_2N_3S_2O$ Anal. Found N 9.45%, S 13.62%, Requires: N 9.11%, S 13.88%].

5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole [3]

A mixture of 5-p-bromobenzoylmethylmercapto-3-(-2-thienyl)-s-triazole hydrobromide (2) (1g, 0.1 mol), P2O5 (4g) and H3PO4 (3ml.) was irradiated under microwave irradiation at 560W for 5-minutes. The resulting mixture was cooled, washed using water and neutralized with aq. K2CO3 solution and then crystallized using ethanol to give orange solid 5-p-Bromophenyl thiazolo[3,2-b]-2-(2-thienyl)-s-triazole [3]. m.p. 155^0C, yield 85%; IR: 690, 830, 835, 1240, 1370, 1410, 1515, 1550, 1600, 1620, 30340, 3080 cm^{-1} [$C_{14}H_8BrN_3S_2$ Anal. Found N 11.47%, S 17.82%, Requires: N 11.60%, S 17.60%].

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