

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

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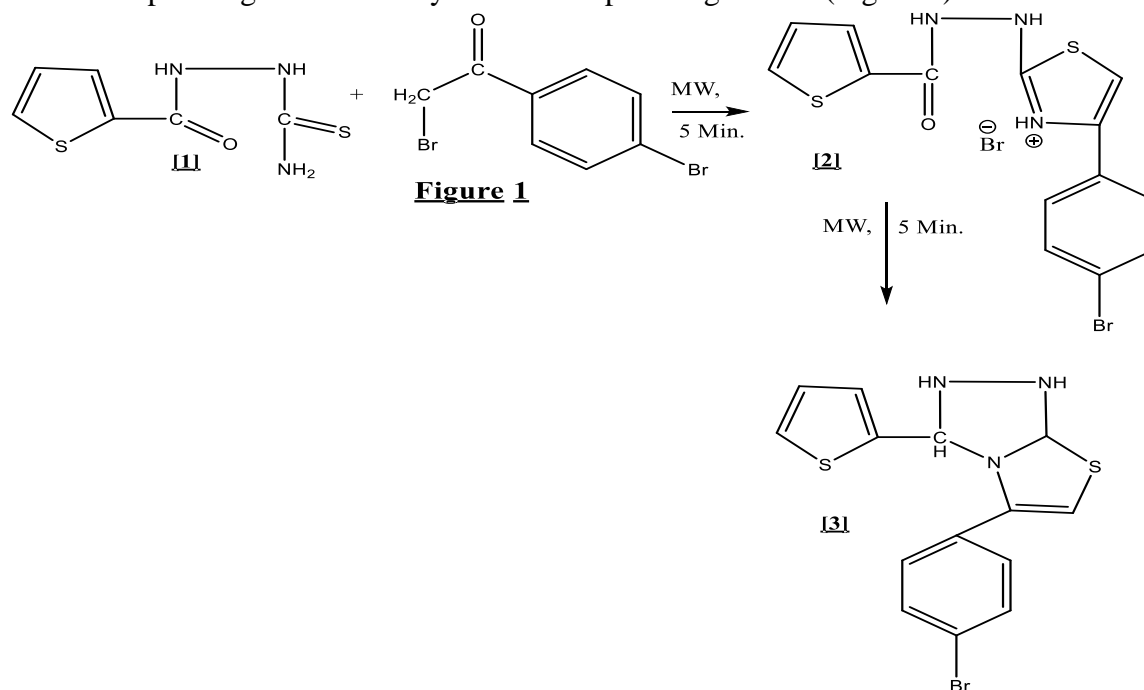
**Abstract:** The 5-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-s-triazole having different biological activities are prepared in high yield using Mont.K-10, KSF under microwave conditions which causes no pollution, reduces the reaction time, provide uniform heating of reaction material and becomes a part of green chemistry by counteracting against the conventional heating methods in Brown chemistry.

**Key Words:** Triazole, Microwave, Heterocyclic, Biological activity.

### Introduction:

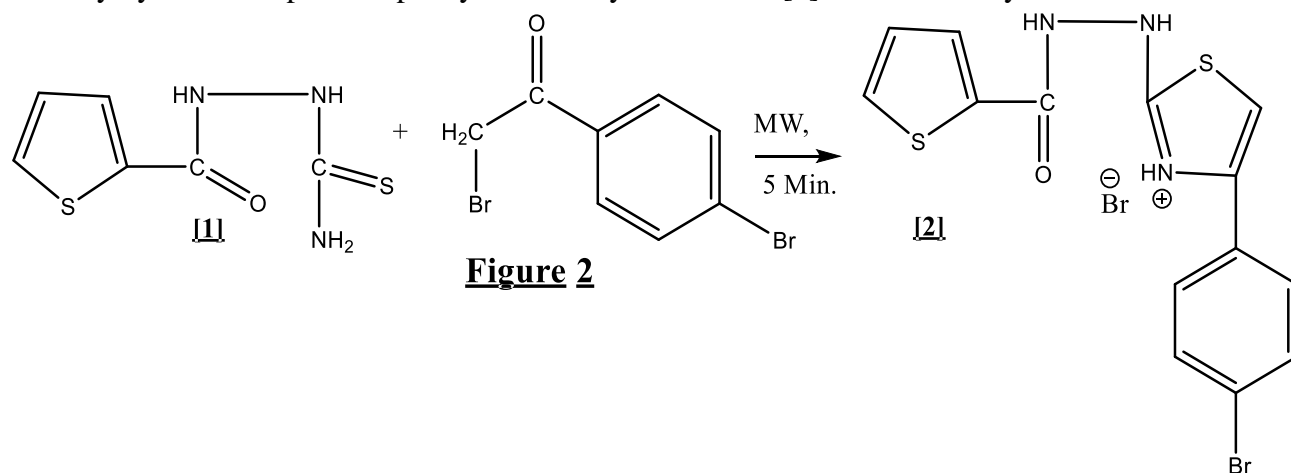
The triazoles, exhibit potent antineoplastic agent<sup>1</sup>, bactericide and a fungicide<sup>2</sup>, insecticidal and acaricidal activities<sup>3</sup>. The triazoles are previously prepared by ordinary heating using Bunsen burner which causes pollution and takes very long time for reaction completion and also have hectic workup process.<sup>4-18</sup> The organic reaction supported by Microwave conditions causes no pollution, reduces the reaction time, causes uniform heating of reaction material.<sup>19-28</sup>

Our research work deals with the synthesis of 5-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-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).

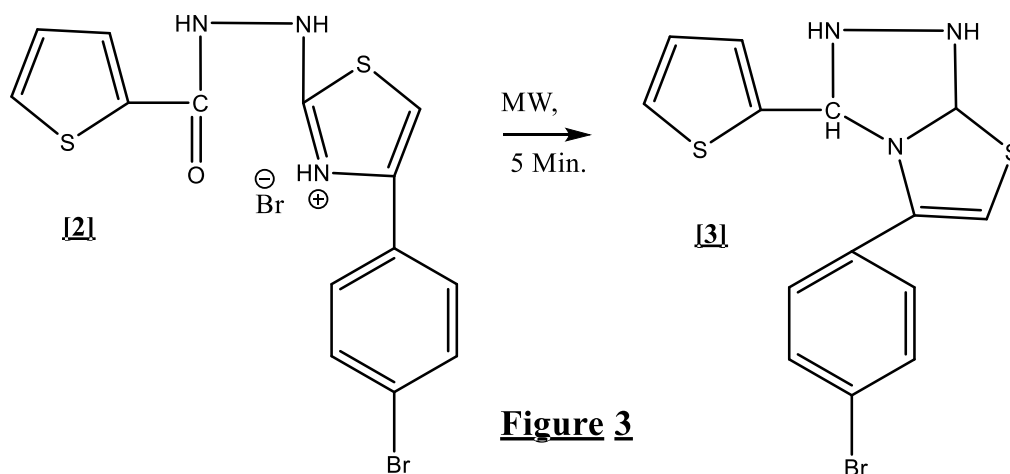


Our research study was started by Traditional Heating methods by refluxing 2-Thienoylthiosemicarbazide [1] with p-bromophenacyl bromide [ $C_6H_5(Br)COCH_2Br$ ] in anhydrous ethanol on steam bath for 5 hours followed by cooling and crystallization with ethanol gives light brown crystals of 2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2]. The compound [2] on heating with  $POCl_3$  on oil bath for 3 hours followed by addition of water, neutralization with aq.  $K_2CO_3$  and crystallization with ethanol gives red colored crystals of 5-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-s-triazole [3]. 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-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-s-triazole [3] by green technique using Microwave irradiations. 2-Thienoylthiosemicarbazide [1] with p-bromophenacyl bromide [ $C_6H_5(Br)COCH_2Br$ ] using Mont. K-10/KSF clay under MW irradiation to give 2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2]. The formation of 2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2] is identified by TLC.



2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2] simultaneously undergo intramolecular condensation under MW irradiations to give red colored crystals of 5-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-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.



**2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2]**

A mixture of 2-Thienoylthiosemicarbazide [1] (1.00g, 0.005 mol), with p-bromophenacyl bromide (1.39g, 0.005 mol), 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-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2]. m.p. 240°C, yield 89%; IR: 680, 830, 840, 1220, 1360, 1400, 1520, 1550, 1600, 1620, 1695, 3040, 3120, 3440 cm<sup>-1</sup> [C<sub>14</sub>H<sub>11</sub>BrN<sub>3</sub>S<sub>2</sub>O Anal. Found N 12.37%, S 18.02%, Requires: N 12.03%, S 18.33%].

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

A mixture of 2-Thienoylhydrazino-4-p-bromophenylthiazole hydrobromide [2] (1.00g) was irradiated under microwave irradiation at 560W for 5-minutes. The resulting mixture was cooled and then crystallized using ethanol to give red colored crystal of 5-p-Bromophenyl-3-(2-thienyl)thiazolo[2,3-c]-s-triazole [3]. m.p. >250°C, yield 95%; IR: 700, 840, 1250, 1380, 1410, 1520, 1610, 1620, 3040, 3080 cm<sup>-1</sup> [C<sub>14</sub>H<sub>8</sub>BrN<sub>3</sub>S<sub>2</sub> Anal. Found N 11.46%, S 17.85%, Requires: N 11.60%, S 17.67%].

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