

A Rapid Synthesis of the 3-Methyl-5-(Methylsulfanyl)-1-Phenyl-1*H*-Pyrazolo[4,3-*e*]-[1,2,4]Triazine from Phenylhydrazone of 5-Acetyl-3-(Methylsulfanyl)-1,2,4-Triazine under Microwave Irradiation

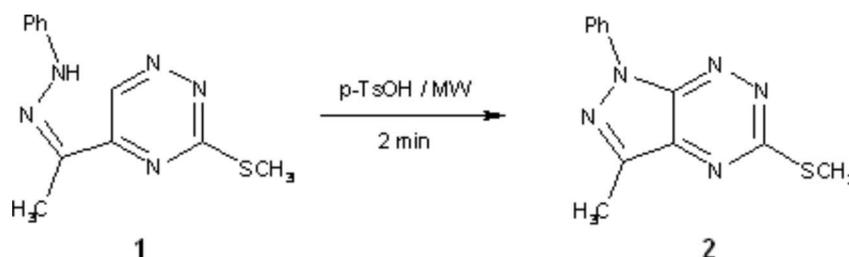
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The combination of microwave irradiation and solvent-free conditions leads to enhanced reaction rates, higher yields of pure products, easier work-up and several advantages of the eco-friendly approach in the framework of green-chemistry [1-2]. Here we report a new route for the preparation of 1*H*-pyrazolo[4,3-*e*][1,2,4]triazine derivative **2** starting from phenylhydrazone of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine (**1**) [3] and *p*-toluenesulfonic acid (*p*-TsOH) under microwave irradiation.



The mixture of the phenylhydrazone **1** (259 mg, 1 mmol) and *p*-toluenesulfonic acid (344 mg, 2 mmol) was heated in quartz container under microwave irradiation [4]. After 2 min the product was eluted with CHCl_3 . The crude product **2** was purified by column chromatography on silica gel (Merck, 230-400 mesh) using chloroform as eluent. Yield of pure 3-methyl-5-(methylsulfanyl)-1-phenyl-1*H*-pyrazolo[4,3-*e*][1,2,4]triazine (**2**) – 149 mg (0.58 mmol, 58%).

Melting Point: 105 °C.

$^1\text{H-NMR}$ (200 MHz, CDCl_3): δ = 2.73 (s, 3H); 2.77 (s, 3H); 7.29-7.40 (m, 1H); 7.50-7.61 (m, 2H); 8.31-8.38 (m, 2H).

IR (KBr, cm^{-1}): 2920, 1590, 1500, 1390, 760.

MS (EI, 70eV; m/z , %): 257 (43) [M^+]; 232 (3); 216 (22); 93 (41); 77 (100).

Elemental Analysis: Calculated for $\text{C}_{12}\text{H}_{11}\text{N}_5\text{S}$: C, 56.03%; H, 4.28%; N, 27.23%. Found: C, 55.67%; H, 4.13%; N, 27.05%.

References:

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- The Prolabo microwave synthesizer SYNTHEWAVE 402 was used with feedback temperature control.

Sample Availability: Available from MDPI.

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