

Research Article

Synthesis of 6-Methoxyisatin-3-thiosemicarbanzone in presence of Mont. K-10, Mont. KSF catalysts under Microwave ir-radiations

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Abstract

The 6-Methoxyisatin-3-thiosemicarbanzone having antihistaminic, antithyroid, antitubercular, antifungal & antibacterial activities synthesized in high yield in presence of heterogeneous Mont. K-10, Mont. KSF catalysts in shorter reaction time under microwave irradiations as compared to under simple conventional heating.

Keywords: Microwave, Aryl, heterocyclic, Conventional heating, thiosemicarbanzone

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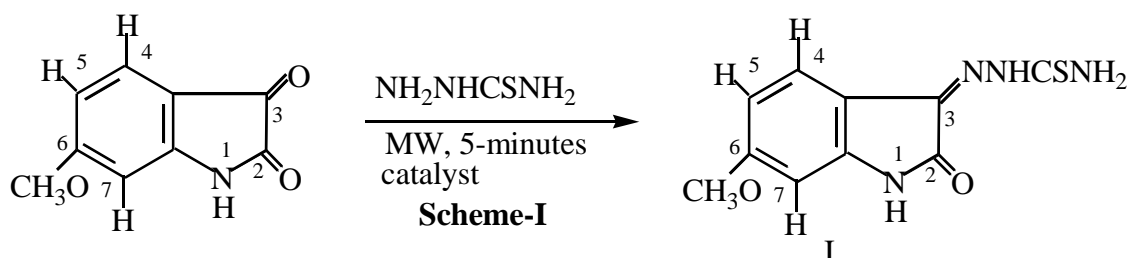
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Introduction

The nitrogen heterocycles exhibit antihistaminic, antithyroid, antitubercular, antifungal, antibacterial, [1-3] anthelmintics activities, antidepressants, platelet aggregation inhibitors, antineoplastic, vulcanization accelerators, photographic sensitizers [4-11] are already been synthesized by different method but they requires longer reaction time and tedious workup [12-21]. Microwave assisted reactions are gaining much more importance in synthetic organic chemistry due to dramatic reduction in time from days to hours and hours to minutes or seconds [22-23].

The present work reports the synthesis of 6-Methoxyisatin-3-thiosemicarbanzone(I) under conventional heating and in presence of heterogeneous catalysts Mont. K-10 and Mont. KSF under microwave irradiation (**Scheme 1**).



A mixture of 6-methoxyisatin in Anhyd. ethanol and thiosemicarbazide in a mixture of water and glacial acetic acid on conventional heating for 1 hour give 6-Methoxyisatin-3-thiosemicarbanzone(I) in 90% yield. It was found that the a mixture of 6-methoxyisatin in Anhyd. ethanol and thiosemicarbazide in a mixture of water and glacial acetic acid under microwave irradiation at 560W for 5-minutes give 6-Methoxyisatin-3-thiosemicarbanzone(I) in 95% yield.

Further it was found that the 6-methoxyisatin on reaction with thiosemicarbazide under microwave irradiation at 560W for 5-minutes in presence of heterogeneous catalysts Mont. K-10 and Mont. KSF give 6-Methoxyisatin-3-thiosemicarbanzone(I) in 97%, 98% yield respectively. It is concluded that yield of product increases under microwave heating in presence of catalyst (**Table 1**).

Table 1 Synthesis of 6-Methoxyisatin-3-thiosemicarbanzone under different conditions

Sr. No.	Condition	%age yield of 6-Methoxyisatin-3-thiosemicarbanzone	Time (in minutes)	Melting point (°C)
1	Without catalyst under conventional heating	90%	5	>250°C
2	Without catalyst under Microwave heating	95%	5	>250°C
3	With Mont.K-10 under MW heating	97%	5	>250°C
4	With Mont. KSF under MW heating	98%	5	>250°C

Experimental

All the melting points reported are uncorrected. Infrared spectra (ν_{\max} in cm^{-1}) were recorded in nujol mull or KBr on a Perkin-Elmer 842/Beckman IR-20/Hitachi 215 spectrometers. The proton magnetic resonance spectra were recorded on a VXR-200 MHz or R-32 Perkin-Elmer 90 MHz spectrometer in CDCl_3 or DMSO-d_6 using tetramethylsilane (TMS) as internal reference standard. The chemical shifts are expressed in δ (ppm) units downfield from TMS. Mass spectra were scanned on a Jeol JMX-DX-300 spectrometer operating at 70 eV. Carbon, hydrogen and nitrogen analyses were carried out on a Yanaco MT-3 (JAPAN) instrument. Thin layer chromatography (TLC) were performed on silica-gel plates using acetone-benzene (1:3 or 1:2) as solvent system and iodine chamber as visualizing agent.

Typical procedure for the synthesis of 6-Methoxyisatin-3-thiosemicarbanzone(I) in presence of solvent under conventional heating

A mixture of 6-methoxyisatin (0.18g, 0.001 mol) in Anhyd. ethanol (2ml) and thiosemicarbazide (0.1g, 0.0011 mol) in a mixture of water (2 ml) and glacial acetic acid (0.5 ml) was heated for 1-hour. A yellow coloured solid formed during irradiation. The solid was filtered, washed well with water and crystallized from ethanol-DMF furnishing yellow crystals. Yield 0.234g (90%), m.p. 265°C. [Found: N, 22.68, S, 12.62. $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}_2\text{S}$ requires N, 22.40; S, 12.80%]; IR: 825, 860 (1, 2, 4-trisubstituted benzene ring), 1115 (C=S), 1125 & 1370 (C-O-C stretching), 1620 (C=N), 1700 (C=O), 3200, 3280, 3400 (NH, NH_2).

Typical procedure for the synthesis of 6-Methoxyisatin-3-thiosemicarbanzone(I) in presence of solvent under microwave heating

A mixture of 6-methoxyisatin (0.18g, 0.001 mol) in Anhyd. ethanol (2ml) and thiosemicarbazide (0.1g, 0.0011 mol) in a mixture of water (2 ml) and glacial acetic acid (0.5 ml) was irradiated under microwave irradiation at 560W for 5-minutes. A yellow coloured solid formed during irradiation. The solid was filtered, washed well with water and crystallized from ethanol-DMF furnishing yellow crystals. Yield 0.247g (95%), m.p. 265°C. [Found: N, 22.68, S, 12.62. $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}_2\text{S}$ requires N, 22.40; S, 12.80%]; IR: 825, 860 (1, 2, 4-trisubstituted benzene ring), 1115 (C=S), 1125 & 1370 (C-O-C stretching), 1620 (C=N), 1700 (C=O), 3200, 3280, 3400 (NH, NH_2).

Typical procedure for the synthesis of 6-Methoxyisatin-3-thiosemicarbanzone(I) in dry media using Mont. K-10, Mont. KSF catalyst under microwave heating

A mixture of 6-methoxyisatin (0.18g, 0.001 mol) and thiosemicarbazide (0.1g, 0.0011 mol) in presence of heterogeneous catalyst Mont. K-10, Mont. KSF was irradiated under microwave irradiation at 560W for 5-minutes. A yellow coloured solid formed during irradiation. The product formed is extracted using ethanol-DMF. Yield 0.252g (97% with Mont. K-10); 0.255g (98% with Mont. KSF), m.p. 265°C. [Found : N, 22.68, S, 12.62. $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}_2\text{S}$ requires N, 22.40; S, 12.80%]; IR: 825, 860 (1, 2, 4-trisubstituted benzene ring), 1115 (C=S), 1125 & 1370 (C-O-C stretching), 1620 (C=N), 1700 (C=O), 3200, 3280, 3400 (NH, NH_2).

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