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Synthesis of 4-methoxy-2-thiomorpholin-4-ylmethyl-1-phenol

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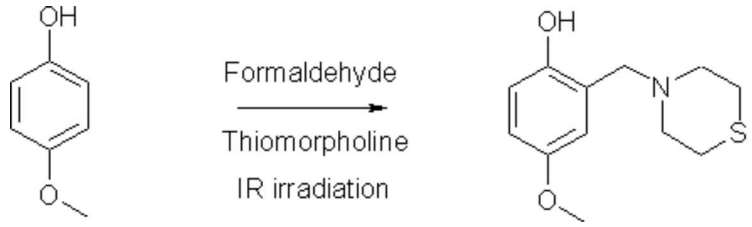

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Infrared light activation as a non-conventional energy source has become an important method that can be used to carry out a wide range of reactions with short reaction times and high yields. Indeed, infrared light heating rapidly increases temperature in absence of the solvent and leads to a uniform energy transfer to the reactants of the chemical reaction. This method does not involve toxic materials, resulting in an economic process, which has clear advantages as an environmentally friendly, solvent-free alternative in organic synthesis. In our experience and, according to the reports in the literature, when the reaction mixtures were refluxed using ethanol as solvent in the absence of infrared light irradiation, the reaction times were in the range of 1–100 hours and the yields were lower.

4-methoxy-2-thiomorpholin-4-ylmethyl-1-phenol (**2**) was prepared from 4-methoxyphenol (**1**), 1 eq. of thiomorpholine and 2 eq. of formaldehyde. They were mixed in a round flask fitted with a condenser. The mixture was irradiated with infrared light using a medicinal infrared lamp (250 Watts) and the reaction was monitored by tlc, and after 8 minutes, the reaction was completed. The mixture was chromatographed on silica gel using solvent gradient hexane/ethyl acetate. Yield 76%

Melting point: 102-104 °C (ethylacetate, uncorrected).

IR (ν cm^{-1}; $CHCl_3$ film): 3452 (O-H), 3010 (C_{sp2}-H Ar), 2985 (C_{sp3}-H).

^{1}H-NMR (200 MHz; $CDCl_3$; Me_4Si, δ_H): 10.08 (1H, s, OH), 7.74 (2H, m), 6.55 (1H, m), 3.73 (3H, s), 3.66 (2H, s, Ar-CH_2), 2.82 (4H, m, -S-CH_2-), 2.71 (4H, m, -N-CH_2-).

^{13}C-NMR (50 MHz; $CDCl_3$; δ_C): 152.5 (C), 151.2 (C), 122.52 (C), 116.4 (CH), 111.5 (CH), 113.6(CH), 62.3 (Ar-CH_2), 55.66 (CH3), 54.36 (-N-CH_2-), 27.82 (-S-CH_2-).

FAB-MS m/z (rel%) (M+1): 240(25%), 215, 180, 154.

Elemental Analysis: Calculated for $C_{12}H_{17}O_2NS$ (239): C 60.22 %, H 7.16 %, N 5.85 %, O 13.37 %, S 13.40 %, found : C 60.49 %, H 7.54 %, N 5.49 %, O 13.05 %, S 13.22 %.

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