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

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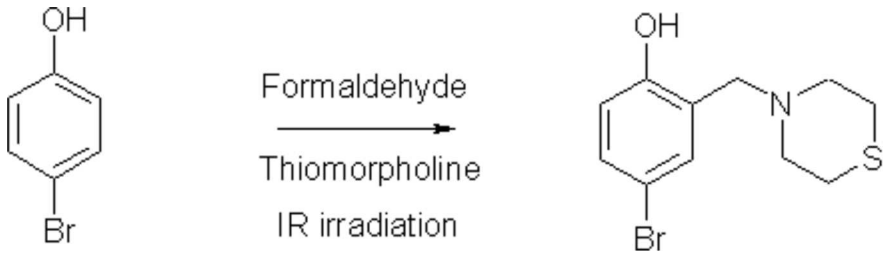

1 2

4-bromo-2-thiomorpholin-4-ylmethyl-1-phenol (2) was prepared from 4-bromophenol (1) and thiomorpholine and formaldehyde (2 eq.) and 1 eq. of thiomorpholine. 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 7 minutes, the reaction was completed. The mixture was chromatographed on silica gel using solvent gradient hexane/ethyl acetate. Yield 63%

Melting point: 130-132 °C (hexane/ethylacetate, uncorrected).

IR (n cm^{-1}; $CHCl_3$ film): 3520 (O-H), 3010 (C_{sp2}-H Ar), 2985 (C_{sp3}-H).

^{1}H-NMR (200 MHz; $CDCl_3$; Me_4Si, δ_H): 10.69 (1H, s, OH), 7.26 (1H, dd, J=8.8Hz, J=2.4Hz), 7.082 (1H, d, J=2.4Hz), 6.70 (1H, d, J=8.8Hz), 3.67 (2H, s, Ar-CH_2), 2.81 (4H, m, -S-CH_2-), 2.74 (4H, m, -N-CH_2-).

^{13}C-NMR (50 MHz; $CDCl_3$; δ_C): 156.7 (C), 131.61 (CH), 131.25 (CH), 122.76 (C), 117.9(CH), 110.88 (C), 61.66 (Ar-CH_2), 54.36 (-N-CH_2-), 27.84 (-S-CH_2-).

FAB-MS m/z (rel%) (M+1): 288(53%), 221, 135

Elemental Analysis: Calculated for $C_{11}H_{14}BrONS$ (287): C 45.99 %, H 4.87 %, N 4.87 %, O 5.57 %, S 11.15 %, Br 25.52, found : C 46.07 %, H 4.98 %, N 4.81 %, O 5.6 %, S 11.23 %, Br 25.34%.

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