

Synthesis of N,N'-bis (a-methylsalicylidene)-3,4'-diaminodiphenyl ether

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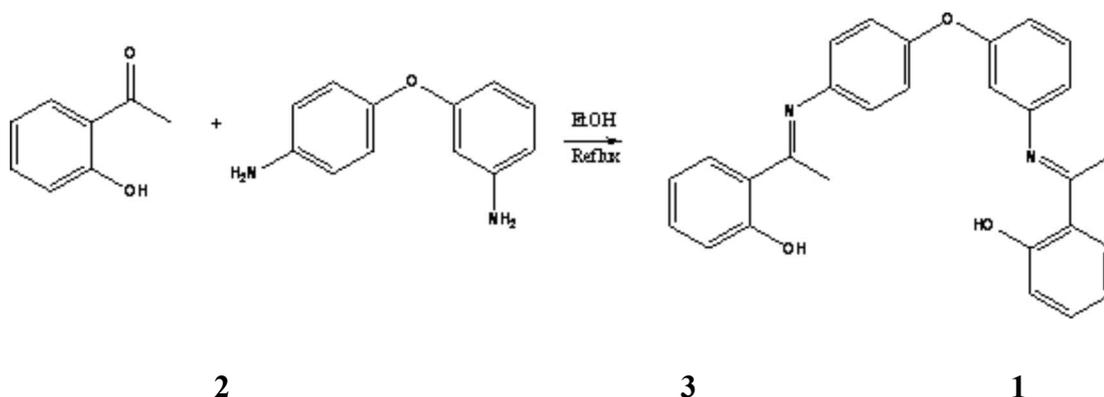
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There have been few reports about the synthesis and application of Schiff-base ligands derived from 2-hydroxyacetophenone [1]. In order to investigate the electronic, steric and geometric effect of a methyl group on an imine carbon on asymmetric catalytic reactions, 2-hydroxyacetophenone (**1**) has been chosen as starting material for synthesizing Schiff-base ligands [2]. Suhai and his coworkers have studied the conformational effects on the proton affinity of the Schiff base in bacteriorhodopsin [3]. Jacobsen and his coworkers have studied about asymmetric catalysis of hetero-ene reaction with tridentate Schiff base Cr(III) complexes[4].



2-Hydroxyacetophenone **1** (0.272 g, 0.24 mL, 2 mmol) and 3,4'-diaminodiphenylether **2** (0.33 g, 1 mmol) were dissolved in 10 mL of warm ethanol. The reaction mixture was refluxed for 10h and allowed to stand aside. The solid crystals were filtered off and washed with ethanol. The pure Schiff base **3** was isolated as a light yellow crystalline solid (yield 78%).

Melting Point: 182-184 °C

IR (KBr, ν , cm^{-1}): 3244(OH), 1620(C=N).

$^1\text{H-NMR}$ (250 MHz, CDCl_3): δ = 1.65(6H, s, CH_3), 6.21(2H, d, Ar), 6.24(2H, d, Ar), 6.88-7.64(5H, m, Ar), 7.94(2H, d, Ar), 14.57(2H, s, OH).

$^{13}\text{C-NMR}$ (62.9 MHz, CDCl_3): δ = 17.22; 111.71; 113.20; 118.22; 119.63; 126.66; 128.88; 132.95; 142.73; 145.60; 146.60; 162.15; 171.23.

MS (m/z): 436 (87%), 317 (40%), 210 (48%), 139 (38%), 91 (90%).

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Sample Availability: Available from MDPI.

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