

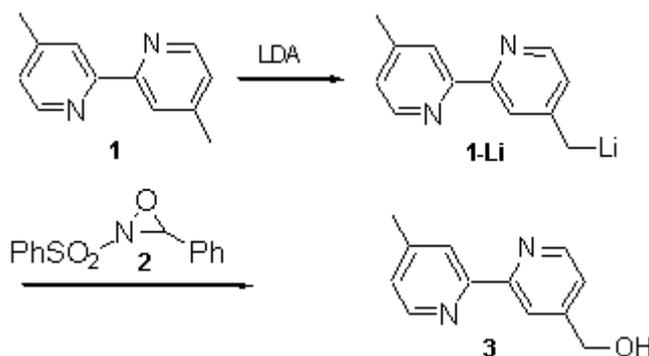
4-Hydroxy-methylen-4'-methyl-2,2'-bipyridine

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A key intermediate in the synthesis of extended bipyridine ligands [1] is 4-hydroxymethylene-4-methyl-2,2'-bipyridine (**3**), because the hydroxy group is readily converted into a variety of other chemical moieties, such as leaving or carbonyl groups. Two syntheses [2] for the preparation of **3** have been reported, both yielding **3** in two steps from **1** in 34% yield. We report here a more convenient one pot procedure giving an improved higher yield.



To a solution of one equivalent of 4,4-dimethyl-2,2'-bipyridene (**1**) in dry THF were added at -78°C 1.02 equivalents of freshly prepared LDA in THF to form the deeply red anion. After stirring for 30 minutes one equivalent of 2-phenylsulfonyl-3-phenyloxaziridine **2** [3] in THF was slowly added whereby the solution turned yellow. The mixture was allowed to warm up to room temperature, quenched with aqueous sat. NH_4Cl , washed with brine and the organic phase was evaporated to dryness. Column chromatography of the crude product on silica ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{OH}/\text{NH}_3$; 100/5/0.5) yielded 52 % of 4-hydroxymethylene-4-methyl-2,2'-bipyridine **3**. All spectroscopic data are identical to reported values in the literature.[2a].

^1H NMR (300 MHz, CDCl_3): δ = 2.34 (s, 3H, CH_3), 4.66 (s, 2H, CH_2), 5.25 (br s, 1H OH), 7.05-7.18 (m, 2H), 8.07-8.19 (m, 2H), 8.39-8.46 (m, 2H).

^{13}C NMR (75 MHz, CDCl_3) δ = 21.4 (CH_3), 63.1 (CH_2), 119.0 (CH), 121.4 (CH), 122.6 (CH), 125.0 (CH), 148.7 (C quart), 148.9 (CH), 149.2 (CH), 152.2 (C quart), 155.9 (C quart), 156.0 (C quart).

IR (KBr): 3200, 1596, 1456, 819.

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3. This reagent was synthesized within few hours from commercially available *N*-benzylidenebenzenesulfonamide according to a known procedure by Davis, F. A.; Chattopadhyay, S.; Towson, J. C.; Lal, S.; Reddy, T *J. Org. Chem.* **1988**, *53*, 2087-2089.

Sample availability: available from the authors and MDPI .

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