

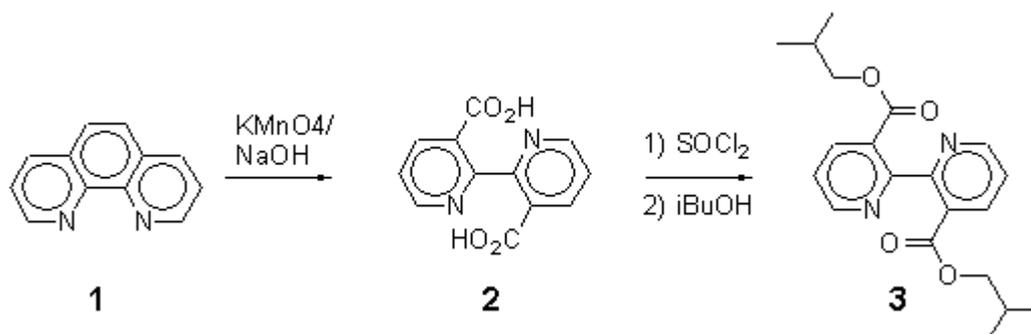
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Diisobutyl 2,2'-Bipyridine-3,3'-dicarboxylate

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The 2,2'-bipyridine-3,3'-dicarboxylic acid (**2**) was prepared from 1,10-phenanthroline (**1**) by a literature procedure [1, 2]. Compound **2** (600 mg, 2.5 mmol) and thionyl chloride (6 ml) were refluxed for 5 hours. The excess amount of thionyl chloride was distilled off and the residue dried in vacuum for 5 hours. Toluene (20 ml) and isobutanol (1.5 ml, 16.2 mmol) were added and the mixture refluxed for 20 hours. Chloroform (40 ml) was added and the mixture was treated with a cold solution of sodium bicarbonate (2.5 %). The organic layer was dried on sodium sulfate and the solvent removed in vacuum, giving 733 mg of crude product. The mixture was purified by chromatography on silica gel column using ether as eluent to afford **3** as a white solid (702 mg, 78%).

M.p. 88-89 °C.

IR (KBr, cm⁻¹): 1710, 1560, 1440, 1265.

¹H-NMR (250 MHz, CDCl₃): 8.74 (dd; 2H, H^{6/6'}, J = 4.8 and 1.6 Hz); 8.37 (dd, 2H, H^{4/4'}, J = 7.9 and 1.6 Hz); 7.44 (dd, 2H, H^{5/5'}, J = 7.9 and 4.8 Hz), 3.86 (d, 4H, 2CH₂, J = 6.6 Hz), 1.68 (m, 2H, 2CH); 0.76 (d, 12H, 4CH₃, J = 6.6 Hz).

Anal. Cal. for C₂₀H₂₄N₂O₄ (356.42): C 67.42, H 6.74, N 7.86; found : C 67.55, H 6.82, N 7.87.

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Sample Availability: Available from the authors and from MDPI. MDPI 17154.

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