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2'-(3'',4''-Methylenedioxyphenyl)imidazo[4',5'-*f*]1,10-phenanthroline

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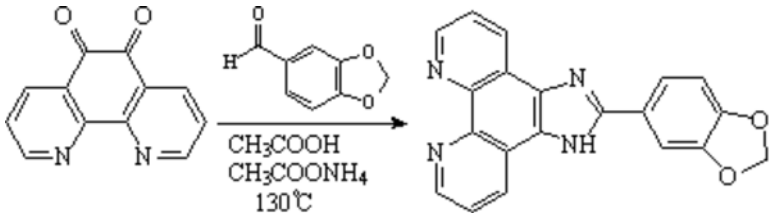

1,10-Phenanthroline-5,6-dione was prepared by a previously published procedure [1]. The title compound was synthesized according to the method for the preparation of imidazole rings established by Steck and Day [2]. A mixture of 3,4-(methylenedioxy)benzaldehyde (8 mmol), 1,10-phenanthroline-5,6-dione (5 mmol), ammonium acetate (100 mmol) and glacial acetic acid (20 cm^3) was refluxed with stirring at 130°C for about 1.5 hour, then cooled to room temperature and diluted with water (*ca.* 20 cm^3). Dropwise addition of concentrated aqueous ammonia with stirring gave a yellow precipitate, which was filtered and washed with water and acetone. The crude product was purified by silica gel filtration (60-100 mesh, ethanol as eluent). The principal yellow band was collected. After most of the ethanol solvent was removed under reduced pressure, the amorphous yellow solid was filtered and washed with acetone and diethyl ester, then dried in vacuum. Yield 1.03 g, 58%.

Elemental analyses: Found C, 69.23; H, 3.64; N, 15.87. Calc. For $C_{20}H_{12}N_4O_2\times0.5H_2O$, C, 68.76; H, 3.75; N, 16.04.

^{1}H NMR (500 MHz, $(CD_3)_2SO$): d 13.50 (br, s, 1H), 9.02 (dd, 2H), 8.90 (d, 2H), 7.85 (m, 2H), 7.80 (m, 2H), 7.12 (d, 1H), 6.19 (s, 1H).

IR (KBr, cm^{-1}): 463 (w), 547 (w), 621 (m), 740 (s), 813 (s), 879 (m), 937 (m), 963 (m), 1036 (s), 1073 (m), 1116 (m), 1190 (w), 1225 (s), 1258 (s), 1350 (s), 1397 (m), 1437 (m), 1471 (s), 1500 (m), 1562 (m), 1604 (m), 2898 (w), 2963 (w), 3001 (w), 3066 (w), 3105 (w), 2688-3105 (br), 3423 (br, m).

FAB-MS $([M+1]^+)$: 341, 50%.

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References

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