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4-*p*-Tolylazo-5-trifluoromethyl-2,4-dihydropyrazol-3-one

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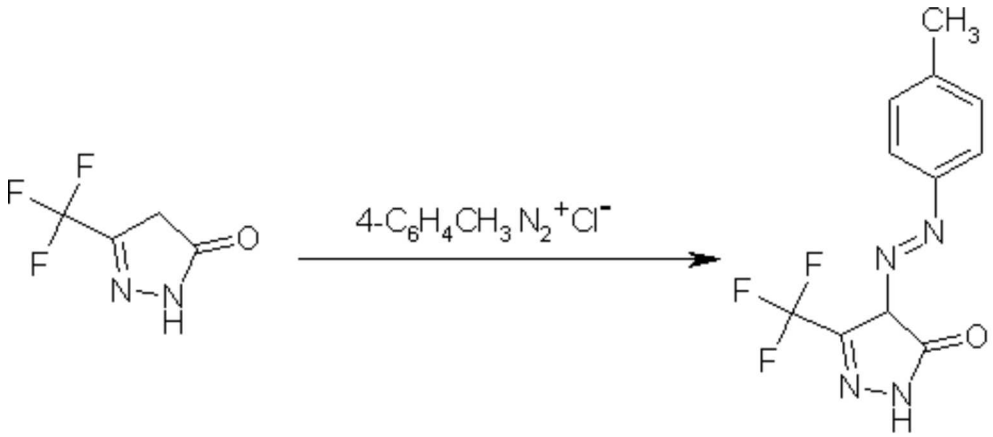

1 2

To a cold solution of 5-trifluoromethyl-2,4-dihydropyrazol-3-one [1,2] (0.76 g, 5 mmol) in ethanol (25 ml) containing sodium acetate (0.82 g, 10 mol), p-tolyldiazonium chloride [ca. 5 mmol; prepared by adding a solution of sodium nitrite (5 mmol / 2 ml H_2O) to an ice cold solution of p-toluidine in 2 ml conc. HCl] was added dropwise with stirring at 0-5°C. The reaction mixture was stirred at room temperature for three hours and the precipitated crude product was filtered, washed with water, dried and crystallized from ethanol to give 1.0 g (74%) of **2** as orange crystals.

M.p. 180-181°C

IR (KBr, cm^{-1}): 3210 (NH), 1670 (CO pyrazolone).

MS (m/z): 270.

^{1}H-NMR (250 MHz, DMSO-d_6): 2.31 (s, 3H, CH_3); 2.45 (s, 1H, CH); 7.21 (dd, 4H, aromatic CH); 12.58 (s, 1H, NH).

^{13}C-NMR (75 MHz, $CDCl_3$): 24.2 (CH_3); 51.5 (CH); 122.2 (2 aromatic carbons); 129.7 (2 aromatic carbons); 134.3, 143.7 (quaternary aromatic carbons); 135.7 (q, CF_3); 153.6 (C=N); 173.1 (CO).

References and Notes

1. Zohdi, H. F.; Elghandour, A. H. H.; Rateb, N. M.; Sallam, M. M. M. *J. Chem. Res.* **1992**, *(S)* 396, *(M)* 3015.

2. Zohdi, H. F.; Rateb, N. M. *Molecules* **2001**, *6*, M261.

Sample Availability: Available from the authors and from MDPI