1-Methyl-3-nitro-5-methoxycarbonyl Pyrazole

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The nitroester 2 was prepared by the addition of meta-chloro-perbenzoic acid (m-CPBA) to 1 [1] according to the reported procedure [2,3].

To a stirred solution of 1 (524 mg, 3.67 mmol) in dry CHCl₃ (9 ml), heated at 70 °C, m-CPBA (3.18 g, 14.8 mmol, 4 eq) dissolved in dry CHCl₃ (23 ml), was added. After 1 h, TLC analysis (AcOEt, Rf 0.5), showed the disappearance of the starting material. The mixture was cooled and filtered through a pad of celite. The filtrate, was washed twice with aqueous 10% NaOH and the organic phase was dried (MgSO₄) and concentrated at reduced pressure. The residue was purified by crystallization (light petroleum/ether) to afford 2 as a white solid (476 mg, 75%).

M.p. 66-68 °C.

TLC (AcOEt/light petroleum 7:3) Rf 0.37.

IR (KBr, cm⁻¹): 1730, 1550, 1380, 1330, 1300, 1270, 1130, 1090, 1000, 840, 760, 750.

¹HNMR (CDCl₃) d: 7.40 (s, 1H, CH); 4.29 (s, 3H, CO₂CH₃); 3.96 (s, 3H, N-C₃H₃).

Anal. calc. for C₆H₇N₃O₄ (185.14): C 38.93, H 3.81, N 22.70; found: C 38.71, H 3.89, N 22.89.

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References


Sample availability: Available from the authors.