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1-(2-ethoxy-2-oxoethyl)-5-methyl-1H-pyrazole-3-ethyl carboxylate

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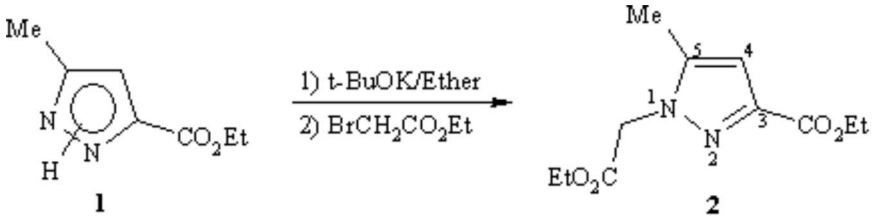

A mixture of **1** (6g; 39mmol) and potassium tert-butoxide (4.62g; 41.3mmol) in 120ml of anhydrous diethyl ether was refluxed for 75min. After cooling at 0°C, a solution of ethylbromoacetate (8.46g; 50.6mmol) in 20ml of anhydrous diethyl ether was slowly added. The reaction mixture was stirred for one night at room temperature then filtered and the solvent was evaporated to dryness. The obtained residue was purified on alumina using hexane as eluant to give a 15% yield of the new compound **2** (yellow oil) (1.4g; 5.83mmol).

^{1}H NMR spectrum of the compound 2 has a methyl group of pyrazol moiety at 2.33 ppm and a H_4 proton at 6.80 ppm with a $^4J(Me\text{-}H_4)$ = 0.72 Hz. Theses couplings being characteristic of a 5-methyl group [1-3]. Thus, the ^{1}H NMR spectrum is consistent with the structure **2**.

^{1}H-NMR ($CDCl_3$, 300 MHz): δ= 6.80 (s, 1H, Pz-H); 5.10 (s, 2H, N-CH_2-); 4.50 (q, 2H, -$\underline{CH_2}$-CH_3-, J=7Hz); 4.40 (q, 2H, -$\underline{CH_2}$-CH_3-, J=7Hz); 2.33 (s, 3H, -CH_3); 1.40 (t, 3H, CH_2-$\underline{CH_3}$, J=7Hz); 1.28 (t, 3H, CH_2-$\underline{CH_3}$, J=7Hz).

^{13}C-NMR ($CDCl_3$, 75 MHz): δ= 167.03; 162.35; 143.30; 141.02; 108.69; 62.05; 60.93; 51.30; 14.39; 14.10; 11.08.

Elemental analysis calculated for $C_{11}H_{16}N_2O_4$: C 55.00, H 6.71, N 11.66. Found: C 55.43, H 6.81, N 11.77.

IR (KBr, cm^{-1}): 1720 and 1740 (C=O).

Mass Spectrometry (ESI): m/z =241(M+1)

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