

## 2-Ethyl 4-methyl 5-amino-3-methylthiophene-2,4-dicarboxylate

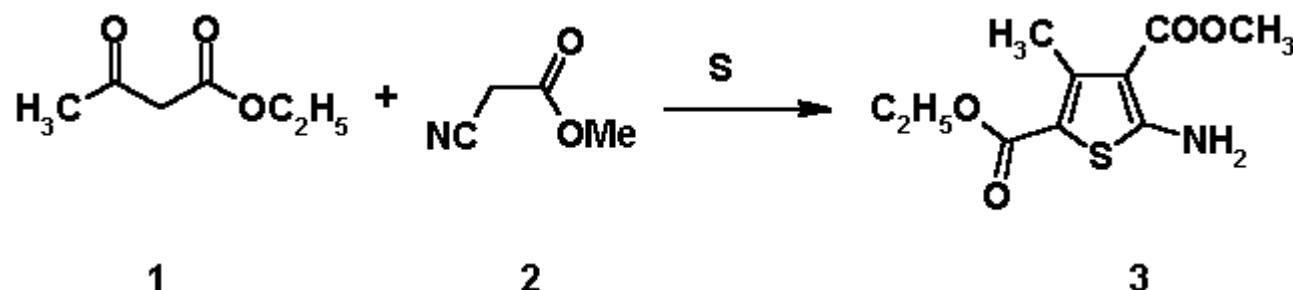
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To a mixture containing ethyl acetoacetate (13.02 g, 0.1 mol), methyl cyanoacetate (3.21 g, 0.1 mol) and sulfur (3.21 g, 0.1 mol) in ethanol (20 ml), morpholine (10 ml) was added dropwise over a period of 30 minutes at 35°C. The reaction mixture was heated at 65°C and stirred for 3 hrs, then allowed to cool to room temperature. The precipitated powder was filtered and washed with ethanol (2x30 ml).

Recrystallization from ethanol gave off-white powder (12.65 g, 52 %).

M.p. 95-97°C (Ethanol, uncorrected).

IR (KBr) (cm<sup>-1</sup>; KBr Disk) 3422, 3306 (NH), 1681 (C=O), 1589 (NH), 1331 (C-N).

<sup>1</sup>H-NMR (400 MHz; CDCl<sub>3</sub>; Me<sub>4</sub>Si, dH): 1.33 (3H, t, J = 7.16 Hz, CH<sub>3</sub>CH<sub>2</sub>O), 2.7 (3H, s, CH<sub>3</sub>), 3.84 (3H, s, CH<sub>3</sub>O), 4.26 (2H, q, J = 7.16 Hz, CH<sub>3</sub>CH<sub>2</sub>O), 6.57 (2H, bs, NH<sub>2</sub>).

<sup>13</sup>C-NMR (dC): 14.35, 16.0, 51.02, 60.38, 108.47, 147.89, 148.0, 162.83, 166.18, 166.46.

Elemental Analysis: Calculated for C<sub>10</sub>H<sub>13</sub>NO<sub>4</sub>S (243.28): C 49.37 %, H 5.39 %, N 5.76 %, S 13.18 %; found : C 49.22 %, H 5.42 %, N 5.84 %, S 13.01 %.

### References

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*Sample availability:* available from the author.

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