

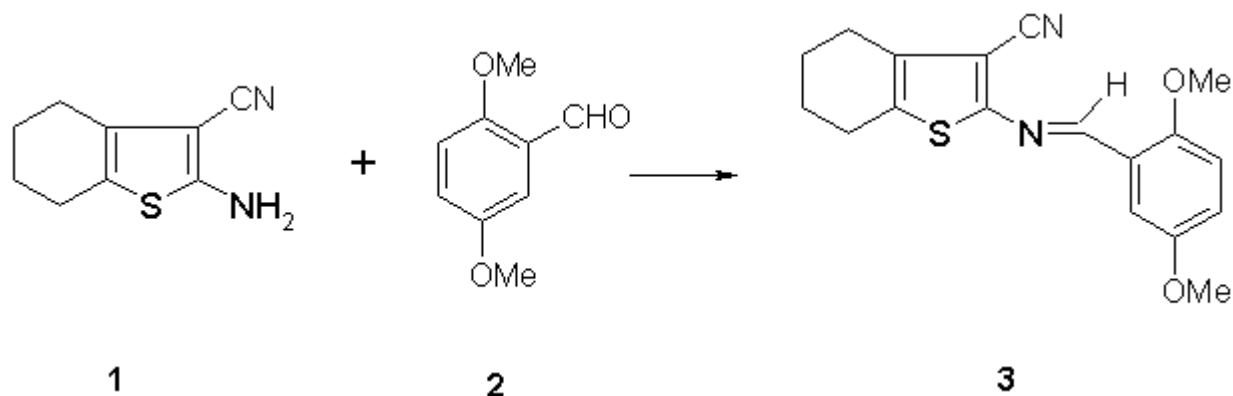
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3-Cyano-2-(2,5-dimethoxybenzalamino)-4-5-tetramethylenethiophene

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3-Cyano-2-(2,5-dimethoxybenzalamino)-4-5-tetramethylenethiophene was prepared by condensation of 2-amino-3-cyano-4,5-tetramethylenethiophene **1** and 2,5-dimethoxybenzaldehyde **2** [1,2]. A mixture of 2-amino-3-cyano-4,5-tetramethylenethiophene **1** (1.0 g, 5.62 mmol) and 2,5-dimethoxybenzaldehyde **2** (0.93 g, 5.62 mmol) in ethanol (20 mL) was heated under reflux for four hours. The reaction mixture was left to cool to room temperature and orange crystals were separated, filtered and washed with ethanol. Rerecrystallization from ethanol gave **3** as orange crystals (1.74 g, 95%).

Mp. 140-142 °C (EtOH, uncorrected).

UV $I_{\max}$ (nm; Acetone)/ ϵ (dm³·mol⁻¹·cm⁻¹): 408/11410.

IR (KBr) 2220 (CN), 1600 (C=N).

¹H-NMR (400 MHz; CDCl₃; Me₄Si): 1.85 (4H, d, 2xCH₂-5,6), 2.67 (4H, dd, 4xCH₂-4,7), 3.84, 3.85 (6H, s, CH₃O), 6.85 (1H, d, $J = 9.0$ Hz), 7.01 (1H, dd, $J_{46} = 3.1$ Hz, $J_{43} = 9.0$ Hz, H-4), 7.72 (1H, d, $J_{64} = 3.1$ Hz, H-6), 8.82 (1H, s, CH=N-).

¹³C-NMR: 22.0, 23.1, 24.3, 25.2, 55.8, 56.1 (2xCH₃O), 106.4 (CN), 110.5, 112.6, 114.6, 121.3, 123.9, 132.1, 134.9, 153.7, 154.7, 154.8 (CH=N), 160.9.

Anal.Calc. for C₁₈H₁₈N₂O₂S (326.420): C 66.23, H 5.56, N 8.58; found : C 66.41, H 5.35, N 8.39.

References

1. Asiri, A. M. *Pak. J. Sci. Ind. Res.* **1998**, *41*, 242.
2. Chalkley, L. *Chem. Rev.* **1929**, *6*, 217.

Sample availability: available from the authors and MDPI.

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