Molbank **2002**, M278 www.molbank.org

2-(1,3-Benzodioxol-5-yl)ethylene-1,1,2-tricarbonitrile

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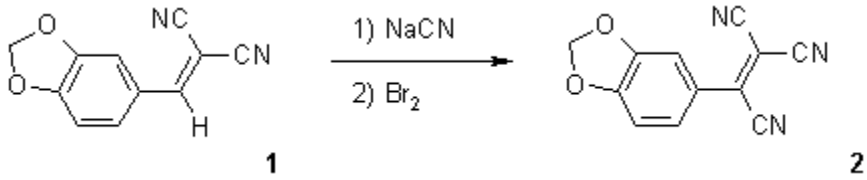
Received: 16 January 2002 / Revised: 15 March 2002 / Accepted: 15 May 2002 / Published: 16 February 2003

Keywords: tricarbonitrile, malononitrile, piperonal



1 **2**

2-(1,3-benzodioxol-5-yl)ethylene-1,1,2-tricarbonitrile (**2**), was prepared from (1,3-benzodioxol-5-ylmethylene)malononitrile (**1**) and sodium cyanide, followed by oxidation using bromine [1,2]. A solution of **1** (1.0 g, 5.0 mmol) in DMF (25 mL) was cooled to 5 ^{o}C followed by dropwise addition of sodium cyanide (0.25g, 5.0 mmol) in water (10 mL). When the addition was completed, the reaction mixture was stirred at room temperature for 3 hrs. Bromine (5 mL) was added and the stirring was continued for further two hours. 1-Hexene(50 mL) was added to destroy the excess bromine. The reaction mixture was poured into water and the precipitated solid was collected by filtration. The product was recrystallizedfrom 1:1 toluene:chloroform as deep orange crystals (0.85g, 75%).

M.p. 129-131 ^{o}C (toluene:chloroform, uncorrected).

UV λmax (nm; Chloroform)/ε ($dm^3.mol^{-1}.cm^{-1}$) 344/6600, 438/11800.

IR (cm^{-1}; KBr Disk) 2226 (CN), 1609 (C=C).

^{1}H-NMR (400 MHz; $CDCl_3$; Me_4Si, δ_H): 6.20 (2H, s, OCH_2O), 7.02 (1H, d, J = 9.72Hz), 7.59 (1H, s, H-2), 7.68 (1H, d, J = 8.2Hz, H-6).

^{13}C-NMR (δ_C): 88.24 (OCO), 103.29, 107.92, 109.64, 111.72, 111.77, 113.78, 122.97, 128.59, 140.06, 149.33, 154.73.

Elemental Analysis: Calculated for $C_{12}H_5N_3O_2$ (223.15): C 64.58%, H 2.24%, N 18.83%; found : C 64.36%, H 2.42%, N 18.98%.

References

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