

Trimethyl 4-(3,4-Dichlorophenyl)-2-hydroxy-2-(2-methoxy-2-oxoethyl)-6-oxo-1,3,5-cyclohexanetricarboxylate**Edmont V. Stoyanov**

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Received: 26 September 2002 / Accepted: 17 October 2002 / Published: 13 April 2003

Keywords: Michael addition, cyclohexanone, dimethyl acetonedicarboxylate, cyclization

Aromatic aldehydes react with dimethyl acetonedicarboxylate in molar ratio 1:2 with spontaneous intramolecular Michael addition to give polysubstituted cyclohexanones [1]. We report now the synthesis of an analogous product from 3,4-dichlorobenzaldehyde.



To a solution of 3,4-dichlorobenzaldehyde (1.75 g, 10 mmol) and dimethyl acetonedicarboxylate (3.48 g, 20 mmol) in 25 ml ethanol, 0.3 ml piperidine was added. The reaction mixture was left to stay at room temperature for 3 days. The separated crystals were filtered off, washed with cold ethanol, recrystallized from dioxane and air-dried. Yield: 3.15 g (62 %).

Colorless crystals, m.p. 162-163 °C (dec.) from dioxane.

¹H NMR (300 MHz, d₆-DMSO): 2.44 (d, 1H, J=17.2 Hz, HCH), 3.00 (d, 1H, J=17.2 Hz, HCH), 3.38 (s, 3H, OCH₃), 3.44 (s, 3H, OCH₃), 3.58 (d, 1H, J=12.2 Hz, H-3), 3.62 (s, 3H, OCH₃), 3.67 (s, 3H, OCH₃), 3.98 (t, 1H, J₁=J₂=12.2 Hz, H-4), 4.48 (d, 1H, J=12.2 Hz, H-5), 4.69 (s, 1H, H-1), 5.61 (s, 1H, OH), 7.28-7.71 (m, 3H arom.).

¹³C NMR (75 MHz, d₆-DMSO): 41.2, 42.5, 51.4, 51.5, 51.7, 51.8, 54.0, 60.5, 62.8, 74.3, 129.2, 130.0 (2xC), 130.6 (2xC), 130.9, 140.9, 167.4, 167.9, 169.4, 169.5.

FT IR (KBr, cm⁻¹): 3523, 3081, 2959, 2925, 1719, 1560, 1474, 1433, 1359, 1310, 1209.

ESI MS [FIA in MeOH, CH₃COONH₄/CH₃COONa]: 527.1 [M+Na]⁺, 522.2 [M+NH₄]⁺.

Reference

1. Haensel, W.; Haller, R. *Arch. Pharm. (Weinheim Ger.)* **1970**, *303*, 334-338.

Sample Availability: Available from the authors and from MDPI.

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