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Trimethyl 2-Hydroxy-2-(2-methoxy-2-oxoethyl)-4-(4-methylphenyl)-6-oxo-1,3,5-cyclohexanetricarboxylate

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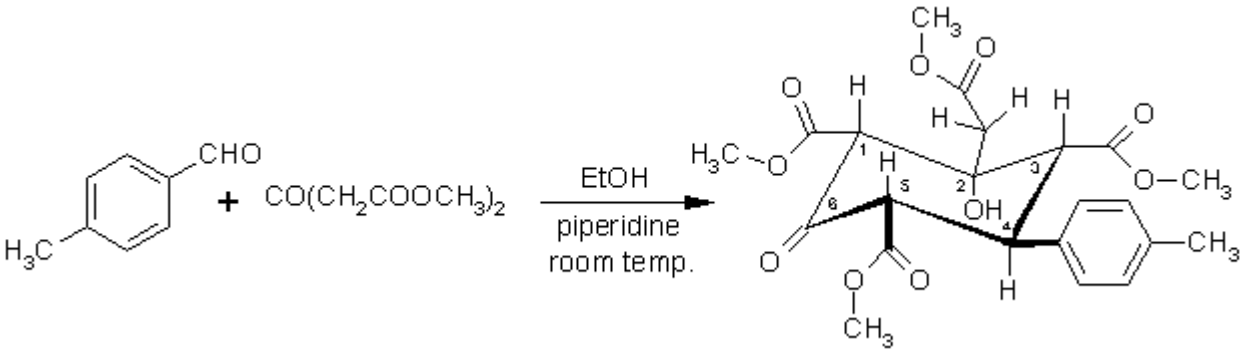
Aromatic aldehydes react with dimethyl acetonedicarboxylate in molar ratio 1:2 with spontaneous intermolecular Michael addition to give polysubstituted cyclohexanones [1]. We report now the synthesis of an analogous product from 4-methylbenzaldehyde.



To a solution of 4-methylbenzaldehyde (1.20 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: 3.18 g (71 %).

Colorless crystals, m. p. 149-150 ºC (dec.) from dioxane.

^{1}H NMR (300 MHz, d_6-DMSO): 2.23 (s, 3H, PhC$\underline{H}_3$), 2.43 (d, 1H, J=17.0 Hz, $\underline{H}$CH), 2.96 (d, 1H, J=17.0 Hz, HC$\underline{H}$), 3.44 (s, 3H, OCH_3), 3.52 (d, 1H, J=12.2 Hz, H-3), 3.56 (s, 3H, OCH_3), 3.60 (s, 3H, OCH_3), 3.66 (s, 3H, OCH_3), 3.94 (t, J1=J2=12.2 Hz, H-4), 4.38 (d, 1H, J=12.2 Hz, H-5), 4.67 (s, 1H, H-1), 5.40 (s, 1H, OH), 7.06 (d, 2H, J=8.0 Hz, 2' and 6'H arom.), 7.19 (d, 2H, J=8.0 Hz, 3' and 5'H arom.).

^{13}C NMR (75 MHz, d_6-DMSO): 41.4, 42.8, 51.1, 51.5, 51.6, 51.7, 54.3, 61.2, 62.8, 66.3, 74.3, 128.1 (2xC), 128.9 (2xC), 136.2, 136.6 (2xC), 167.7, 168.2, 169.5, 169.8.

FT IR (KBr, cm^{-1}): 3511, 2953, 1729, 1516, 1495, 1364.

ESI MS [FIA in MeOH, CH_3COONH_4/CH_3COOK]: 468.2 $[M+NH_4]^+$, 489.2 $[M+K]^+$.

Reference

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

Sample Availability: Available from the authors and from MDPI.