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Ethyl 6-formyl-8-methoxy-2-oxo-2*H*-chromene-4-carboxylate

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A mixture of vanillin 1 (0.5g, 3.3mmol) and triphenylphosphine (0.86g, 3.3mmol) was dissolved in CH₂Cl₂. The reaction mixture was cooled in ice bath to -5 °C. Dimethylacetylenedicarboxylate 2 (0.56g, 3.3mmol) in CH₂Cl₂ was added dropwise over a period of 10 min under stirring. The reaction mixture was then refluxed for 4-5 hours, the solvent was removed under reduced pressure and the solid mass was purified by recrystallization from ethanol to give the desired product as yellow crystals (0.45g, 50%).

M.p. 118 ^OC (recrystallized from EtOH, uncorrected).

UV 1_{max} (nm; EtOH)/e (dm³.mol⁻¹.cm⁻¹) 335/15875 IR (cm⁻¹, KBr) 2980, 2863 (C-H, aldehydic), 1725 (C=O), 1600 (C=C).

 1 H-NMR (400 MHz; CDCl₃; Me₄Si) d_H 9.99 (s, 1H, CHO), 8.44 (d, 1H, J = 1.3 Hz, H-3), 7.62 (d, 1H, J= 1.3 Hz, H-5), 7.08 (s, 1H, H-7), 4.5 (q, 2H, J = 7.0 Hz, CH₂O), 4.02 (s, 3H, CH₃O), 1.47 (t, 3H, J = 7.0 Hz, CH₃).

¹³C-NMR (100 MHz; CDCl₃; Me₄Si) d 13.85, 56.22, 62.67, 110.33, 116.29, 120.26, 123.33, 128.19, 132.44, 141.67, 147.83, 158.31, 163.02, 190.31.

Anal.Calc. for C₁₄H₁₂O₆ (276.241): C 60.87, H 4.38; found : C 60.61, H 4.23.

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References

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