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

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A mixture of Vanillin 1 (0.5 g, 3.3 mmol) and triphenylphosphine (0.86 g, 3.3 mmol) was dissolved in CH₂Cl₂. The reaction mixture was cooled in ice bath to -5 °C. Dimethylacetylenedicarboxylate (0.56 g, 3.3 mmol) in CH₂Cl₂ was added dropwise over a period of 10 min with 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 recrystalization from ethanol.

M.p. 182 ^oC (EtOH, uncorrected).

 $UV 1_{max} (nm; EtOH)/e (dm^3.mol^{-1}.cm^{-1}) 326/14000$

IR nmax (cm⁻¹; KBr Disk) 2956, 2865 (C-H, aldehydic), 1764 (C=O), 1725 (C=O), 1607 (C=C).

¹H-NMR (400 MHz; CDCl₃; Me₄Si) d_H 10.15 (s, 1H, CHO), 8.6 (s, 1H, H-3), 7.7 (s, 1H, H-5), 7. 4 (s, 1H, H-7), 4.2 (s, 6H, 2xCH₃O).

¹³C-NMR dC 53, 56, 110.6, 111.8, 116.5, 120.7, 123.6, 132.1, 132.7, 141.6, 148.4, 158.5, 190.5.

Anal.Calc. for C₁₃H₁₀O₆ (262.215): C 59.55, H 3.84; found: C 59.23, H 3.95.

References

- 1- Y. Issa, H-S. Rahim and Z. Afsaneh, Tetrahedron Letters, 1998, 39 (16), 2391.
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