

Original Article

Synthesis And Spectral Characterization Of 3-Benzoyl-6-Chlorochroman-4-Ones (Flavanones)

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Abstract

Three 3-benzoyl-6-chlorochroman-4-ones, [3-I(a-c)], were obtained by the piperidine-catalysed Claisen-Schmidt condensation of 1-(5-chloro-2-hydroxyphenyl)-3-phenylpropane-1,3-dione with 4-chlorobenzaldehyde, 4-methylbenzaldehyde or benzaldehyde, and characterized by ¹H NMR, IR and UV spectroscopy, elemental analysis and mass spectrometry. Each flavanone shows the two vicinally coupled ring-methine doublets, H-2 near δ 5.4 and H-3 near δ 5.9, with $J \approx 11$ Hz, establishing the trans-diaxial relationship of the two protons; the conjugated ring carbonyl absorbs near 1680 cm^{-1} and the principal UV band lies at 308–312 nm. The molecular ions carry the chlorine isotope patterns required by the assigned formulae, and the benzoyl cation at m/z 105 is the base peak. Representative spectra of [3-I(a)] are presented; yields were 72–76%.

Keywords: chroman-4-one; flavanone; Claisen-Schmidt condensation; vicinal coupling; trans configuration

Introduction

Flavanones are among the most widely studied flavonoid sub-classes and are valued for their antioxidant, antimicrobial and anti-inflammatory properties (Harborne & Williams, 2000; Middleton et al., 2000; Pietta, 2000). The 3-aryl-substituted flavanones considered here are especially useful, because the extra acyl group at C-3 makes them convenient springboards to a variety of heterocycles. They are prepared by condensing a 1-(2-hydroxyphenyl)-1,3-propanedione, obtained by the Baker-Venkataraman route (Baker, 1933; Mahal & Venkataraman, 1934), with an aromatic aldehyde under mild base catalysis; the reaction is of the Claisen-Schmidt type (Claisen & Claparède, 1881; Schmidt, 1881) and closes the pyran ring by intramolecular oxa-Michael addition. The relative configuration set up at C-2 and C-3 in this ring-closure can be read directly from the proton spectrum, since the coupling constant between H-2 and H-3 depends on their dihedral angle and a large value point to a trans-diaxial arrangement. The present paper describes the synthesis of three such chlorinated flavanones and their characterization by proton, infrared and ultraviolet spectroscopy.

Experimental

1. General

Reagent-grade materials and purified solvents were used (Furniss et al., 1989); reactions were monitored by TLC on silica gel G (benzene). Melting points are uncorrected. IR spectra were recorded as KBr discs, ¹H NMR at 300 MHz in CDCl₃ with TMS as internal standard, UV in ethanol, and mass spectra under electron impact; assignments follow standard reference works (Silverstein et al., 2005; Pavia et al., 2015; Kemp, 1991).

2. Synthesis

A mixture of 1-(5-chloro-2-hydroxyphenyl)-3-phenylpropane-1,3-dione (0.01 mol) and the aromatic aldehyde (0.012 mol) was refluxed in absolute ethanol (25 mL) with piperidine (0.5 mL) for 15–20 min.

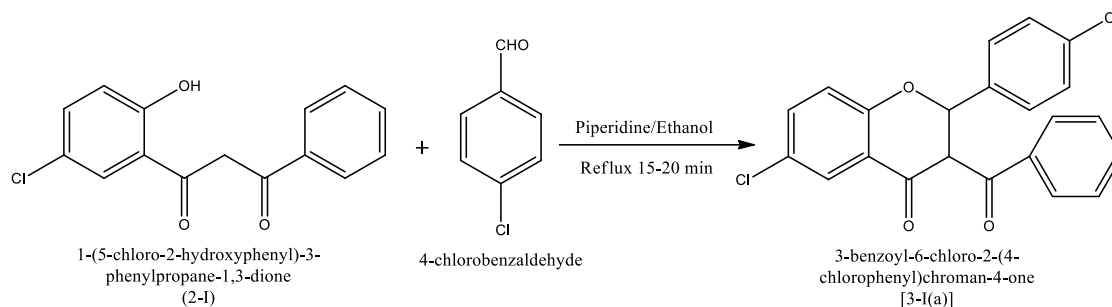
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The cooled mixture was acidified with dilute HCl, and the solid was filtered, washed and crystallised from ethanol-acetic acid to give the flavanone. The representative reaction, that of the 4-chlorobenzaldehyde member [3-I(a)],



Scheme 1. Claisen-Schmidt synthesis of the representative flavanone [3-I(a)] (reproduced from the thesis).

3. Characterization data

3-I(a) 3-Benzoyl-6-chloro-2-(4-chlorophenyl) chroman-4-one. Light-yellow crystals; m.p. 148 °C; yield 76%; Shinoda test positive. Anal. Calcd for $C_{22}H_{14}Cl_2O_3$ (397): C, 66.52; H, 3.55. Found: C, 66.47; H, 3.58. IR (KBr): 1680 (C=O), 1605, 1045 cm^{-1} . 1H NMR (300 MHz, $CDCl_3$): δ 5.38 (d, J = 11 Hz, 1H, H-2), 5.94 (d, J = 11 Hz, 1H, H-3), 7.60 (m, 13H). UV (EtOH) λ_{max} : 310, 252 nm. MS: m/z 396/398/400 (M, M+2, M+4; two Cl), 105 (base, $PhCO^+$), 77.

3-I(b) 3-Benzoyl-6-chloro-2-(p-tolyl) chroman-4-one. Light-yellow; m.p. 155 °C; yield 74%. Anal. Calcd for $C_{23}H_{17}ClO_3$ (376.5): C, 73.31; H, 4.55. Found: C, 73.26; H, 4.58. IR: 1680, 1605 cm^{-1} . 1H NMR: δ 2.37 (s, 3H, $ArCH_3$), 5.36 (d, J = 11 Hz, H-2), 5.91 (d, J = 11 Hz, H-3), 7.50 (m, 12H). UV: 312, 252 nm. MS: m/z 376/378, 105 (base).

3-I(c) 3-Benzoyl-6-chloro-2-phenylchroman-4-one. Light-yellow; m.p. 132 °C; yield 72%. Anal. Calcd for $C_{22}H_{15}ClO_3$ (362.5): C, 72.83; H, 4.17. Found: C, 72.79; H, 4.20. IR: 1680, 1605 cm^{-1} . 1H NMR: δ 5.35 (d, J = 11 Hz), 5.92 (d, J = 11 Hz), 7.60 (m, 14H). UV: 308, 250 nm. MS: m/z 362/364, 105 (base).

Results and Discussion

Condensation of the diketone with the three aldehydes gave the flavanones as pale-yellow solids in 72–76% yield. Ring-closure is confirmed by the negative ferric chloride reaction and by the positive Shinoda (Mg/HCl) test characteristic of the flavanone nucleus (Shinoda, 1928). The

is shown in Scheme 1; the p-tolyl [3-I(b)] and phenyl [3-I(c)] members were prepared analogously from 4-methylbenzaldehyde and benzaldehyde, respectively.

elemental analyses agree with the calculated compositions to within 0.05%.

1. Electronic spectra

The three flavanones absorb between 308 and 312 nm (Figure 1), the substituent on the C-2 aryl ring having only a small influence on the band position. The relatively short wavelength, compared with the open-chain diketone precursor, reflects the interruption of conjugation on cyclisation to the saturated C-2–C-3 unit.

2. Infrared spectra

The infrared spectrum confirms the chroman-4-one framework (Figure 2). The conjugated ring ketone absorbs strongly near 1680 cm^{-1} , aromatic C=C near 1605 cm^{-1} and the pyranone C–O–C stretch near 1045 cm^{-1} . The absence of any broad hydroxyl band confirms that the phenolic oxygen is now committed to the ring.

3. Proton magnetic resonance

The proton spectrum settles both constitution and configuration (Figure 3). Each flavanone gives two mutually coupled one-proton doublets, near δ 5.4 (H-2, the benzylic methine bearing the C-2 aryl group) and δ 5.9 (H-3, flanked by the ring and the exocyclic benzoyl carbonyl). Their coupling constant of about 11 Hz is the crucial value: through the Karplus relation such a large vicinal coupling corresponds to a near-antiperiplanar arrangement, so that H-2 and H-3 are trans-diaxial and the C-2 aryl and C-3 benzoyl groups both occupy equatorial positions. The p-tolyl member [3-I(b)] additionally shows the aryl-methyl singlet at δ 2.37.

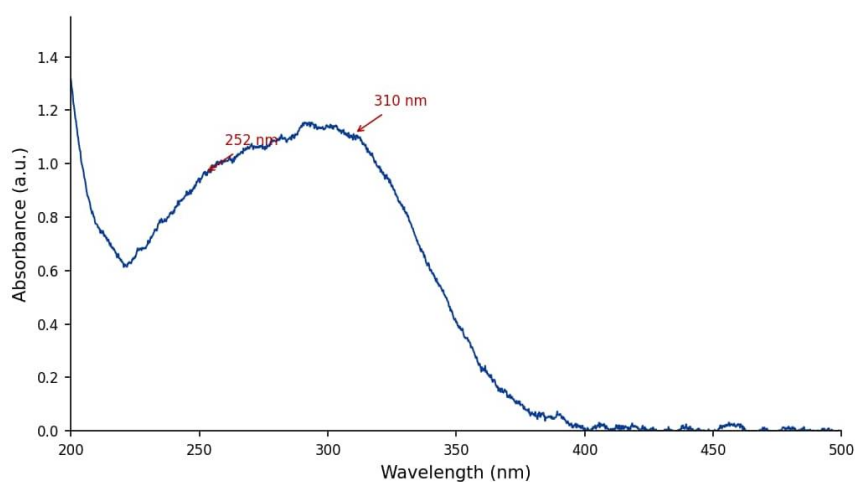


Figure 1. UV-Visible spectrum of flavanone [3-I(a)] (original spectrum, thesis data).

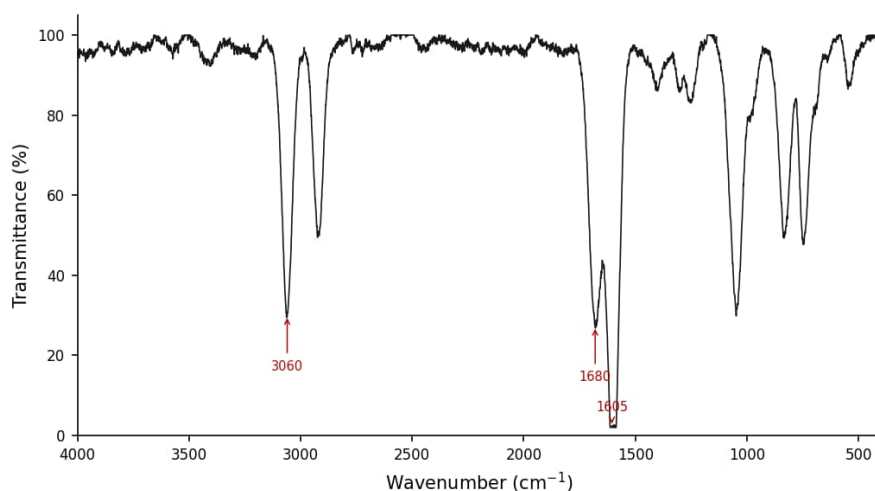


Figure 2. IR spectrum (KBr) of flavanone [3-I(a)] (original spectrum, thesis data).

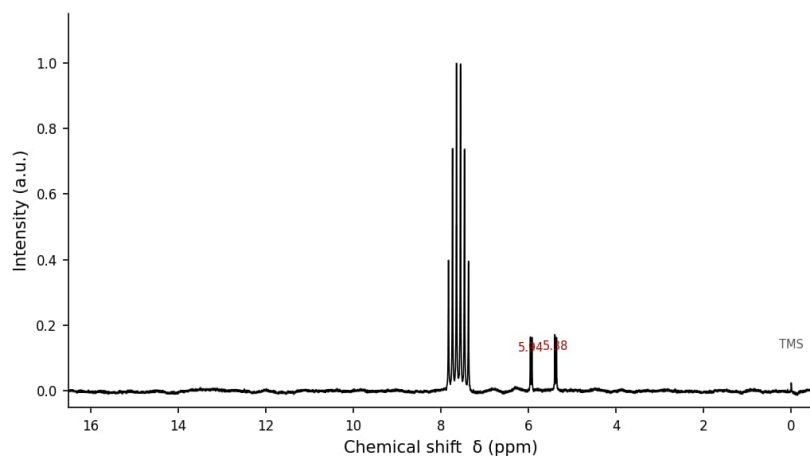


Figure 3. ¹H NMR spectrum (300 MHz, CDCl₃) of flavanone [3-I(a)] (original spectrum, thesis data).

Conclusion

Three 3-benzoyl-6-chloroflavanones have been prepared by Claisen-Schmidt condensation and characterized. The pair of vicinally coupled doublets at δ 5.4 and 5.9 ($J \approx 11$ Hz) establishes the chroman-4-one constitution and the trans-2,3 configuration; the ring ketone

absorbs near 1680 cm^{-1} and the principal UV band near 310 nm. The analytical and mass-spectral data confirm the structures.

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Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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