J. Chem. Sci. Vol. 123, No. 5, September 2011, pp. 673–679.

An efficient ultrasound promoted catalyst-free protocol for the synthesis of chromeno[4,3-b]quinolin-6-ones

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MS received 31 December 2010; revised 24 May 2011; accepted 30 May 2011

Abstract. A convenient, catalyst-free protocol for the quantitative synthesis of fused chromeno[4,3-b]quinolin-6-ones has been developed by simple one-pot reaction of substituted anilines with 4-chloro-3-formylcoumarin using ultrasound irradiation. The protocol offers the advantages of mild reaction conditions, short reaction times and high yields.

Keywords. Ultrasound; 4-chloro-3-formylcoumarin; anilines; chromeno-quinoline.

1. Introduction

Functionalized quinoline derivatives are integral component of several natural products which are used as antibacterial,[1] antiasthmatic,[2] antifungal,[3] anti-inflammatory[4] and anticancer[5] agents. Further, they are qualified as valuable starting materials for the synthesis of nano and mesostructures with improved electronic and photonic properties.[6] The synthesis of quinoline derivatives thus continues to be an active area of heterocyclic chemistry research, and the synthesis of various substituted quinolines has been largely explored.[7] On the other hand, coumarin derivatives are known to be equally important molecules endowed with wide spectrum of medicinal properties including antibacterial,[8] antiinflammatory,[9] antitumor[10] and anti-HIV[11] activities. They are also used as perfumes,[12] dyes[13] and fluorescent indicators.[14] Chromeno-quinolines are fused poly heterocyclic systems comprising both coumarin and quinoline motifs which are known to possess interesting biological properties like bacteriostatic activity,[15] glucocorticoid modulators,[16] antiinflammatory effects[17] and selective progesterone receptor modulators.[18] Thus the structural features and the biological applications prompt intense research by the organic chemists for the development of novel methodologies for their synthesis.

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2. Experimental

2.1 *General remarks*

^{1}HNMR spectra were recorded at 400 MHz with a Varian Gemini FT-NMR spectrometer. The chemical shifts are reported in δppm relative to tetramethylsilane (TMS). The Fourier transform FT–IR spectra were recorded using a Perkin-Elmer 16650 FT-IR spectrometer. The solvents and reagents were used without further purification.

2.2 *Synthesis of chromeno[4,3-b]quinolin-6-one (**3a–n**)*

A mixture of 4-Chloro-3-formylcoumarin (**1**) (1.0 mmol) and anilines **2a–n** (1.0 mmol) in 5 ml of ethanol were subjected to ultrasound irradiation (33 KHz) for 15 min resulting in precipitation of white solids which were filtered and washed with methanol to yield **3a–n** in 94–97% yield.

2.2a *6H-Chromeno[4,3-b]quinolin-6-one **(3a)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.24 (s, 1H), 8.85 (m, 1H), 8.25 (d, 1H), 8.04 (m, 1H), 7.91 (m, 1H), 7.62 (m, 2H), 7.49 (m, 2H); ^{13}C NMR (400 MHz, $CDCl_3$): 160.3, 158.4, 152.7, 146.7, 134.5, 132.4, 128.6, 128.1, 127.0, 126.6, 124.2, 122.5, 119.6, 118.6, 118.7, 114.4; IR (KBr, ν_{max}/cm^{-1}): 1735, 1732, 2965; EI-MS: m/z = 247.06.

2.2b *9-Methyl-6H-chromeno[4,3-b]quinolin-6-one **(3b)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.12 (s, 1H), 8.77 (m, 1H), 8.13 (d, 1H), 7.76 (s, 1H), 7.74 (d, 1H), 7.60 (d, 1H), 7.58 (m, 1H), 7.40 (m, 1H), 2.59 (s, 3H). ^{13}C NMR (400 MHz, $CDCl_3$): 161.4, 152.5, 149.7, 148.8, 139.9, 137.5, 135.7, 131.9, 129.2, 127.8, 127.3, 125.1, 124.8, 119.7, 117.3, 115.7, 21.5; IR (KBr, ν_{max}/cm^{-1}): 1735, 1734, 2923, 1188; EI-MS: m/z = 261.27

2.2c *9-Chloro-6H-chromeno[4,3-b]quinolin-6-one **(3c)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.15 (s, 1H), 8.77 (m, 1H), 8.19 (d, 1H), 8.01 (d, 1H), 7.86 (m, 1H), 7.61 (m, 1H), 7.44 (m, 1H), 7.26 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 160.9, 152.8, 149.9, 149.5, 139.9, 134.3, 133.4, 132.6, 131.2, 127.8, 127.7, 125.3, 125.1, 119.4, 117.5, 116.6; IR (KBr, ν_{max}/cm^{-1}): 3061, 1744, 1176; EI-MS: m/z = 281.44

2.2d *9-Nitro-6H-chromeno[4,3-b]quinolin-6-one **(3d)***: Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.18 (s, 1H), 8.73 (m, 1H), 8.16 (d, 1H), 8.01 (d, 1H), 7.82 (m, 1H), 7.60 (m, 1H), 7.44 (m, 1H), 7.24 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 159.8, 155.6, 152.2, 143.4, 135.7, 130.6, 126.9, 126.7, 126.3, 125.2, 125.1, 120.4, 118.9, 117.9, 117.7, 112.4; IR (KBr, ν_{max}/cm^{-1}): 1735, 1743, 1179; EI-MS: m/z = 292.25

2.2e *9-Bromo-6H-chromeno[4,3-b]quinolin-6-one **(3e)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.20 (s, 1H), 8.76 (m, 1H), 8.45 (s, 1H), 7.89 (d, 1H), 7.73 (m, 1H), 7.63 (m, 1H), 7.45 (m, 1H), 7.26 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 162.5, 159.4, 150.3, 146.2, 133.3, 132.3, 130.1, 128.1, 127.4, 126.9, 125.5, 125.1, 122.3, 121.3, 118.8, 113.2; IR (KBr, ν_{max}/c^{-1}): 1737, 1734, 1174; EI-MS: m/z = 326.14.

2.2f *9-Fluoro-6H-chromeno[4,3-b]quinolin-6-one **(3f)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.25 (s, 1H), 8.77 (m, 1H), 8.26 (m, 1H), 8.27 (m, 1H), 7.67 (m, 1H), 7.58 (m, 1H), 7.45 (m, 1H), 7.26 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 162.6, 160.1, 158.7, 154.4, 145.3, 136.6, 132.9, 127.1, 126.4, 122.9, 122.4, 118.2, 116.9, 116.1, 115.9, 113.9; IR (KBr, ν_{max}/cm^{-1}): 1735, 1740, 1218. EI-MS: m/z = 265.4

2.2g *10-Bromo-6H-chromeno[4,3-b]quinolin-6-one **(3g)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.17 (s, 1H), 8.75 (m, 1H), 8.46 (s, 1H), 7.90 (d, 1H), 7.73 (m, 1H), 7.63 (m, 1H), 7.45 (m, 1H), 7.24 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 161.3, 158.4, 154.3, 141.1, 131.6, 130.7, 129.1, 126.9, 126.5, 124.9, 122.3, 123.1, 122.3, 119.8, 117.8, 113.7; IR (KBr, ν_{max}/c^{-1}): 1735, 1737, 1172; EI-MS: m/z = 326.17.

2.2h *9,10-Difluoro-6H-chromeno[4,3-b]quinolin-6-one **(3h)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.46 (s, 1H), 8.63 (m, 1H), 8.25 (m, 1H), 7.89 (m, 2H), 7.70 (m, 1H), 7.52 (m, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 162.7, 158.2, 155.3, 152.8, 148.4, 134.2, 128.2, 125.5, 124.2, 120.1, 119.2, 118.7, 118.2, 116.5, 115.4, 115.7; IR (KBr, ν_{max}/cm^{-1}): 1735, 1739, 1176; EI-MS: m/z = 283.14.

2.2i *10,11-Difluoro-6H-chromeno[4,3-b]quinolin-6-one **(3i)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.47 (s, 1H), 8.65 (m, 1H), 8.28 (m, 1H), 7.88 (m, 2H), 7.75 (m, 1H), 7.53 (m, 1H). ^{13}C NMR (400 MHz, $CDCl_3$): 161.7, 156.2, 153.2, 151.3, 145.3, 136.7, 131.8, 127.4, 127.1, 126.4, 125.6, 124.4, 121.5, 120.3, 119.3, 118.1; IR (KBr, ν_{max}/cm^{-1}): 3046, 1734, 1737, 1178; EI-MS: m/z = 283.24.

2.2j *10-Fluoro-6H-chromeno[4,3-b]quinolin-6-one **(3j)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.21 (s, 1H), 8.77 (m, 1H), 8.05 (m, 1H), 7.85 (m, 1H), 7.62 (m, 1H), 7.42 (m, 1H), 7.39 (d, 1H), 7.26 (s, 1H); ^{13}C NMR (400 MHz, $CDCl_3$): 166.9, 161.1, 152.6, 150.7, 140.9, 132.7, 131.9, 125.4, 125.1, 119.4, 118.7, 118.4, 117.4, 115.3, 113.3, 113.1; IR (KBr, ν_{max}/c^{-1}): 3070, 1739, 1743, 1181; EI-MS: m/z = 265.05.

2.2k *9-Methoxy-6H-chromeno[4,3-b]quinolin-6-one **(3k)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.10 (s, 1H), 8.75 (m, 1H), 8.15 (d, 1H), 7.56 (m, 2H), 7.41 (m, 2H), 7.26 (s, 1H), 3.99 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): 161.5, 158.4, 152.4, 147.7, 147.5, 138.8, 132.2, 131.7, 131.1, 128.5, 126.9, 124.8, 119.8, 117.3, 115.9, 105.6, 55.7; IR (KBr, ν_{max}/cm^{-1}): 2990, 1735, 1737, 1237; EI-MS: m/z = 277.07.

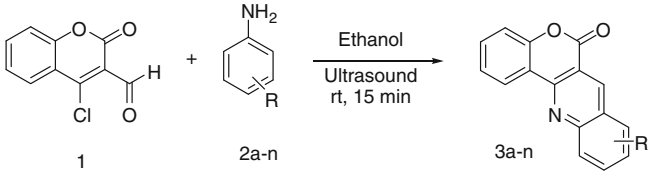
2.2l *10-Methoxy-6H-chromeno[4,3-b]quinolin-6-one **(3l)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.20 (s, 1H), 8.86 (m, 1H), 7.39-7.62 (m, 5H), 7.26 (s, 1H), 3.99 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): 162.7, 159.4, 153.4, 148.7, 147.4, 138.4, 132.7, 130.7, 129.1, 128.2, 126.4, 123.5, 118.9, 117.7, 116.1, 104.6, 54.6; IR (KBr, ν_{max}/cm^{-1}): 3063,1734, 1735, 1181; EI-MS: m/z = 277.07.

2.2m *10-Methyl-6H-chromeno[4,3-b]quinolin-6-one **(3m)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.14 (s, 1H), 8.77 (m, 1H), 8.10 (d, 1H), 7.72 (s, 1H), 7.70 (d, 1H), 7.60 (d, 1H), 7.57 (m, 1H), 7.40 (m, 1H), 2.56 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): 161.3, 152.6, 151.2, 149.5, 144.4, 140.3, 132.1, 129.7, 128.9, 128.4, 125.2, 124.8, 119.7, 117.2, 114.9, 22.2; IR (KBr, ν_{max}/cm^{-1}): 1735,1737, 2923, 1178; EI-MS: m/z = 261.26.

2.2n *10-Methoxy-6H-chromeno[4,3-b]quinolin-6-one **(3n)***: White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.65 (s, 1H), 8.75 (m, 1H), 7.89 (d, 1H), 7.81 (d, 1H), 7.60–7.30 (m, 3H), 7.2 (m, 1H), 4.05 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): 161.8, 159.4, 153.4, 148.7, 147.4, 138.4, 132.7, 130.7, 129.1, 128.2, 126.4, 123.5, 118.9, 117.7, 116.1, 104.6, 54.6; IR (KBr, ν_{max}/c^{-1}): 1735, 1737, 1178; EI-MS: m/z = 277.27.

3. Results and discussions

Chromeno[4,3-b]quinolin-6-one derivatives (**3a–n**) are important class of chromeno-quinolines and the most conventional approach for their synthesis involves the reaction of 4-hydroxycoumarins with anilines and paraformaldehyde at 220–240°C under vacuum.[19] Tabakovic *et al.* have reported their synthesis starting from 4-hydroxycoumarin under Vilsmeier-Haack conditions.[20] Asherson *et al.* have reported their synthesis from 3-((dimethylamino)methyl)-4-hydroxy- 2H-chromen-2-one and anilines but with lower yields.[21] Haber *et al.* have demonstrated stoichiometric $AlCl_3$ catalysed synthesis of the compounds by using 4-chloro-3-formylcoumarin and aniline in refluxing tetrahydrofuran.[22] Bandyopadhyay *et al.*[23] and Wu *et al.*[24] have used 4-oxo-4H-chromene-3-carbaldehyde and 4-chloro-3- formylcoumarin respectively for the synthesis of the chromeno[4,3-b]quinolin-6-ones. All the above methods require either Lewis acid catalysts or high temperatures for their synthesis.



Scheme 1. Synthesis of fused chromeno-quinolines.

Recently, ultrasonication methods have gained wide applicability in several organic synthetic protocols where the reaction conditions have been either modified and/or improved using the ultrasound irradiation.[25] We have earlier reported L-proline catalysed three component asymmetric Mannich reaction where ultrasonication conditions have efficiently enhanced the rate of the reaction along with improvement in the yields and enantioselectivities.[26] In view of the enormous potential of chromeno[4,3-b]quinolin-6-ones for various biological applications, we have envisaged an operationally benign catalyst-free protocol for their synthesis under ultrasound irradiation (scheme 1).

First, 4-chloro-3-formylcoumarin and *p*-methylaniline were subjected to ultrasound irradiation in various solvents. As shown in table 1 (entries 1–6), ultrasonication promotes the formation of the fused chromeno-quinolines **3b** in different solvents with varying reaction rates and yields. The results show that choice of solvent has profound effect on both the rates of reaction and yields. While the reaction did not proceed in water, solvents like dichloromethane and chloroform gave comparatively lower yields (~30%) after 30 min of ultrasound irradiation. Acetonitrile afforded 60% while methanol afforded 85% of the required product in 20 and 15 minutes respectively. Ethanol proved to be the solvent of choice which afforded 95% of the required product as a white precipitate in 15 min without the requirement for column purification.

The optimized reaction conditions were further extended to the reaction of various aniline substrates possessing diverse electronic features making the protocol rather general and the results are listed in table 2. For example, the electron-rich anilines having *p*-Me, *p*-OMe, *o*-OMe, *m*-Me, *m*-OMe functional groups afforded the respective products in 15 minutes with yield between 94 and 97% (entries 2, 11–14, table 2). Similarly, the electron withdrawing *p*-nitroaniline afforded the required product in 96% yield (entry 4,

Table 1. Effect of solvents for the synthesis of coumarino-quinolines from 4-chloro-3-formylcoumarin and aniline.[a]

Entry	Solvent	Time (min)	Yield[b](%)
1	C_2H_5OH	15	95
2	CH_3OH	15	85
3	CH_3CN	20	60
4	CH_2CL_2	30	30
5	$CHCl_3$	30	30
6	Water	45	NR[c]

[a]All the reactions were carried out using **1** (1.0 mmol), 2**b** (1.0 mmol) at room temperature under ultrasound conditions,
[b]determined by ^{1}H NMR.
[c]No reaction.

Table 2. Synthesis of chromeno[4,3-b]quinolin-6-ones.

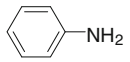
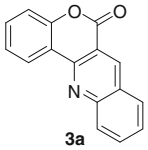
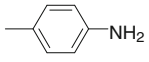
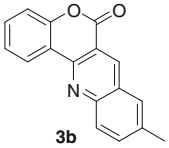
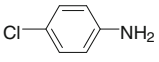
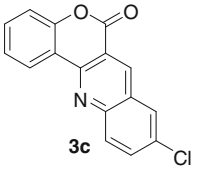
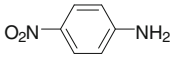
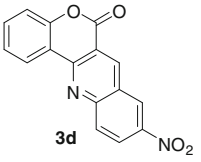
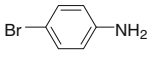
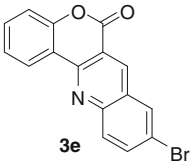
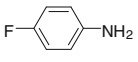
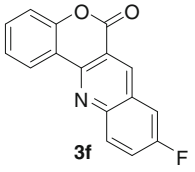
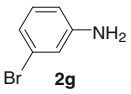
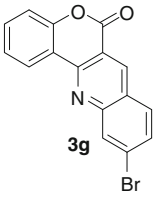
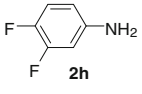
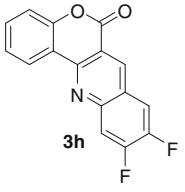
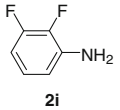
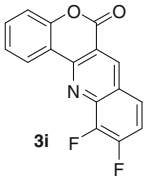
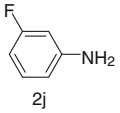
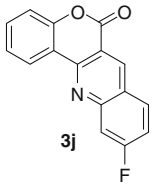
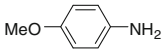
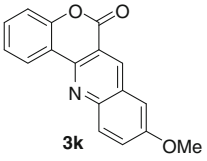
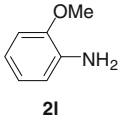
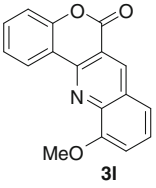
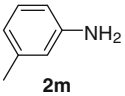
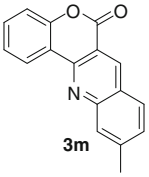
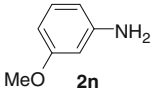
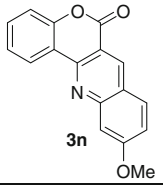
Entry	Anilines	Chromeno-quinoline **3a-n**	Yield (%)[a]
1	 **2a**	 **3a**	95
2	 **2b**	 **3b**	97
3	 **2c**	 **3c**	96
4	 **2d**	 **3d**	96
5	 **2e**	 **3e**	95
6	 **2f**	 **3f**	96
7	 **2g**	 **3g**	95
8	 **2h**	 **3h**	96

table 2). Anilines having various mono- and di-substituted halogens also gave satisfactory yields (94–96%) for the respective chromeno[4,3-b]quinolin-6-ones within 15 minutes under ultrasonication conditions (entries 3, 5–10, table 2). All the products were characterized by ^{1}HNMR, IR, and EI-Mass analysis and were

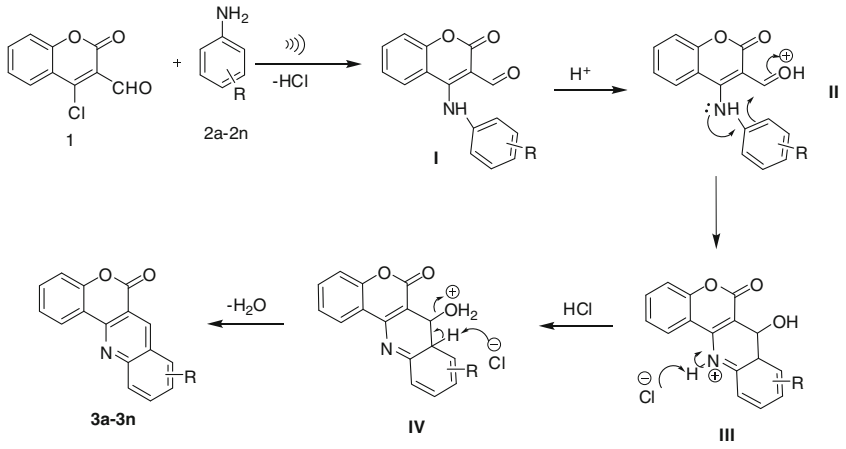
Table 2. (*continued*).

Entry	Anilines	Chromeno-quinoline **3a–n**	Yield (%)[a]
9	 **2i**	 **3i**	94
10	 2j	 **3j**	97
11	 **2k**	 **3k**	96
12	 **2l**	 **3l**	95
13	 **2m**	 **3m**	94
14	 **2n**	 **3n**	95

[a]Yields refer to the precipitated products.

found to be in good agreement with those reported in the literature.[22]

A plausible mechanism for the synthesis of **3a–3n** is proposed in scheme 2. The first step of the reaction involves nucleophilic attack of aniline on **1** resulting in the *N*-alkylation intermediate **I**. The second step involves the activation of the aldehyde group by the *in situ* generated HCl, which acts as a protic acid promoting the cyclization of the aromatic ring to generate intermediate **III**. Elimination of the water molecule in the final step affords fused quinoline products **3a–3n**. The ultrasound energy in the present protocol efficiently causes acoustic cavitation, which results in extreme temperatures and pressures on microsecond time scale causing dramatic influence on the rates of the reaction.[25d]



Scheme 2. Plausible mechanism for the synthesis of chromeno[4, 3-b]quinolin-6-ones.

4. Conclusion

In conclusion, we have reported an efficient, fast, catalyst-free ultrasonication methodology for the synthesis of fused chromeno[4,3-b]quinolin-6-one derivatives. The present methodology offers the advantages of mild reaction conditions, short reaction times; high yields and avoids the requirement of Lewis acid catalysts. Further, the protocol does not require column chromatography purification.

Acknowledgements

The authors thank Mr. A Sankar Rao and Mrs. A Yesodha, Founder Trustees of Sankar Foundation, for the kind support and encouragement.

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