

The effective reaction of 2-chloro-3-formylquinoline and acetic acid/sodium acetate under microwave irradiation

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Abstract

Pyrano[2,3-*b*]quinolin-2-ones have been synthesized efficiently from 2-chloro-3-formylquinolines in a single step by treating with sodium acetate and acetic acid under microwave irradiation. The structures of the compounds have been established by IR, NMR and mass spectral data. Unexpectedly, 3-formylquinolin-2(1*H*)-ones were exclusively formed in very high yields by changing the molar ratio of acetic acid and sodium acetate in just 1.5 to 2.5 min.

Keywords: pyrano, microwave, quinolines, solventless, acetic acid.

1. Introduction

The preparation of pyranoquinolines has received significant attention (Kamal *et al.*, 2004; Yadav *et al.*, 2002; Nagarajan *et al.*, 2001) in previous years because of the broad spectrum of their biological properties such as psychotropic, antiallergic, anti-inflammatory and estrogenic activities (Yamada *et al.*, 1992; Faber *et al.*, 1984). In addition, pyranoquinoline derivatives are found to possess a wide range of pharmacological activity (Magesh *et al.*, 2004). Further, several bioactive alkaloids which are widely distributed in nature contain a pyranoquinoline moiety (Ih-Sheng Chen *et al.*, 1997; Dagne *et al.*, 1988). Among the pyranoquinolines, pyrano[2,3-*b*]quinolin-2-one systems are of interest because they are linear benzaza analogues of coumarins and they constitute the parent ring structures of pyranoquinoline alkaloids such as khaplofoline, which occurs in the *rutaceae* plant family (Michael 1995; Brader *et al.*, 1996).

So far, only a few methods have been reported (Meth-Cohn *et al.*, 1981; Tilakraj and Ambekar 1985) for the construction of pyrano[2,3-*b*]quinolin-2-ones, which generally involve 2-chloro-3-formylquinolines (**1**) as starting materials. In previous methods, (Sekar and Rajendra Prasad 1998) 2-chloro-3-formylquinolines (**1**) were converted into 3-formylquinolin-2(1*H*)-ones which on treatment with malonic acid followed by PPA cyclisation afforded pyrano[2,3-*b*]quinolin-2-ones. Later this method was modified by the condensation of 2-chloro-3-formylquinolines with acetic anhydride and sodium acetate in a single step to give pyrano[2,3-*b*]quinolin-2-ones. Similar methods of preparation of 4-methoxy- pyrano[2,3-*b*]quinolin-2-one and some 3-substituted-pyrano[2,3-*b*]quinolin-ones have also been reported (Narasimhan and Bhagwat 1979; Venkatesh Kumar and Rajendran 2003).

However, these methods have some disadvantages, which include multiple steps, longer reaction time, and use of toxic organic solvents, harsh reaction conditions and requirement of excess of reagents such as hydrochloric acid, acetic anhydride and poly phosphoric acid.

Microwave irradiation using commercial domestic oven has been recently used to accelerate organic reactions, the high heating efficiency giving remarkable rate enhancement and dramatic reduction in reaction time (Jolivet *et al.*, 1996). As a part of a research project to develop environmentally benign organic reactions, we have recently reported (Nadaraj *et al.*, 2010; 2011) the synthesis of simple quinolines, pyrimido- and pyrazoloquinolines under microwaves. Hence, our new approach reported herein involves the use of microwave irradiation in the synthesis of pyrano[2,3-*b*]quinolin-2-ones under mild conditions and unexpected formation of 3-formylquinolin-2(1*H*)-ones as intermediates.

2. Materials and Method

Melting points (mp) were recorded on Boetieus microheating table and are uncorrected. IR (KBr) spectra were recorded on a Shimadzu-8201 FT spectrophotometer. ¹H NMR & ¹³C NMR spectra were recorded on Bruker AMX-500 (500 MHz) spectrophotometer, using TMS as an internal reference and Mass spectra were recorded at 70 eV on a Joel JMS-D-300 instrument. Microwave irradiations were carried out using a Kenstar, OM-20ESP, 2450 MHz, domestic microwave oven with adjustable irradiation power.

2.1 General procedure for the synthesis of pyrano[2,3-*b*]quinolin-2-ones (**2a-j**):

A mixture of 2-chloro-3-formylquinoline (0.5 mmol), glacial acetic acid (123 mmol) and sodium acetate (50 mmol) was taken in a closed Teflon vessel and irradiated in a microwave oven at power 320W for the specified time (**Table 2**). After irradiation, the reaction mixture was poured into crushed ice. The product obtained was filtered, washed, dried and recrystallized from ethyl acetate.

2.1.1 Pyrano[2,3-*b*]quinolin-2-one (2a): IR (KBr): $\nu=1621\text{ cm}^{-1}$, 1740 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=6.56$ (d, ³*J* (H,H) = 9 Hz, 1H; C₃-H), $7.50\text{--}7.85$ (m, 3H; C₇-H, C₈-H & C₉-H), 7.95 (d, ³*J* (H,H)= 8.3 Hz, 1H; C₆-H), 8.10 (d, ³*J* (H,H) = 9 Hz, 1H; C₄-H), 8.41 (s, 1H; C₅-H); ¹³C NMR (DMSO-*d*₆): $\delta=117.5$, 122.5 , 126.5 , 132.3 , 134.2 , 136.8 , 137.9 , 140.5 , 143.2 , 146.2 , 156.3 , 162.3 ; MS *m/z*: 197 [M⁺]; elemental analysis calcd (%) for C₁₂H₇NO₂; C 73.09, H 3.58, N 7.10; found: C 73.07, H 3.58, N 7.06.

2.1.2 7-Methoxypyran[2,3-*b*]quinolin-2-one (2e): IR (KBr): $\nu=1622\text{ cm}^{-1}$, 1735 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=3.86$ (s, 3H; C₇-OCH₃) 6.52 (d, ³*J* (H,H) = 9 Hz, 1H; C₃-H), $7.15\text{--}8.23$ (m, 4H; C₄-H, C₆-H, C₈-H & C₉-H), 8.42 (s, 1H; C₅-H); ¹³C NMR (DMSO-*d*₆): $\delta=56.5$, 116.9 , 120.9 , 125.8 , 131.5 , 134.0 , 136.2 , 137.8 , 140.3 , 144.2 , 145.8 , 157.1 , 162.2 ; EI-MS *m/z*: 227 [M⁺]; elemental analysis calcd (%) for C₁₃H₉NO₃; C 68.72, H 3.99, N 6.16; found: C 68.70, H 4.00, N 6.13.

2.1.3 9-Methoxypyran[2,3-*b*]quinolin-2-one (2g): IR (KBr): $\nu=1620\text{ cm}^{-1}$, 1720 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=3.90$ (s, 3H; C₉-OCH₃), 6.40 (d, 1H; C₃-H), $7.34\text{--}7.82$ (m, 4H; C₄-H, C₆-H, C₇-H & C₈-H) 8.42 (s, 1H; C₅-H); ¹³C NMR (DMSO-*d*₆): $\delta=56.7$, 118.2 , 122.4 , 126.3 , 132.7 , 134.2 , 136.4 , 138.1 , 140.2 , 143.2 , 145.5 , 156.2 , 162.1 ; MS *m/z*: 227 [M⁺]; elemental analysis calcd (%) for C₁₃H₉NO₃; C 68.72, H 3.99, N 6.16; found: C 68.69, H 3.99, N 6.13.

2.1.4 7-Bromopyran[2,3-*b*]quinolin-2-one (2h): IR (KBr): $\nu=1620\text{ cm}^{-1}$, 1725 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=6.38$ (d, 1H; C₃-H), $7.42\text{--}7.88$ (m, 4H; C₄-H, C₆-H, C₈-H & C₉-H), 8.45 (s, 1H; C₅-H); ¹³C NMR (DMSO-*d*₆): $\delta=119.2$, 124.1 , 127.2 , 133.2 , 135.1 , 136.9 , 138.4 , 140.3 , 143.9 , 147.0 , 156.3 , 162.5 ; MS *m/z*: 276 [M⁺]; elemental analysis calcd (%) for C₁₂H₆NO₂Br: C 52.17, H 2.19, N 5.07; found: C 52.18, H 2.19, N 5.10.

2.2 General procedure for synthesis of 3-formylquinolin-2(1H)-ones (**3a-j**):

A mixture of 2-chloro-3-formylquinoline (0.5 mmol), glacial acetic acid (87.5 mmol) and sodium acetate (25 mmol) was taken in an open Teflon vessel and irradiated in a microwave oven at power 320 W for the specified time (**Table 3**). After irradiation, the reaction mixture was poured into crushed ice. The precipitated product was filtered, washed with water, dried and recrystallized from aqueous acetic acid.

2.2.1 3-formylquinolin-2(1H)-one (3a): IR (KBr): $\nu=1550\text{ cm}^{-1}$, 1680 cm^{-1} (C=O), 3200 cm^{-1} H NMR (DMSO-*d*₆): $\delta=7.25$ (t, 1H; C₇-H), 7.35 (d, 1H; C₈-H), 7.66 (t, 1H; C₆-H), 7.92 (d, 1H; C₅-H), 8.52 (s, 1H; C₄-H), 10.24 (s, 1H; CHO), 12.25 (s, 1H; NH); ¹³C NMR (DMSO-*d*₆): $\delta=118.2$, 120.1 , 126.3 , 132.5 , 134.3 , 138.2 , 142.8 , 144.3 , 164.3 , 191.2 ; MS *m/z*: 173 [M⁺]; elemental analysis calcd (%) for C₁₀H₇NO₂; C 69.36, H 4.07, N 8.09; found: C 69.33, H 4.05, N 8.06.

2.2.2 6-Methoxy-3-formylquinolin-2(1H)-one (3e): IR (KBr): $\nu=1545\text{ cm}^{-1}$, 1675 cm^{-1} (C=O), 3200 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=3.91$ (s, 3H; C₆-OCH₃), 7.35 (d, 1H; C₈-H), 7.59 (d, 1H; C₇-H), 8.04 (s, 1H; C₅-H), 8.35 (s, 1H; C₄-H), 10.41 (s, 1H; CHO), 11.52 (s, 1H; NH); ¹³C NMR (DMSO-*d*₆): $\delta=56.5$, 118.2 , 120.9 , 126.4 , 132.6 , 134.2 , 138.1 , 142.9 , 144.2 , 164.2 , 191.1 ; MS *m/z*: 203 [M⁺]; elemental analysis calcd (%) for C₁₁H₉NO₃; C 65.02, H 4.46, N 6.89; found: C 65.05, H 4.44, N 6.86.

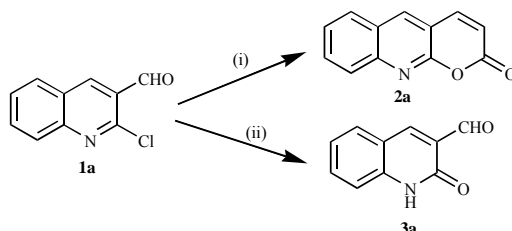
2.2.3 8-Methoxy-3-formylquinolin-2(1H)-one (3g): IR (KBr): $\nu=1550\text{ cm}^{-1}$, 1685 cm^{-1} (C=O), 3200 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=3.92$ (s, 3H; C₈-OCH₃), $7.39\text{--}7.60$ (m, 2H; C₆- & C₇-H), 8.05 (d, 1H; C₅-H), 8.38 (s, 1H; C₄-H), 10.39 (s, 1H; CHO), 11.52 (s, 1H; NH); ¹³C NMR (DMSO-*d*₆): $\delta=56.7$, 119 , 121.0 , 126.5 , 132.2 , 135.1 , 138.6 , 143.1 , 144.6 , 164.2 , 190.9 ; MS *m/z*: 203 [M⁺]; elemental analysis calcd (%) for C₁₁H₉NO₃; C 65.02, H 4.46, N 6.89; found: C 65.07, H 4.42, N 6.90.

2.2.4 6-Bromo-3-formylquinolin-2(1H)-one (3h): IR (KBr): $\nu=1545\text{ cm}^{-1}$, 1680 cm^{-1} (C=O), 3200 cm^{-1} (C=O); ¹H NMR (DMSO-*d*₆): $\delta=7.25\text{--}7.65$ (m, 2H; C₇-H & C₈-H), 8.20 (s, 1H; C₅-H), 8.45 (s, 1H; C₄-H), 10.25 (s, 1H; CHO), 11.01 (s, 1H; NH).

3. Results and Discussion

To identify an efficient reagent/catalyst for the synthesis of pyrano[2,3-*b*]quinolin-2-one derivatives, we initially examined the reaction of 2-chloro-3-formylquinoline (**1**) with acetic acid and sodium acetate under microwave irradiation. Various parameters such as different molar ratio of acetic acid and sodium acetate, irradiation power and time and using closed or open Teflon vessel were studied and optimized (**Table 1, Methods 1-5**).

When we irradiated (**Method 1**) 0.5 mmol of 2-chloro-3-formylquinoline (**1a**) with 123 mmol of acetic acid and 50 mmol of sodium acetate in a closed Teflon vessel, for 5 min at power 320 W, pyrano[2,3-*b*]quinolin-2-one (**2a**) was formed in good yield (92%) (**Scheme 1**).



Scheme 1. Reagents and conditions

- (i) Acetic acid (123 mmol), sodium acetate (50 mmol), microwave, 320W, 5.00 min.
(ii) Acetic acid (87.5 mmol), sodium acetate (25mmol), microwave, 320 W, 2.20 min.

Table 1. Microwave irradiation of 2-chloro-3-formylquinoline (**1a**) with acetic acid and sodium acetate under different conditions

Methods	Reagent/ Catalyst	Molar ratio (mmol)	Irradiation Power (W)	Time (min)	Product formed	Yield (%)	Teflon Vessel type
1	AcOH/ NaOAc	123 and 50	320	5.00	2a	92	Closed
2	AcOH/ NaOAc	87.5 and 25	320	2.20	3a	98	Open
3	AcOH	175	480	15.00	2a	67	Open or Closed
4	NaOAc	50	480	10.00	3a	80	Open
5	AcOH/ NaOAc	123 and 50	320	5.00	2a & 3a	-	Open

The above reaction has also been tried out with lesser amount of acetic acid and sodium acetate (87.5 mmol and 25 mmol) in an open Teflon vessel, at power 320W (**Method 2**). When the irradiation time was 2.20 min, we got a single product (**3a**) in 98% yield but different from pyranoquinoline **2a**. IR spectrum of **3a** showed absorption at 1680 cm^{-1} corresponding to free aldehyde group. The spectral ($^1\text{H-NMR}$ & Mass) and analytical data attested the compound **3a** to be 3-formylquinolin-2(1*H*)-one, which is also a good intermediate in the synthesis of other quinoline heterocycles (Rajendran and Vijayalakshmi 1994, 1998) (**Scheme 1**).

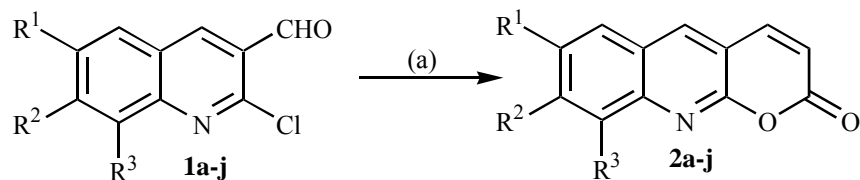
Then 2-chloro-3-formylquinoline (**1a**) irradiated with acetic acid alone in either closed or open vessel (**Table 1, Method 3**). The target product pyranoquinoline **2a** was obtained in lesser yield (67%). But, this method required more amount of acetic acid (175 mmol) and high irradiation power and time.

The solid-state condition was also checked for the reaction: Irradiation with only sodium acetate failed to give pyrano[2,3-*b*]quinolin-2-one but afforded 3-formylquinolin-2(1*H*)-one (**3a**) in 80% yield. This reaction also required high irradiation power and more time for complete conversion (**Table 1, Method 4**).

When method 1 was repeated in an open vessel, (**Table 1, Method 5**), a mixture of **2a** and **3a** was formed. Hence, with all these trials, we have found that, methods 1 and 2 are efficient ways to synthesize pyrano[2,3-*b*]quinolin-2-one (**2a**) and 3-formylquinolin-2(1*H*)-one (**3a**) respectively. To establish the generality and applicability of these methods, various substituted 2-chloro-3-formylquinolines (**1b-j**) were subjected to the same reaction conditions (**Methods 1 and 2**) to furnish the corresponding quinolines **2b-j** and **3b-j** in good yields (**Tables 2 and 3**).

All the yields were calculated from crystallized products and the products were identified by comparison of analytical data (mp, mmp, IR, NMR, and Mass) with those reported or with authentic samples prepared by the conventional methods (Meth-Cohn *et al.*, 1981). Some new derivatives (2e,g,h & 3e,g,h) were also prepared and reported.

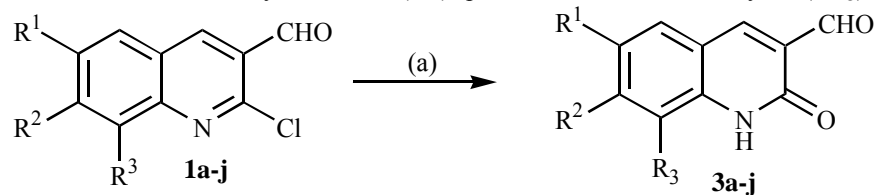
Table 2. Microwave Synthesis of 2-oxo-2H-pyrano[2,3-b]quinolines (2a-j)



(a) acetic acid (123 mmol), sodium acetate (50 mmol), mw, 320W

Entry	Product	R ¹	R ²	R ³	Time (min)	Yield (%)	mp (Lit.mp)°C
1	2a	H	H	H	5.00	92	243-244 (243)
2	2b	CH ₃	H	H	6.20	95	245-247 (240)
3	2c	H	CH ₃	H	6.50	81	273-275 (274)
4	2d	H	H	CH ₃	6.40	86	231-232 (230)
5	2e	OCH ₃	H	H	5.00	78	224-225
6	2f	H	OCH ₃	H	5.00	81	226-227 (225)
7	2g	H	H	OCH ₃	6.00	89	260-262
8	2h	Br	H	H	9.00	70	262-264
9	2i	Cl	H	H	8.20	75	210-212 (215)
10	2j	H	Cl	H	8.50	76	282-283 (280)

Table 3. Microwave Synthesis of 2(1H)-quinolinone-3-carboxaldehydes (3a-j)



(a) acetic acid (87.5 mmol), sodium acetate (25 mmol), mw, 320W

Entry	Product	R ¹	R ²	R ³	Time (min)	Yield (%)	mp (Lit.mp)°C
1	3a	H	H	H	2.20	98	304-305 (298)
2	3b	CH ₃	H	H	2.00	94	272-274 (275)
3	3c	H	CH ₃	H	2.00	93	296-297(294)
4	3d	H	H	CH ₃	1.80	96	285-287 (284)
5	3e	OCH ₃	H	H	1.50	98	275-276
6	3f	H	OCH ₃	H	1.70	97	265-267 (263)
7	3g	H	H	OCH ₃	1.50	97	260-262
8	3h	Br	H	H	2.50	72	>300 (342-344)
9	3i	Cl	H	H	2.20	87	>300 (357-358)
10	3j	H	Cl	H	2.50	75	>300 (340)

4. Conclusion

The procedures described above provide a useful, clean, fast and efficient alternative for the preparation of both pyrano[2,3-*b*]quinolin-2-ones and 3-formylquinolin-2(1*H*)-one. Prominent among the advantages of these new methods are operational simplicity, good yield in a very short reaction time, solvent-free conditions, very inexpensive, easily available reagent and catalyst and easy workup procedure employed.

Acknowledgement

The author VN is grateful to Director of Collegiate Education, Govt. of Tamilnadu, India, for financial support. Author thank to Research centre, Indian Institute of Science, Bangalore, INDIA for providing ¹HNMR spectral data.

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Received March 2011

Accepted July 2011

Final acceptance in revised form July 2011