A Green Approach for the Synthesis of 6H-chromeno[4,3-b] quinolin-6-one derivatives

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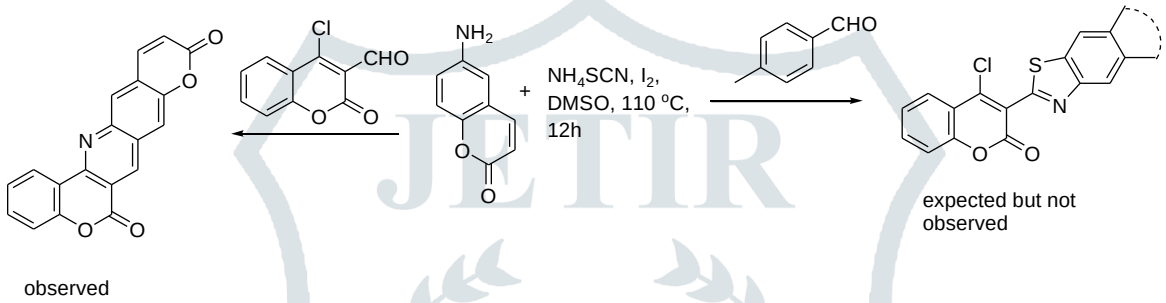
Abstract :A green one pot synthesis of 6H-chromeno[4,3-b] quinolin-6-one derivatives which are the main core of the natural product of Polyneomarline C using ammonium thiocyanate is described. Reaction of 4-chloro-3-formyl coumarin or different β-chloro α-β-unsaturated aldehyde and different aromatic amine or 4-amino and 6-amino coumarin in water-methanol solvent (2:1) at 60 ºC afforded the desired products. The advantages of this strategy are good yields, no chromatographic separation and free from oxidant, heavy metal catalyst and toxic by-products. The 4-chloro-3-formyl coumarin is obtained by VilsmeierHaack reaction of 4-hydroxy coumarin.



Keywords: 6H-Chromeno[4,3-b] quinolin-6-one, green synthesis, ammonium thiocyanate, 4-chloro-3-formyl coumarin, neat reaction, Polyalthianemoralis C.

I. Introduction

In last one or two decades, one-pot tandem chemical transformation without metal catalyst has widely been used for the synthesis of complex organic molecules. A variety of chemical conversions, like oxidation, reduction, substitution, condensation, etc. have been developed using this principle.[1] Aqueous reaction without using catalyst has many advantages such as decrease of wastes, less toxicity, maximum efficiency, decrease energy requirement of the reactions, designing bio-degradable products, economic and time factor. Hence, heterocyclic ring formation using this green protocol is an active and attractive field. Coumarin derivatives represent the core structure of many naturally occurring compounds with significant biological activities.[2] Lamellarinsand related pyrrole derived alkaloids isolated from diverse marine organisms are well known for their remarkable biological activities.[3] The coumarin derivatives fused with aza-heterocycles specially the pyridine nucleus have been reported to possess antiallergic,[4a]antidiabetic[4b]and analgesic[4c,d,e]properties. Santiagonamine is a naturally occuring pyridine fused coumarin derivatives found in the stems and branches of Berberisdarwinii Hook which is a shrub that is found in South America having wound-healing properties.[5] Goniothaline[6] is an another natural pyridocoumarin alkaloid isolated from the Australian rainforest plant Goniothalamusaustralis having antimalarial activity against a chloroquine-sensitive Plasmodium falciparum line (3D7). PolyneomarlineC[7] is also a natural 6H-chromeno[4,3-b]quinolin-6-onederivative isolated from the Polyalthianemoralis A. DC. used as Chinese herbal medicine. Coumarin fused pyridine[8] derivatives have been reported to possess anti-hypertensive activities, anti-HIV activity, androgen receptor antagonist activity, optoelectronic activity and can act as fluorescence dyes. All these observations establish the importance and bioactivity of pyridine fused coumarin derivatives. So, the synthesis of pyridine fused comarin is increasing trends of Organic Chemists. Many methods[9-10] to synthesize of these types of compounds have been developed by many group of scientists using different types of Lewis acids/Bases, metal catalysts with different solvents. The 6H-chromeno[4,3-b]quinolin-6-one skeleton constitutes the backbone of Polyneomarline C (Figure 1). We were interested to prepare some non-natural analogs of this type compounds by easy process. Many synthesis of 6H-chromeno[4,3-b]quinolin-6-one derivatives has been described in the literature[11-12] by some groups using different reagents, catalyst, solvents andultrasound irradiation. In our present work we have reported a green one pot synthesisof 6H-chromeno[4,3-b]quinolin-6-one derivatives which are the main core of the natural product of Polyneomarline C using ammonium thiocyanate by reaction of 4-chloro-3-formyl coumarin[13] and different aromatic amine in water-methanol solvent (2:1) at 60ºC and various aromatic amines. In this method there is no need for chromatographic separation.

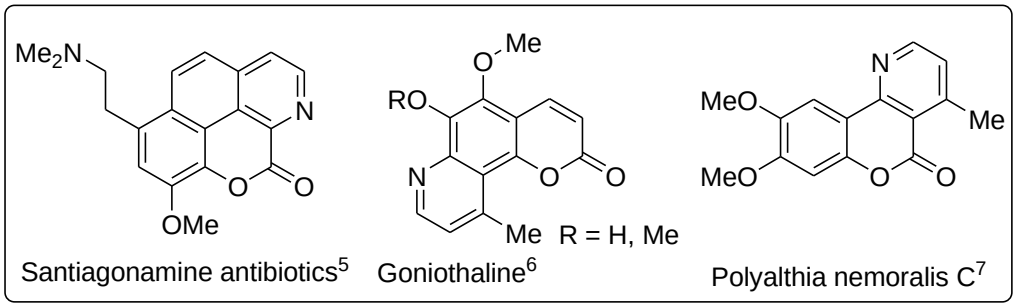


Figure 1: Naturalpyridocoumarin scaffolds

II. RESULTS AND DISCUSSION

Recently A. Hajra and his co-worker have developed a novel, simple method for the synthesis of 2-aylbenzothiazoles by I_2-mediated oxidative annulation of arylbenzaldehydes, aromatic amines,and ammonium thiocyanate as sulphur source. Applying the same methodolology, our target was to synthesize 2-coumarinyl benzothiazole starting from 4-chloro-3-formyl coumarin instead of aromatic aldehyde but we got 6H-chromeno[4,3-b]quinolin-6-one derivatives. So our first task was to find out an optimal condition to prepare the 6H-Chromeno[4,3-b]quinolin-6-onein best possible yield using different methodologies. Reaction between 4-chloro-3-formyl coumarin (1 equivalent) and 4-chloroaniline (1 or 2 equivalent) (**Scheme 1**) was studied under different conditions with different temperatures and times. But in all cases only pyridocoumarin derivatives were obtained but no 2-coumarinylbenzothiazoles was found. Results of the detailed studies are summarized in Table 1. It was found that reaction of **1** and **2a** inwater-methanol solvent (2:1) at 60°C in presence of ammonium thiocyanate proved to be the best condition and leads to the desired product in 98% yield within 2 hours. Here ammonium thiocyante acts as an acid catalyst not the source of sulphur. Thus treatment of 4-chloro 3-formyl coumarin (1equivalent) and different aromaticamines (1 equivalent) proceeded to give the corresponding 6H-Chromeno[4,3-b]quinolin-6-one derivatives under same condition (Table**2**). The reaction between Chloro-aldehyde of α-tetralone and 4-chloro aniline under similar condition did not give any cyclised product (scheme 1). The ^{1}H-NMR (400 MHz, DMSO-d_6) data is in conformity with the assigned structure for the 6H-Chromeno[4,3-b]quinolin-6-onederivatives **3d** and given bellow (Fig. 2) and HRMS data are also in agreement with the molecular formula of **3d**. In general, the reactions are very clean, without any side product with remarkable yields and donot require any chromatographic separation. Recrystallization from hot acetone provides analytically pure sample. The probable mechanism is not certain. Here pyridine ring instead of aylbenzothiazoles is formed due the presence of ortho chlorine atom of the substrate.Immine or enaminoimine is not formed due to high reactivity of 4-chloro-3-formylcoumarin as it contain electron withdrawing lactone as well as chlorine atom.

Table 1. Optimization studies in the selective formation ofpyrido[3,2-c]coumarinderivatives (3a)[a]

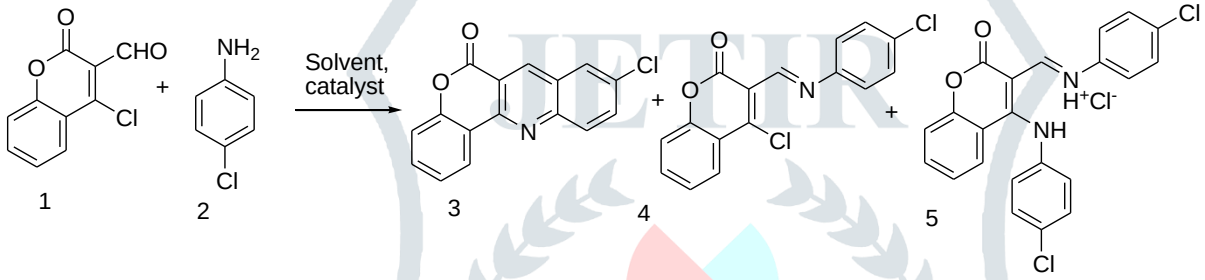
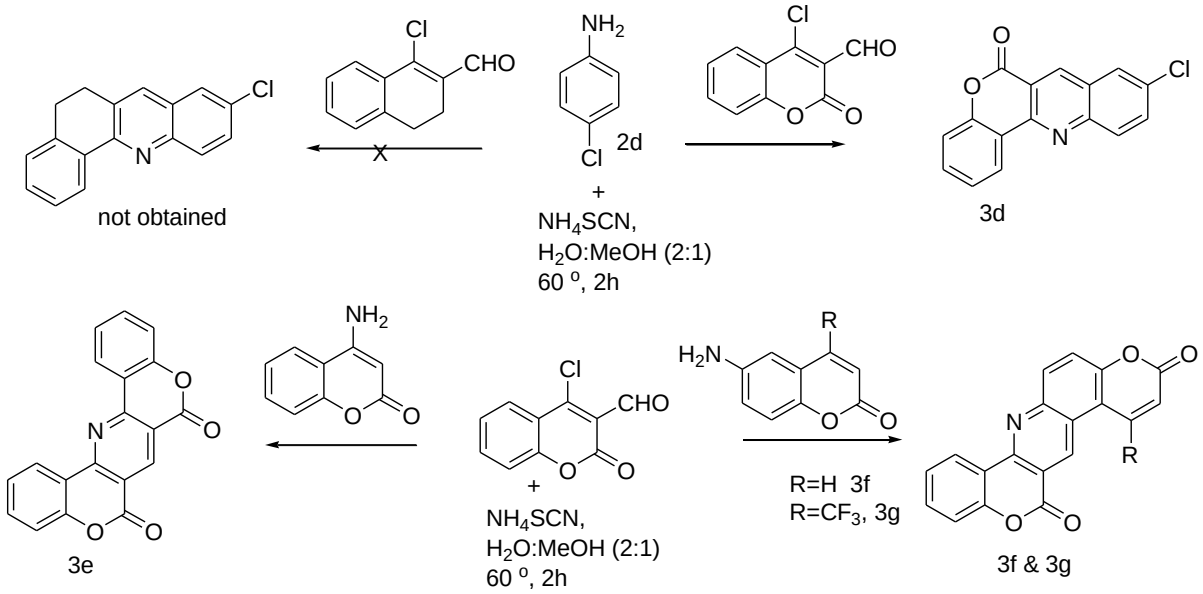


Table 1. Optimization studies in the selective formation of pyridocoumarin 3d

Entry	reactants	Solvent	additive	Temp (°C)	Time(h)	Product (3)	Product (4/5)[10b,c]
1	1a:1b	DMSO	NH_4SCN/I_2	110	12	95%	-
2	1a:1b	H_2O:MeOH	NH_4SCN/I_2	reflux	12	93%	-
3	1a:1b	H_2O:EtOH	NH_4SCN/I_2	reflux	12	50%	-
4	1a:1b	H_2O:MeOH	NH_4SCN	reflux	2	96%	-
5	**1a:1b**	**H_2O:MeOH**	**NH_4SCN**	**60**	**2**	**98%**	**-**
6	1a:1b	H_2O:MeOH	NH_4SCN	80	2	98%	-
7	1a:1b	H_2O:MeOH	-	reflux	12	trace	-

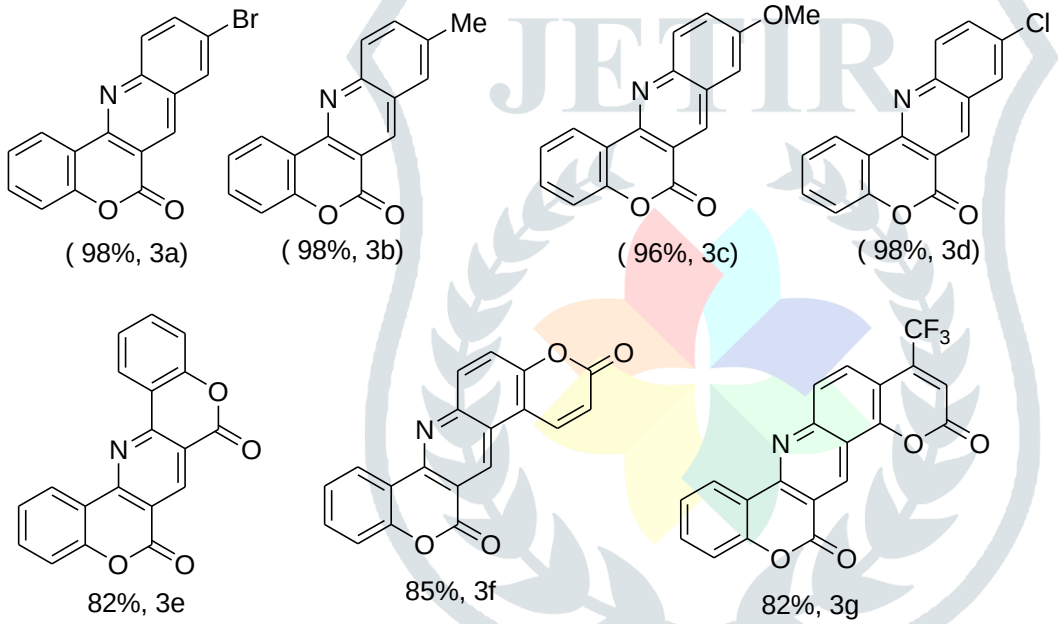
(a=2; b=3d in equivalent)

Conditions: Best optimum condition for the reaction is shown in bold entry



Scheme 1. Formation of the pyridocoumarin derivatives

Table 2. Substrate scope ofreaction for the synthesis of **3(a-g)**



Experimental:

Preparation of 6H-chromeno[4,3-b]quinolin-6-onederivatives 3 (a-g): General method.

Compound 1 (1.0 equiv.) and 2 (1.0 equiv.) were taken with 2mL MeOH, 4 mL H_2O and NH_4SCN (1.0 equiv.) in a round-bottom flask (25 ml) and it was placed in a heating oil bath. The heating of the resulting mixture was carried out up to 2 hours in open flask at 60 °C. The progress of the reaction was monitored by TLC. Excess methanol was distilled out, the pale yellow solid mass was filtered, washed with water and dried under vacuum to obtain the 6H-chromeno[4,3-b]quinolin-6-one **3(a-g)** in 82-98% yield.

9-Bromo-6H-chromeno[4,3-b]quinolin-6-one (3a)

White solid, yield, 98%; mp 260-261°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6) 7.49 (m, 2H),7.71 (t, 1H, J = 7.4 Hz), 8.13 (brs, 2H), 8.66 (d, 2H, J = 9.4 Hz), 9.35 (s, 1H) ppm;^{13}CNMR (100 MHz, DMSO-d_6): 110.1, 115.0, 117.4, 123.7, 124.4, 124.6, 127.9, 129.0, 129.3, 130.5, 132.9, 133.1, 141.3, 152.5, 154.1, 161.2; HRMS (ESI, 70 eV): m/z = 325.9821 (M^++H), 327.9791 (M^++H+2) [Calcd mass for $C_{16}H_9BrNO_2$: 325.9817 (M^++H), 327.9786 (M^++H+2)].

9-Methyl-6H-chromeno[4,3-b]quinolin-6-one[11c] (3b)

White solid, yield, 98%; mp 231-233°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6) 2.56 (s, 3H), 7.49 (t, 2H, J = 8.3 Hz), 7.69 (m, 1H), 7.88 (dd, 1H, J = 1.6 & 8.7 Hz), 8.08 (s 1H), 8.13 (d, 1H, J = 8.7 Hz), 8.66 (d, 1H, J = 8.0 Hz 1H), 9.25 (s, 1H) ppm;HRMS (ESI, 70 eV): m/z = 262.0874 (M^++H) [Calcd mass for $C_{17}H_{12}NO_2$: 262.0868 (M^++H)].

9-Methoxy-6H-chromeno[4,3-b]quinolin-6-one[11a,b](3c):

White solid, yield, 96%; mp 228-230°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6) 3.86 (s, 3H), 7.22 (m, 2H), 7.39-7.47 (m, 3H), 8.10 (d, 1H, J = 9.6 Hz), 8.70(m, 1H), 9.0 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): 56.4, 107.2, 107.3, 117.7, 120.0, 124.7, 125.4, 127.4, 129.0, 130.8, 132.6, 132.7, 139.4, 147.3, 152.5, 158.4, 161.1 ppm;HRMS (ESI, 70 eV): m/z = 278.0811 (M^++H)[Calcd mass for $C_{17}H_{12}NO_3$: 278.0817 (M^++H)]

9-Chloro-6H-chromeno[4,3-b]quinolin-6-one[11c] (3d)

White solid, yield, 95%; mp 243-245°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6) 7.51 (t, 2H, J = 8.0 Hz), 7.7 (t, 1H, J = 7.7 Hz), 8.03 (dd, 1H, J = 2.2 & 9.0 Hz),8.24 (d, 1H, J = 9.0 Hz), 8.50 (d, 1H, J = 2.2 Hz), 8.68 (d, 1H, J = 7.4 Hz), 9.33 (s, 1H) ppm; ^{13}C NMR (^{13}C NMR 100 MHz, DMSO-d_6)):117.7, 117.9, 121.8, 125.2, 125.6, 128.9, 130.2, 131.4, 133.4, 134.6, 134.7, 140.7, 150.4, 150.5, 153.3, 161.6; HRMS (ESI, 70 eV): m/z = 282.0329 (M^++H) [Calcd mass for $C_{16}H_9ClNO_2$: 282.0322 (M^++H)]

Compound(3f):
White solid, yield, 82%; mp 250-252°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6)7.29 (d, 1H, J = 7.7 Hz), 7.43 (d, 1H, J = 8.1 Hz), 7.65-7.74 (m, 3H, J = 7.8 Hz), 8.20 (d, 1H, J = 8.1 Hz), 8.69 (brs, 2H), 9.14 (s, 1H) ppm.
Compound(3g):
White solid, yield, 85%; mp 246-248°C (Acetone); ^{1}H NMR (400 MHz, DMSO-d_6)) 7.36 – 7.52 (m, 2H), 7.70 (m, 2H), 7.99-8.20 (m, 2H), 8.44 (d, 1H, J = 8.4 Hz), 8.63 (d, 1H, J = 8.4 Hz), 9.32 (s, 1H) ppm.
Compound (3g):
White solid, yield, 82%; mp237-239°C (Acetone)^{1}H NMR (400 MHz, DMSO-d_6) 7.40 (m, 2H), 7.73 (t, 1H, J = 7.5 Hz), 7.92 (t, 1H, J = 7.4 Hz), 8.02 (d, 1H, J = 8.5 Hz), 8.27 (d, 1H, J =8.1 Hz), 8.89 (s, 1H), 9.36 (s, 1H).

III. CONCLUSION:

In conclusion, we have developed an environmentally benign method for thesynthesis of 6H-Chromeno[4,3-b]quinolin-6-one derivatives backbone of natural product Polyneomarline C scaffolds in good to excellent yields in presence of ammonium thiocyanate at 60 °C in aqueous methanol solvent without chromatographic separation from 4-chloro-3-formylcoumarin and different substituent aromatic amine.The most noteworthy features of the present protocol are simplicity of procedure, high atom economy, cost effective and superior yields.

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