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An Expedient One-pot Approach for the Synthesis of 2-Amino-4*H*-chromenes Catalyzed by 3-Nitrophenylboronic Acid

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Abstract: 3-Nitrophenylboronic acid has been used as a green catalyst for the synthesis of 2-amino-4*H*-chromenes *via* a one-pot three component condensation reaction of aromatic aldehydes, substituted phenols and malononitrile in solvent ethanol. The mild reaction conditions, environmentally benign, easy experimental workup and excellent yields of desired products are some of the delightful features of the method which make it as an attractive and effective protocol.

Keywords: One-pot synthesis, 3-Nitrophenylboronic acid, 2-Amino-4*H*-chromenes, Phenols, Malononitrile, Multi-component reaction.

Introduction

2-Aminochromenes are the significant class of heterocyclic compounds being the main components of several natural products and are generally employed as cosmetic agents, food additives and potential biodegradable agrochemicals [1]. The 4*H*-chromene derivatives present the variety of pharmacological properties such as anti-cancer [2], anti-HIV [3], antitumor [4], anti-leukemic [5], antibacterial [6], anti-malarial [7] and anti-anaphylactic activities [8]. Moreover 2-aminochromenes are components of several natural products like calanolides, calanone, calophyllolides [9].

The synthesis of 2-aminochromenes involves a one-pot three-component coupling reaction of aromatic aldehyde, malononitrile and substituted phenol. Nowadays multi-component reactions (MCRs) provide significant advantages over normal linear step synthesis, in terms of easy work-up procedures and purification, short reaction time, energy and raw-material consumption. Thus, MCRs offer benefits in both economic and environment [10]. Several protocols for the synthesis of 2-amino-4*H*-chromenes have been reported [11-17]. Many homogeneous catalysts have been used for this condensation including ammonium salts [18], I_2/K_2CO_3 [19], $TiCl_4$ [20], $InCl_3$ [21],

methanesulfonic acid [22] and hetero-polyacid [23]. However, some of the reported methods require prolonged reaction time, reagents in stoichiometric amounts, toxic solvents and generate moderate recoveries in the final product.

Nowadays in synthetic organic chemistry, arylboronic acid has been successfully used in a variety of organic transformations as an efficient green Lewis acid catalyst [24-26]. Arylboronic acids are mostly crystalline solids, non-toxic, easily handled and stable in air and moisture which point out that they are of relatively low toxicity and have low environmental impact. Moreover the arylboronic acids having electron-withdrawing substituent on aromatic ring used as an effective Lewis acid catalyst in organic synthesis due to its enhanced acidity [27-29]. Thus, due to environmentally benign impact of arylboronic acids, herein we report successful method for synthesis of 2-amino-4*H*-chromenes in excellent yields under ambient temperature condition using 3-nitrophenylboronic acid as catalyst.

Experimental

All solvents were used as commercial anhydrous grade without further purification. The column chromatography was carried out over silica gel (80-120 mesh). Melting points were determined in open capillary tube and are uncorrected. ^{1}H spectra were recorded on a Bruker 300 MHz spectrometer in $CDCl_3$ solvent and TMS as an internal standard. ^{13}C NMR spectra were recorded on a Bruker-300 MHz spectrometer in $CDCl_3$ solvent. Mass spectra were taken on Polaris-Q Thermoscintific GC-MS.

General procedure for synthesis of 2-amino-4*H*-chromenes: A mixture of aromatic aldehyde (2.0 mmol), malononitrile (2.0 mmol) and substituted phenols (2.0 mmol) and 3-nitrophenylboronic acid (20 mol %) as catalyst was added in the solvent ethanol (10 mL) and the reaction mixture was stirred at room temperature for appropriate time (Table 1 and 2). After completion of reaction as indicated by thin layer chromatography (pet ether: ethyl acetate 8:2), ethanol was evaporated under reduced pressure and the residue was extracted with ethyl acetate (3×10 mL). The organic layer was washed with water, dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The crude obtained product was purified by column chromatography using silica gel mesh 80-120 to afford the pure product.

2-amino-4-(4-chlorophenyl)-4*H*-chromene-3-carbonitrile (4b): ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.82 (s, 1H); 6.19 (s, 2H, NH_2), 6.91-7.04 (m, 2H) 7.31-7.42 (m, 2H), 7.75-7.95 (m, 4H); ^{13}C-NMR (300 MHz, $CDCl_3$): δ 35.2, 55.4, 110.2, 115.8, 118.0, 121.2, 124.0, 126.9, 129.0, 132.1, 136.4, 142.6, 151.2, 166.4; GC-MS (m/z): 282 (M+); Elem. Anal for $C_{16}H_{11}ClN_2O$: C, 67.97; H, 3.92; N, 9.91; Found C, 67.94; H, 3.98; N, 9.89.

2-amino-6-chloro-4-(4-chlorophenyl)-4*H*-chromene-3-carbo-nitrile (4f): ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.74 (s, 1H); 6.37(s, 2H, NH_2), 6.90-7.09 (m, 3H), 7.38-7.60 (m, 4H). ^{13}C-NMR (300 MHz, $CDCl_3$): δ 30.5, 54.8, 112.4, 115.4, 117.0, 121.0, 126.2, 128.4, 131.7, 135.0, 138.2, 143.2, 153.6, 163.5; GC-MS (m/z): 316 (M+); Elem. Anal for $C_{16}H_{10}Cl_2N_2O$: C, 60.59; H, 3.18; N, 8.83; Found C, 60.55; H, 3.21; N, 8.80.

2-amino-4-(4-chlorophenyl)-7-hydroxy-4*H*-chromene-3-carbonitrile (4j): ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.60 (s, 1H); 6.71 (s, 2H, NH_2), 6.98-7.11 (m, 2H), 7.38 (d, 1H, J = 7.2 Hz), 7.54-7.81 (m, 4H), 9.61 (s, 1H, OH); ^{13}C-NMR (300 MHz, $CDCl_3$): δ 37.8, 55.0, 110.2, 115.2, 120.5, 122.4, 126.4, 128.6, 132.1, 136.3, 141.8, 144.7, 151.6, 163.1; GC-MS (m/z): 298(M+); Elem. Anal for $C_{16}H_{11}ClN_2O_2$: C, 64.33; H, 3.71; N, 9.38; Found C, 64.30; H, 3.75; N, 9.41.

2-amino-4-(4-chlorophenyl)-4*H*-benzochromene-3-carbonitrile (5b) : ^{1}H NMR (300 MHz, $CDCl_3$): δ δ 5.02 (s, 1H), 6.89 (s, 2H, NH_2), 7.08 (d, 1H, J = 8.2Hz), 7.28-7.40 (m, 4H), 7.49-7.54 (m, 3H), 7.80 (d, 1H, *J* = 8.0 Hz), 8.18 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR (300 MHz, $CDCl_3$): δ 27.2, 54.8, 115.2, 117.8, 120.1, 122.8, 124.0, 125.8, 127.1, 128.0, 128.9, 129.8, 131.2, 132.7, 135.9, 142.2, 147.5, 166.6; GC-MS, *m*/*z*: 332 (M+); Elemental Analysis: Anal. Calcd for $C_{20}H_{13}ClN_2O$: C, 72.18; H, 3.94; N, 8.42; Found C, 72.22; H, 3.96; N, 8.46.

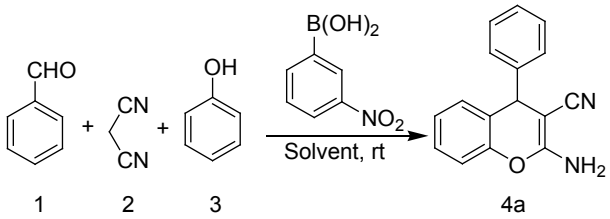
2-amino-4-(4-methoxyphenyl)-4*H*-benzochromene-3-carbonitrile (5c): ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.76 (s, 3H), 4.88 (s, 1H), 6.92 (s, 2H, NH_2), 7.02-7.15 (m, 4H), 7.38-7.57 (m, 4H), 7.82 (d, 1H, *J* = 8.2 Hz), 8.12 (d, 1H, *J* = 8.2 Hz); ^{13}C NMR (300 MHz, $CDCl_3$): δ 30.6, 52.8, 62.4, 111.4, 115.8, 117.2, 119.6, 122.0, 123.8, 125.1, 126.5, 127.8, 129.8, 132.0, 134.1, 139.4, 146.2, 149.1, 159.3; GC-MS, *m*/*z*: 328 (M+); Elemental Analysis: Anal. Calcd for $C_{21}H_{16}N_2O_2$: C, 76.81; H, 4.91; N, 8.53; Found C, 76.84; H, 4.86; N, 8.56.

3-amino-1-(4-nitrophenyl)-1*H*-benzochromene-2-carbonitrile (5k): ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.46 (s, 1H), 7.05 (s, 2H, NH_2), 7.22 (d, 1H, *J* = 8.0), 7.40-7.69 (m, 4H), 7.82 (d, 1H, *J* = 9.0 Hz), 7.92-7.98 (m, 2H), 8.13 (m, 2H); ^{13}C NMR (300 MHz, $CDCl_3$): δ 30.6, 56.8, 110.5, 115.4, 118.8, 121.0, 123.9, 124.6, 125.8, 127.8, 129.3, 130.9, 131.9, 134.6, 146.4, 149.3, 152.6, 162.6; GC-MS, *m*/*z*: 343 (M+); Elemental Analysis: Anal. Calcd for $C_{20}H_{13}N_3O_3$: C, 69.96; H, 3.82; N, 12.24; Found C, 69.90; H, 3.78; N, 12.20.

3-amino-1-(4-fluorophenyl)-1*H*-benzochromene-2-carbonitrile (5l): ^{1}H NMR (300 MHz, $CDCl_3$): 5.38 (s, 1H), 7.18 (s, 2H, NH_2), 7.27 (d, 1H, *J* = 8.8 Hz), 7.39 (d, 1H, *J* = 8.8 Hz), 7.50-7.65 (m, 4H), 7.75-7.82 (m, 2H), 7.95-8.04 (m, 2H); ^{13}C NMR (300 MHz, $CDCl_3$): δ 32.8, 62.5, 112.8, 115.8, 116.4, 118.0, 121.8, 124.2, 126.7, 128.0, 128.6, 129.0, 131.8, 134.2, 137.4, 143.4, 152.4, 165.2; GC-MS, *m*/*z*: 316 (M+); Elemental Analysis: Anal. Calcd for $C_{20}H_{13}FN_2O$: C, 75.94; H, 4.14; N, 8.86; Found C, 75.90; H, 4.18; N, 8.80.

Results and Discussion

In continuation of our interest in synthesis of heterocycles [30-33], herein we describe the synthesis of 2-amino-4*H*-chromenes in presence of catalytic amount of 3-nitrophenylboronic acid. The synthesis of 2-amino-4*H*-chromenes was accomplished by a one-pot three-component reaction of aromatic aldehydes, malononitrile and substituted phenols using 3-nitrophenylboronic acid as catalyst in solvent ethanol under ambient temperature. Initially, we focused on optimization of suitable solvent and catalyst loading for the model reaction of benzaldehyde, malononitrile and phenol under ambient temperature condition (**Scheme1**).



Scheme 1: One-pot Synthesis of 2-amino-4-phenyl-4*H*-chromene-3-carbonitrile.

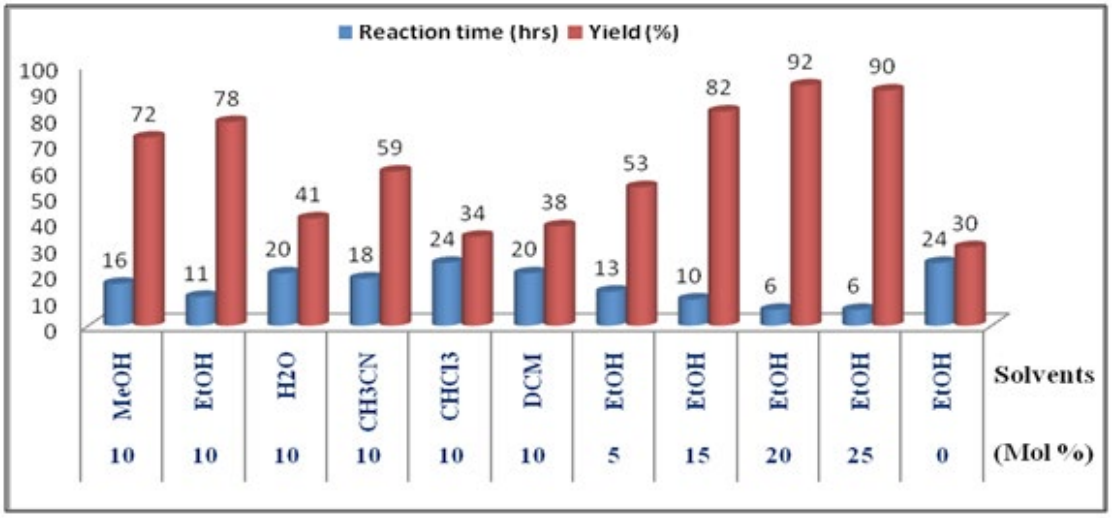
Various solvents were screened to test the efficiency of 3-nitrophenylboronic acid (10 mol%), as shown in **Figure 1**, it is noteworthy to reveal that the polar-protic solvents methanol and ethanol afforded enhanced yield than the non-polar solvents and the pleasing result was observed in the solvent ethanol. The catalyst worked most efficiently by phasing out of the desired product. Furthermore the reaction in solvents water and acetonitrile afforded 41% and 59% yield respectively. In the non-polar solvent chloroform and dichloromethane

desired product was obtained in inferior yields with extended reaction time. As a result we continued our studies in the solvent ethanol.

Subsequently, we studied the influence of catalyst loading on the model reaction. It was observed that amount of the catalyst plays a major role in determining the desired product yield. On diminishing catalyst loading of 3-nitrophenylboronic acid to 5 mol%, reaction afforded lower yield 53% with elongated reaction time. When catalyst loading was enhanced to 15 mol%, an improved result was obtained. The reaction was completed within 10 hours and offered 82% yield of the desired product. The best optimized reaction condition was accomplished at the catalyst loading of 3-nitrophenylboronic acid to 20 mol% in ethanol. The model reaction offered product 4a in excellent yield 92% within 6 hours. More increase in the catalyst loading to 25 mol% did not show an improvement in the product yield or reaction time. The model reaction in absence of catalyst in solvent ethanol showed reduced performance with respect to the yield and reaction time. When the reaction did stirred for 24 hours, it offered 30% product yield.

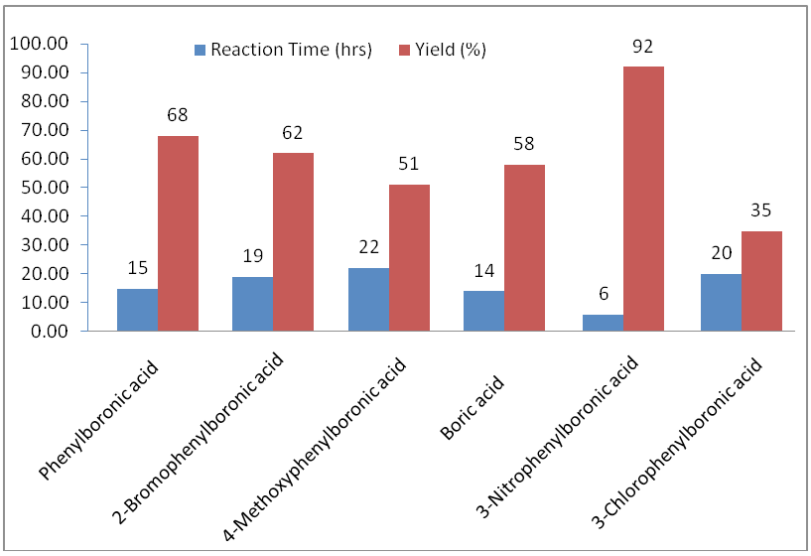
Furthermore, to perceive importance of 3-nitrophenylboronic acid, the effect of other organoborons as the catalyst (20 mol%) were screened for the present reaction in solvent ethanol at room temperature condition. The results are presented in **Figure 2**. When the reaction was carried out in presence of phenylboronic acid, it led to the desired product 4a in 68% yield in 15 hours. With the catalysts 2-bromophenylboronic acid and 4-methoxyphenylboronic acid reaction was sluggish and afforded 62% and 51% yield respectively. The catalyst boric acid and 3-chlorophenylboronic acid gave 58% and 35% yield correspondingly.

To study the scope of the reaction after the optimization of reaction condition, we have examined a wide range of aromatic aldehydes with substituted phenols and malononitrile to obtain the corresponding 2-amino-4*H*-chromenes in excellent yields (Table 1, entry



[a]Conditions: Benzaldehyde (2 mmol), Malononitrile (2 mmol), phenol (2 mmol), 3-Nitrophenylboronic acid (mol %), Solvent (10 mL) at room temperature. Reaction was monitored by thin layer chromatography.
[b]Isolated yield

Figure 1. Optimization of the solvent and catalyst loading for the synthesis of 2-amino-4*H*-chromenes[a,b]

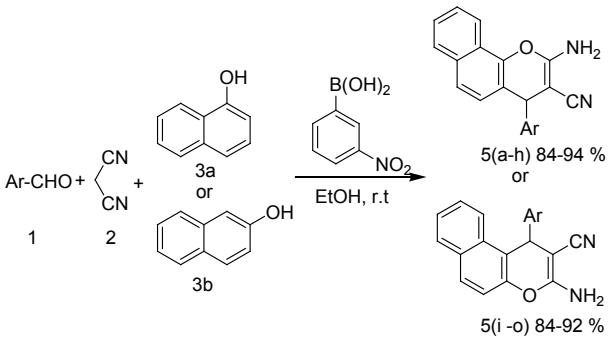


[a]Conditions: Benzaldehyde (2 mmol), Malononitrile (2 mmol), phenol (2 mmol), Catalyst (20 mol %), EtOH (10 mL) at room temperature. Reaction was monitored by thin layer chromatography.
[b]Isolated yield

Figure 2. Optimization of the catalyst for the one-pot synthesis of 2-amino-4*H*-chromenes.[a,b]

1-12).

Furthermore we expanded the scope of our protocol by using various aromatic aldehydes with α or β-naphthol and malononitrile to obtain the corresponding 2-amino-4*H*-chromenes in excellent yields (**Scheme 2).** The results obtained are listed in Table 2 **(Entry 1-15).**



Scheme 2: One-pot three component synthesis of 2-amino-benzochromenes

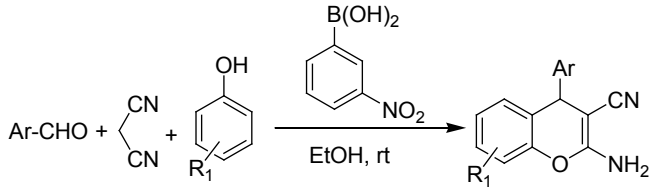
The reaction proceeded smoothly with aromatic aldehyde having electron-withdrawing or electron-releasing substituents. It was remarked that the aromatic aldehydes having electron-withdrawing substituents reacted rapidly and offered high yield of the desired product as compared to those having electron-donating substituents.

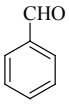
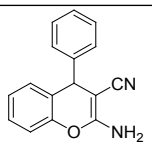
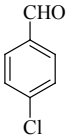
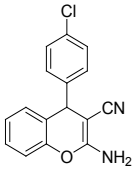
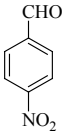
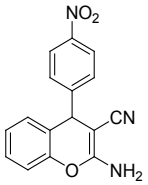
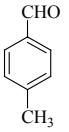
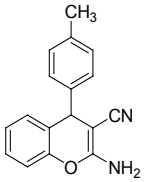
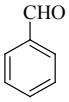
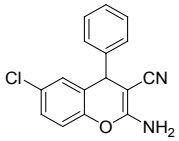
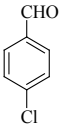
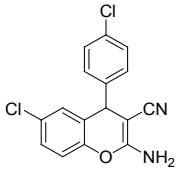
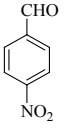
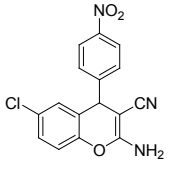
Conclusion

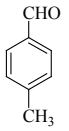
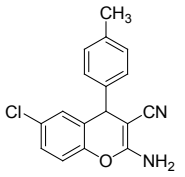
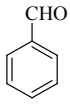
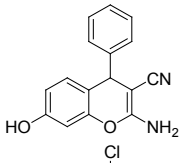
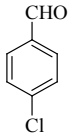
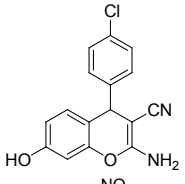
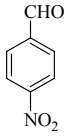
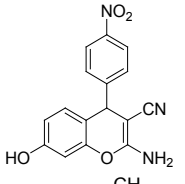
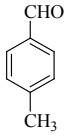
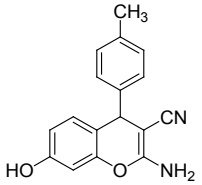
In conclusion, we have demonstrated a convenient and green one-pot protocol for the synthesis of 2-amino-4*H*-chromenes by the condensation of aromatic aldehydes, substituted phenol and malononitrile in presence of 3-nitrophenylboronic acid as catalyst under ambient temperature condition. This modified protocol offers enhanced performance over the many conventional methods. The delightful features of this protocol are easy to accomplish, use of environmentally benign reaction solvents and catalyst, mild reaction condition, cost efficiency and excellent yields of 2-amino-4*H*-chromene derivatives.

Acknowledgements

Table 1: 3-Nitrophenylboronic acid catalyzed one-pot synthesis of 2-amino-4*H*-chromenes[a].

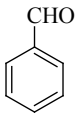
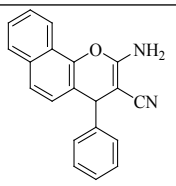
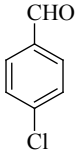
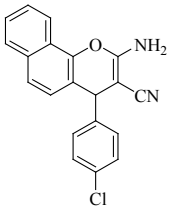
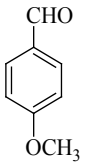
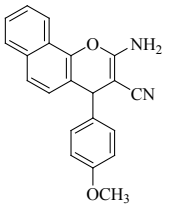


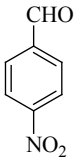
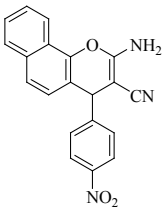
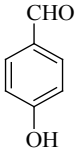
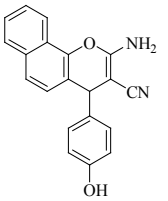
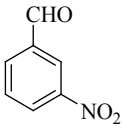
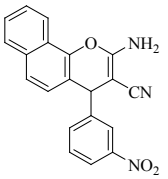
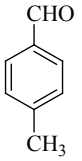
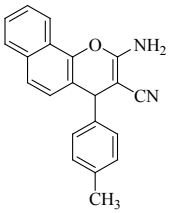
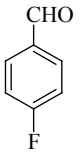
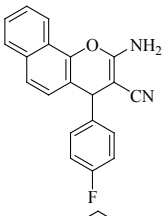
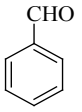
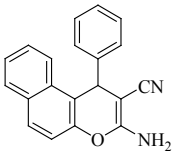
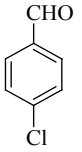
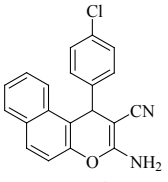
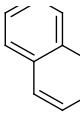
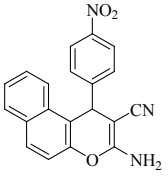
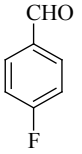
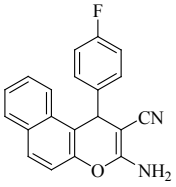
Entry	Aldehyde	R_1	Products	Time (h)	Mp. (°C)	Yield (%)[b]
1		H	 4a	6.00	150-152	92
2		H	 4b	7.00	175-177	88
3		H	 4c	5.00	214-216	91
4		H	 4d	6.00	166-168	88
5		4-Cl	 4e	7.00	170-172	85
6		4-Cl	 4f	6.00	193-195	88
7		4-Cl	 4g	5.30	153-155	92

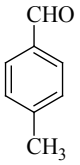
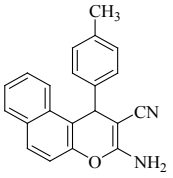
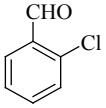
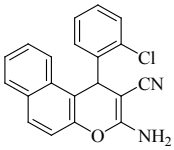
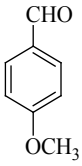
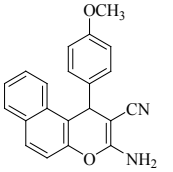
8		4-Cl	 4h	7.30	220-222	88
9		3-OH	 4i	7.00	232-234	87
10		3-OH	 4j	7.00	207-209	90
11		3-OH	 4k	6.00	161-163	93
12		3-OH	 4l	8.00	183-185	89

[a]Conditions: Aromatic Aldehydes (2 mmol), Malononitrile (2 mmol), Phenols (2 mmol), 3-Nitrophenylboronic acid (20 mol %), Ethanol (10 mL), rt. Reaction was monitored by thin layer chromatography. [b]Isolated yield

Table 2: 3-Nitrophenylboronic acid catalyzed one-pot synthesis of 2-amino 4*H*-chromenes[a]

Entry	Aldehyde	Phenol (3a/3b)	Products	Time (h)	Mp. (^{o}C)	Yield (%)[b]
1		α-naphthol	 5a	7.00	208-210	89
2		α-naphthol	 5b	6.00	231-233	88
3		α-naphthol	 5c	6.30	115-116	85

4		α-naphthol	 5d	5.30	237-238	94
5		α-naphthol	 5e	7.00	185-187	88
6.		α-naphthol	 5f	6.00	210-212	89
7		α-naphthol	 5g	6.30	204-206	84
8		α-naphthol	 5h	5.00	235-237	91
9		β-naphthol	 5i	6.00	281-283	86
10		β-naphthol	 5j	6.00	205-207	88
11		β-naphthol	 5k	5.30	184-186	90
12		β-naphthol	 5l	6.00	229-231	92

13		β-naphthol	 5m	6.30	269-270	87
14		β-naphthol	 5n	7.00	257-259	86
15		β-naphthol	 5o	7.00	116-118	84

[a]Conditions: Aroamtic aldehyde (2 mmol), Malononitrile (2 mmol), α or β-Naphthol (2 mmol), 3-Nitrophenylboronic acid (20 mol%), Ethanol (10 mL) at room temperature. Reaction was monitored by thin layer chromatography. [b]Isolated yield

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