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# One-Pot Synthesis of 7-Alkyl-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-ones in Presence of Alum

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A simple, green and efficient solvent-free procedure for the synthesis of 7-alkyl-6*H*,7*H*-naphtho[1',2': 5,6]pyrano[3,2-c]chromen-6-ones, from aldehydes, β-naphthol and 4-hydroxycoumarin in presence of alum at 120 ºC was described.

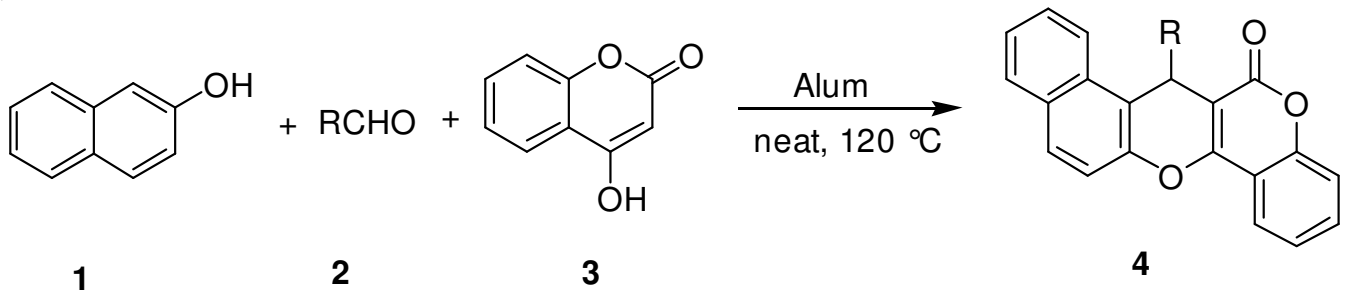
**Key Words: Naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one; 4-Hydroxycoumarin; Alum, Solvent-free.**

## INTRODUCTION

The rapid assembly of molecular diversity utilizing multi-component reaction (MCRs) has received a great deal of attention, most notably for the construction of heterocyclic drug-like libraries[1]. These methodologies have great utility, particularly, when they lead to the formation of privileged medicinal heterocyclic compounds. In the past few decades, the synthesis of new heterocyclic compounds has been a subject of great interest due to their wide applicability. Heterocyclic compounds occur widely in nature and are essential to life. Among a large variety of heterocyclic compounds, heterocycles containing chromene moiety are of interest since these compounds have shown interesting biological properties such as antioxidant[2], antibacterial[3], antirhinovirus[4], cytotoxic[5], anticancer[6], antimicrobial[7], antihypertensive activities[8]. Therefore, a number of methods have been reported for the synthesis of chromene derivatives[9]. Despite the available methods, the development of new synthetic methods for the efficient preparation of heterocycles containing chromene ring fragment is therefore an interesting challenge. Recently, alum [$KAl(SO_4)_2 \cdot 12H_2O$], which is relatively non-toxic and inexpensive catalyst, has emerged as an efficient alternative catalyst for a variety of prominent organic reactions such as Biginelli[10], Pechmann reaction[11] and also used for the synthesis of 1,8-dioxo-octahydroxanthenes[12], isoquinolonic acids[13], trisubstituted imidazoles[14], 1'*H*-spiro[isoindoline-1,2'-quinazoline]-3,4'(3'*H*)-diones[15], 1,3,4-oxadiazoles[16] and 1,5-benzodiazepines[17]. In this paper, we wish to report a rapid and highly efficient method for synthesis of 7-alkyl-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-ones in the presence of alum under solvent-free conditions (**Scheme-I**).

## EXPERIMENTAL

NMR spectra were determined on Bruker AV-400 spectrometer at room temperature using TMS as internal standard, coupling constants (*J*) were measured in Hz; Elemental analysis




1 2 3 4

**Scheme-I**

were performed by a Vario-III elemental analyzer; Melting points were determined on a XT-4 binocular microscope and were uncorrected. Commercially available reagents were used throughout without further purification unless otherwise stated.

**General procedure for the preparation of 7-alkyl-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-ones (4):** A mixture of β-naphthol (1 mmol), aldehyde (1 mmol), 4-hydroxyco-umarin (1 mmol) and alum (0.1 mmol) was heated at 120 ºC for an appropriate time. After completion of reaction, as indicated by TLC, the reaction mixture was cooled to room temperature and the contents were poured into ice-cold water and stirred for 5 min. The products were collected by filtration, washed with water and then recrystallized from ethanol to afford the chromene derivative. The catalyst in the aqueous phase could be recovered by removing the water under vacuum then washing with acetone and drying at room temperature.

**7-Phenyl-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4a):** White powder, m.p. 281-282 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.11 (d, 1H, *J* = 7.6 Hz), 8.00 (d, 1H, *J* = 8.4 Hz), 7.90-7.84 (m, 2H), 7.60-7.34 (m, 8H), 7.22 (t, 2H, *J* = 7.6 Hz), 7.14-7.11 (m, 1H), 5.89 (s, 1H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ: 173.2, 160.1, 153.5, 152.7, 146.9, 141.2, 135.9, 132.9, 130.6, 130.5, 128.5, 124.7, 122.3, 117.2, 115.5, 115.3, 113.3, 106.1, 34.3; Anal. calcd for $C_{26}H_{16}O_3$: C 82.96, H 4.28; found: C 83.06, H 4.19.

**7-(4-Chlorophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4b):** White powder, m.p. 267-268 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.20 (d, 1H, *J* = 7.6 Hz), 7.97 (d, 1H, *J* = 8.0 Hz), 7.91-7.86 (m, 2H), 7.69-7.40 (m, 8H), 7.18 (d, 2H, *J* = 8.4 Hz), 6.11 (s, 1H); $^{13}$C NMR (DMSO-$d_6$, 100 MHz) δ: 174.6, 160.7, 154.8, 152.5, 147.0, 142.9, 133.3, 131.9, 130.8, 130.7, 130.4, 129.1, 128.8, 127.9, 125.9, 125.2, 123.9, 132.2, 117.7, 117.0, 116.2, 114.1, 104.5, 35.8; Anal. calcd for $C_{26}H_{15}O_3Cl$: C 76.01, H 3.68; found: C 76.12, H 3.70.

**7-(4-Fluorophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4c):** White powder, m.p. 253-254 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.20 (d, 1H, *J* = 7.6 Hz), 7.98 (d, 1H, *J* = 8.0 Hz), 7.87 (t, 2H, *J* = 9.6 Hz), 7.67 (t, 1H, *J* = 8.0 Hz), 7.50-7.39 (m, 7H), 6.90 (t, 2H, *J* = 8.4 Hz), 6.11 (s, 1H); $^{13}$C NMR (DMSO-$d_6$, 100 MHz) δ: 173.5, 165.6, 156.2, 153.0, 147.4, 139.5, 133.5, 131.8, 130.9, 130.0, 129.9, 129.7, 128.6, 127.5, 126.0, 125.5, 125.3, 123.9, 123.3, 117.4, 117.3, 116.5, 115.3, 115.1, 100.0, 35.5; Anal. calcd for $C_{26}H_{15}O_3F$: C 79.18, H 3.83; found: C 79.23, H 3.79.

**7-(4-Methylphenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4d):** White powder, m.p. 230-231 ºC; $^1$H NMR (DMSO-$d_6$, 400 MHz) δ: 8.17-8.14 (m, 1H), 8.06-8.01 (m, 2H), 7.90 (d, 1H, *J* = 7.6 Hz), 7.72-7.68 (m, 2H), 7.54-7.45 (m, 4H), 7.26 (d, 2H, *J* = 8.0 Hz), 7.01 (d, 2H, *J* = 8.0 Hz), 5.71 (s, 1H), 2.14 (s, 3H); $^{13}$C NMR (DMSO-$d_6$, 100 MHz) δ: 172.9, 160.7, 154.5, 152.4, 147.0, 141.1, 136.4, 133.1, 131.8, 130.8, 130.1, 129.4, 129.1, 128.8, 127.8, 125.8, 125.1, 123.9, 123.1, 117.6, 117.0, 116.8, 114.1, 105.1, 35.9, 20.9; Anal. calcd for $C_{27}H_{18}O_3$: C 83.06, H 4.65; found: C 83.11, H 4.59.

**7-(4-Nitrophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4e)**. White powder, m.p. 257-258 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.18 (d, 1H, *J* = 6.4 Hz), 8.07 (d, 2H, *J* = 8.8 Hz), 7.94-7.87 (m, 3H), 7.71-7.64 (m, 3H), 7.53-7.41 (m, 5H), 6.22 (s, 1H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ: 176.8, 160.1, 153.0, 150.7, 147.4, 146.6, 133.7, 131.9, 130.7, 130.4, 129.4, 128.8, 127.8, 125.9, 125.8, 125.6, 123.7, 123.5, 123.1, 117.4, 116.6, 116.0, 99.0, 36.3; Anal. calcd for $C_{26}H_{15}NO_5$: C 74.10, H 3.59; found: C 74.26, H 3.49.

**7-(3-Nitrophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4f):** White powder, m.p. 249-250 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.17-8.12 (m, 2H), 8.01-7.85 (m, 5H), 7.63-7.58 (m, 2H), 7.52-7.35 (m, 5H), 5.97 (s, 1H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ: 176.2, 161.4, 155.4, 152.7, 148.5, 147.3, 145.1, 135.0, 132.5, 131.9, 130.6, 130.4, 129.3, 128.8, 127.7, 125.6, 124.4, 123.4, 123.1, 122.9, 122.2, 117.1, 116.8, 115.0, 114.1, 103.8, 36.3; Anal. calcd for $C_{26}H_{15}NO_5$: C 74.10, H 3.59; found: C 74.02, H 3.51.

**7-(2,4-Dichlorophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4g):** White powder, m.p. 267-268 ºC; $^1$H NMR (DMSO-$d_6$, 400 MHz) δ: 8.18 (d, 1H, *J* = 7.2 Hz), 8.04 (d, 2H, *J* = 8.0 Hz), 7.97 (d, 1H, *J* = 7.2 Hz), 7.73-7.67 (m, 2H), 7.52-7.48 (m, 6H), 7.26 (m, 1H), 6.02 (s, 1H); *13*C NMR ($CDCl_3$, 100 MHz) δ: 175.5, 161.6, 155.0, 152.6, 147.3, 143.2, 132.0, 131.8, 131.0, 129.5, 128.8, 128.6, 128.5, 128.4, 127.4, 126.9, 125.3, 124.2, 123.6, 122.7, 116.9, 116.7, 116.6, 114.5, 105.2, 36.4; Anal. calcd for $C_{26}H_{14}O_3Cl_2$: C 70.13, H 3.17; found: C 70.20, H 3.12.

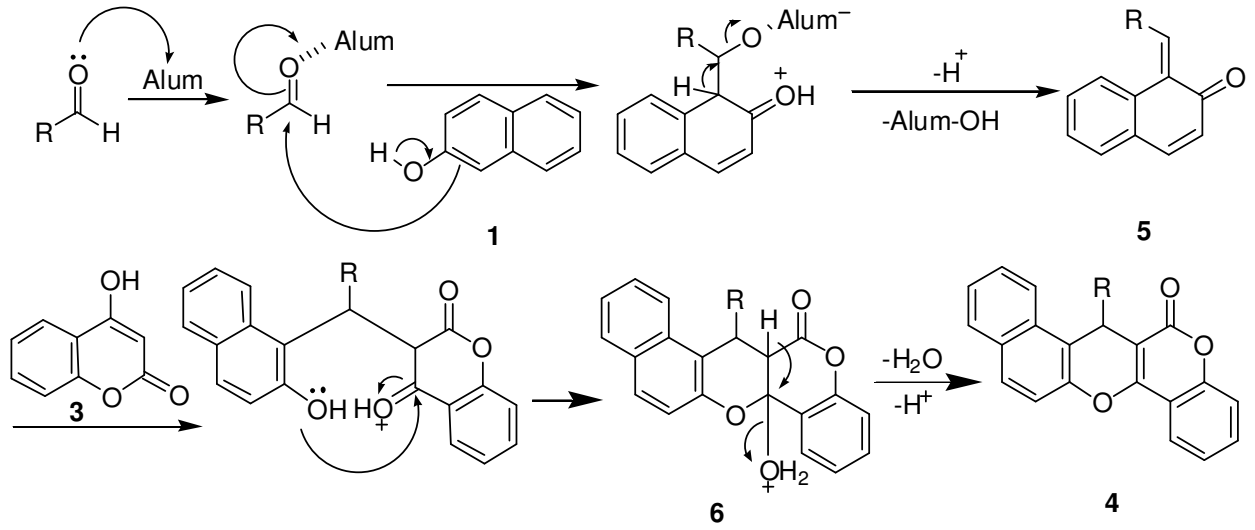
**7-(3,4-Dichlorophenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4h):** White powder, m.p. 256-257 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.20 (d, 1H, *J* = 7.6 Hz), 7.93-7.86 (m, 3H), 7.70-7.66 (m, 1H), 7.55-7.36 (m, 7H), 7.29 (m, 1H), 6.07 (s, 1H); $^{13}$C NMR ($CDCl^3$, 100 MHz) δ: 173.5, 161.5, 155.3, 152.7, 147.3, 143.2, 132.6, 132.4, 131.8, 131.1, 130.8, 130.4, 130.3, 130.1, 128.7, 128.2, 127.7, 125.6, 124.3, 123.3, 122.8, 116.9, 116.8, 115.3, 114.2, 104.1, 35.8; Anal. calcd for $C_{26}H_{14}O_3Cl_2$: C 70.13, H 3.17; found: C 70.10, H 3.24.

**7-(4-Methoxyphenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4i):** White powder, m.p. 214-215 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.19 (d, 1H, *J* = 8.0 Hz), 8.02 (d, 1H, *J* = 8.4 Hz), 7.86-7.82 (m, 2H), 7.66-7.62 (m, 1H), 7.50-7.37 (m, 7H), 6.73 (m, 2H), 6.07 (s, 1H), 3.68 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ: 177.0, 160.0, 158.2, 153.0, 147.3, 136.1, 133.3, 132.0, 131.1, 129.5, 129.4, 128.5, 127.4, 126.0, 125.4, 125.2, 124.1, 123.4, 117.8, 117.3, 116.5, 113.8, 100.4, 55.1, 35.3; Anal. calcd for $C_{27}H_{18}O_4$: C 79.79, H 4.46; found: C 79.85, H 4.40.

**7-(2,5-Dimethoxyphenyl)-6*H*,7*H*-naphtho[1',2':5,6]pyrano[3,2-c]chromen-6-one (4j):**. White powder, m.p. 191-192 ºC; $^1$H NMR ($CDCl_3$, 400 MHz) δ: 8.29 (d, 1H, *J* = 8.4 Hz), 8.11 (d, 1H, *J* = 7.6 Hz), 7.81-7.79 (m, 2H), 7.58-7.32 (m, 7H), 6.87-6.78 (m, 2H), 6.63-6.60 (m, 1H), 6.13 (s, 1H), 3.89 (s, 3H), 3.68 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ: 174.5, 161.4, 155.4, 153.7, 151.3, 147.1, 137.5, 133.3, 131.8, 131.5, 129.1, 128.3, 127.4, 125.1, 124.0, 123.9, 122.7, 117.4, 117.2, 116.9, 115.5, 114.5, 113.0, 112.2, 56.9, 55.5, 31.2; Anal. calcd for $C_{28}H_{20}O_5$: C 77.05, H 4.62; found: C 77.10, H 4.58.

## RESULTS AND DISCUSSION

Initially, to choose the optimum conditions, first, the effect of solvent on the rate of the reaction was studied for the




**Scheme-II**

preparation of 7-phenyl-6*H*,7*H*-naphtho[1',2': 5,6]pyrano[3,2-c]chromen-6-one from the reaction of β-naphthol (1 mmol), benzaldehyde (1 mmol) and 4-hydroxycoumarin (1 mmol) and alum (0.1 mmol) in several solvents or under solvent-free conditions at reflux temperature or 120 ºC (Table-1). Under solvent-free conditions, the reaction proceeded smoothly and giving short reaction time and high yield.

TABLE-1
SOLVENT OPTIMIZATION FOR THE SYNTHESIS OF 7-PHENYL-6*H*,7*H*-NAPHTHO[1',2': 5,6]PYRANO[3,2-*c*]CHROMEN-6-ONE

| Entry | Solvent | Temp. / ºC | Time/ min | Yield/ %[a] |
|---|---|---|---|---|
| 1 | MeOH | Reflux | 180 | 0 |
| 2 | $H_2O$ | Reflux | 180 | 0 |
| 3 | $CH_2Cl_2$, | Reflux | 180 | 0 |
| 4 | DMF | 120 | 240 | 20 |
| 5 | $CH_3CN$ | Reflux | 240 | 15 |
| 6 | Neat | 120 | 45 | 86 |

[a]Isolated yield.

Next, to determine the optimum amount of alum, the reaction was carried out by varying amount of the catalyst (Table- 2). The maximum yield was obtained with 10 mol % alum. Further increase in amount of the catalyst in the standard reaction did not have any significant effect on the product yield. With this result, we extended this method to a variety of aldehydes to investigate its scope and generality. The results are summarized in Table-3. Benzaldehyde and other aromatic aldehydes containing electron-withdrawing groups (such as halide and nitro) or electron-donating groups (such as methyl and methoxy) were treated with β-naphthol and 4-hydroxycoumarin to give the corresponding 7-alkyl-6*H*,7*H*-naphtho-[1',2': 5,6]pyrano[3,2-c]chromen-6-ones in good to excellent yields. All of the products 4 exhibited a singlet in their $^1$H spectra at δ = 5.71-6.13 ppm for H-7 and a distinguishing peak at δ = 31.2-36.4 ppm for C-7 in their $^{13}$C NMR spectra. The resonances of carbonyl groups in their $^{13}$C NMR spectrum of 4 appeared at δ = 172.9-177.0 ppm.

A role of alum has been proposed to activate the aldehyde by binding of alum with the oxygen atom, which ultimately enhances the electrophilicity of the aldehyde and leads to a decrease in reaction time. The proposed mechanismis shown in **Scheme-II**. In β-naphthol the electron density at the benzylic C-1 position (which is in conjugation with the aromatic ring) is higher than that at the C-3 position. Thus the regioselective formation of the *o*-quinone methide from this compound involving the C-1 and C-2 positions is favoured. In simple phenolic compounds and 1-naphthol (which are weaker nucleophiles compared to β-naphthol the electron density at the *ortho* position of the hydroxyl group is not sufficient for the reaction of these compounds with the aldehydes leading to the formation of the corresponding *o*-quinone methides.

TABLE-2
AMOUNTS OF CATALYST OPTIMIZATION FOR THE SYNTHESIS OF 7-PHENYL-6*H*,7*H*-NAPHTHO-[1',2': 5,6]PYRANO[3,2-*c*]CHROMEN-6-ONE

| Entry | Alum (mol %) | Time (min) | Yield (%)[a] |
|---|---|---|---|
| 1 | 0 | 240 | 0 |
| 2 | 1 | 120 | 29 |
| 3 | 5 | 60 | 68 |
| 4 | 10 | 45 | 86 |
| 5 | 15 | 45 | 84 |
| 6 | 20 | 40 | 86 |

[a] Isolated yield

TABLE-3
PREPARATION OF 7-ALKYL-6*H*,7*H*-NAPHTHO [1',2': 5,6]PYRANO[3,2-*c*]CHROMEN-6-ONES[A]

| Entry | R | Time/ min | Product | Yield/ %[b] |
|---|---|---|---|---|
| 1 | $C_6H_5$ | 45 | **4a** | 86 |
| 2 | 4-Cl-$C_6H_4$ | 40 | **4b** | 82 |
| 3 | 4-F-$C_6H_4$ | 40 | **4c** | 83 |
| 4 | 4-Me-$C_6H_4$ | 60 | **4d** | 85 |
| 5 | 4-$NO_2$-$C_6H_4$ | 35 | **4e** | 91 |
| 6 | 3-$NO_2$-$C_6H_4$ | 40 | **4f** | 88 |
| 7 | 2,4-$Cl_2$-$C_6H_3$ | 40 | **4g** | 88 |
| 8 | 3,4-$Cl_2$-$C_6H_3$ | 45 | **4h** | 87 |
| 9 | 4-MeO-$C_6H_4$ | 60 | **4i** | 78 |
| 10 | 2,5-$MeO_2$-$C_6H_3$ | 80 | **4j** | 80 |

[a] Reaction conditions: β–naphthol (1 mmol); aldehyde (1 mmol); 4-hydroxycoumarin (1 mmol); alum (0.1 mmol); 120 °C; neat; [b] Isolated yield.

## Conclusion

In conclusion, we described a mild, convenient method for the preparation of some new chromenes by the condensation reaction of β-naphthol, aromatic aldehydes and 4-hydroxycoumarin using cheap, non-toxic, very soluble in water, recyclable and easily available alum catalyst under solvent-free conditions. Additionally, this new reaction might be a useful tool for high-throughput organic synthesis.

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