$Zr(HSO_4)_4$-Catalyzed One-Pot Three-Component Synthesis of 7-Alkyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-ones

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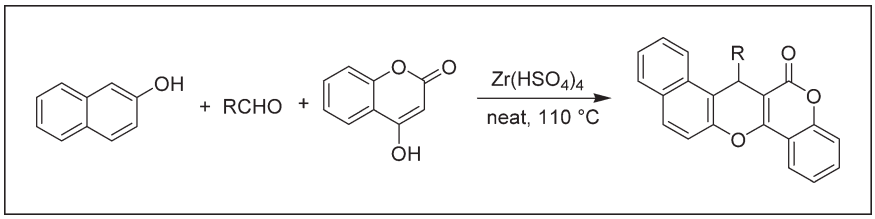

A new, one-pot, simple thermally efficient and solvent-free method for the preparation of 7-alkyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-ones by condensation of β-naphthol, aromatic aldehydes, and 4-hydroxycoumarin using $Zr(HSO_4)_4$ as a safe and efficient catalyst is described. This method has the advantages of high yields, a cleaner reaction, simple methodology, short reaction times, easy workup, and greener conditions.

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INTRODUCTION

Chromenes are important biologically active heterocyclic compounds, which posses antioxidant [1], antibacterial [2], antirhinovirus [3], cytotoxic [4], anticancer [5], antimicrobial [6], and antihypertensive activities [7]. A variety of chromene derivatives have been isolated form a variety of plants [8]. Therefore, a number of methods have been reported for the synthesis of phthalazine derivatives [9]. Nevertheless, the development of new synthetic methods for the efficient preparation of heterocycles containing phthalazine ring fragment is an interesting challenge.

In recent years, metal hydrogen sulfates have been used as an efficient reagent in organic chemistry [10]. A broad range of reactions including deprotection, oxidation, C—C, C—N, and C—O bond formation, and cleavage took place in the presence of these reagents under mild and heterogeneous conditions. In addition, stability, cheapness, ability to produce highly efficient products in a short time, and in many cases reusability is among other important advantages of these reagents.

We herein report that $Zr(HSO_4)_4$ efficiently catalyze the one-pot syntheses of 7-alkyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-ones by condensation of β-naphthol, aromatic aldehydes, and 4-hydroxycoumarin under solvent-free conditions (Scheme 1).

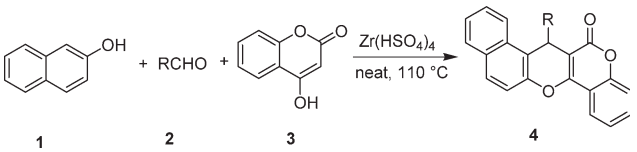
RESULTS AND DISCUSSION

Initially, we conducted the reaction of β-naphthol, benzaldehyde, and 4-hydroxycoumarin in the presence of various metal hydrogen sulfates such as $NaHSO_4$, $Fe(HSO_4)_3$, $Zr(HSO_4)_4$, $Al(HSO_4)_3$ separately at 110°C under solvent-free conditions. The corresponding 7-phenyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-one was formed in 65%, 52%, 91%, and 82% yield (Table 1). $Zr(HSO_4)_4$ was thus selected as the most effective catalyst to carry out this reaction.

Next, to optimize the amount of catalyst and the reaction temperature, the reaction of β-naphthol, benzaldehyde, and 2-hydroxy-1,4-naphthoquinone was studied under solvent-free conditions in the presence of $Zr(HSO_4)_4$ at different temperatures. The results were summarized in Table 2 and showed that the reaction using 5 mol % $Zr(HSO_4)_4$ at 110°C proceeded in highest yield.

With this optimized procedure in hand, the scope of application of this three-component reaction was examined using different aldehydes as staring materials. As seen from Table 3, aromatic aldehydes having electron-donating as well as electron-withdrawing groups were uniformly transformed into the corresponding 7-alkyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-ones in high to excellent yields within 30 min. Substituents on the aromatic ring had no obvious effect on yield or reaction time under the above optimal conditions (Table 3). All of the products **4** exhibited a singlet in their ^1H 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.

Scheme 1



The reusability of the catalyst was tested in the synthesis of 7-phenyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*]chromen-6-ones. The catalyst was recovered after each run, washed with CH_2Cl_2, dried in an oven at 100°C for 30 min prior to use, and tested for its activity in the subsequent run and fresh catalyst was not added. The catalyst was tested for three runs. It was seen that the catalyst displayed very good reusability (Table 3, entry 1).

The plausible mechanism of the reaction is shown in Scheme 2. The reaction likely proceeds *via* initial formation of *ortho*-quinone methide (**5**). The oxonium species (**6**) is then formed on reaction with 2-hydroxynaphthalene-1,4-dione, which then undergoes dehydration to afford the desired product (**4**). 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 *ortho*-quinone methide from this compound involving the C-1 and C-2 positions is favored. 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 *ortho*-quinone methides.

In conclusion, we have developed a highly efficient methodology for three-component reaction of β-naphthol, aromatic aldehydes, and 4-hydroxycoumarin catalyzed by safe $Zr(HSO_4)_4$, furnishing a class of 7-alkyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-*c*] chromen-6-ones in high yield. This method is advantageous in terms of simplicity and mildness and hopefully could find wide application in synthesis of complex chromene-containing compounds.

Table 1

Synthesis of 7-phenyl-6*H*,7*H*-naphtho[1′,2′:5,6]pyrano[3,2-c]chromen-6-one using metal hydrogen sulfates[a].

Entry	Metal hydrogen sulfates	Time (min)	Yield (%)[b]
1	–	120	0
2	$NaHSO_4$	45	65
3	$Fe(HSO_4)_3$	90	52
4	$Zr(HSO_4)_4$	20	91
5	$Al(HSO_4)_3$	20	82

[a] Reaction conditions: β-naphthol (1 mmol), benzaldehyde (1 mmol), 4-hydroxycoumarin (1 mmol), metal hydrogen sulfates (0.05 mmol), neat, 110°C.
[b] Isolated yield.

Table 2

Synthesis of 14-phenyl-14*H*-dibenzo[*a*,*i*]xanthene-8,13-dione under various conditions[a].

Entry	$Zr(HSO_4)_4$ (mol %)	Temperature (°C)	Time (min)	Yield (%)[b]
1	0	110	120	0
2	1	110	45	52
3	2	110	45	62
4	3	110	30	68
5	4	110	30	79
6	4	120	30	82
7	5	25	60	0
8	5	50	45	12
9	5	90	45	69
10	5	100	30	83
11	5	110	20	91
12	5	120	20	90
13	5	130	20	89
14	6	100	20	86
15	6	110	20	90
16	7	110	20	87

[a] Reaction conditions: β-naphthol (1 mmol), benzaldehyde (1 mmol), 4-hydroxycoumarin (1 mmol), neat.
[b] Isolated yield.

EXPERIMENTAL

NMR spectra were determined on Bruker AV-400 instrument at room temperature using tetramethylsilane (TMS) as internal standard. Coupling constants (*J*) were measured in Hz. Infrared spectra were determined on FTS-40 infrared spectrometer. Elemental analysis was performed by a Vario-III elemental analyzer. Mass spectra were taken on a Macro mass spectrometer (Waters) by electrospray method. Melting points

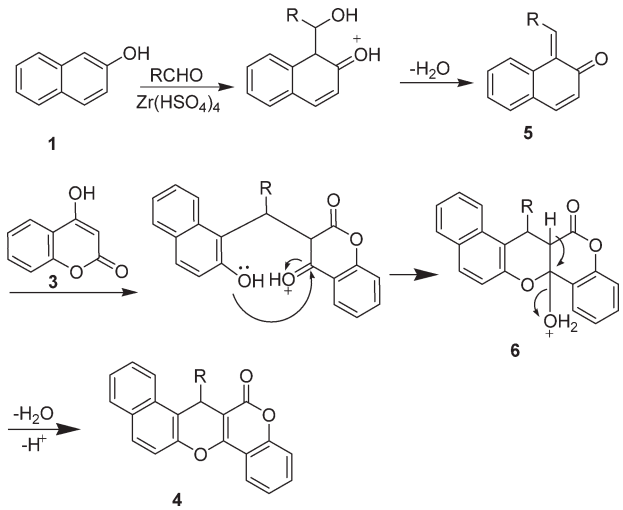
Table 3

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	20	**4a**	91 (87, 84, 79)[c]
2	4-Cl-C_6H_4	25	**4b**	89
3	4-F-C_6H_4	25	**4c**	85
4	4-Me-C_6H_4	15	**4d**	92
5	4-NO_2-C_6H_4	25	**4e**	89
6	3-NO_2-C_6H_4	30	**4f**	81
7	2,4-Cl_2-C_6H_3	25	**4g**	85
8	3,4-Cl_2-C_6H_3	25	**4h**	87
9	4-MeO-C_6H_4	20	**4i**	85
10	2,5-MeO_2-C_6H_3	25	**4j**	82

[a] Reaction conditions: β-naphthol (1 mmol), aldehyde (1 mmol), 4-hydroxycoumarin (1 mmol), $Zr(HSO_4)_4$ (0.05 mmol), 110°C, neat.
[b] Isolated yield.
[c] Yields after three times of catalyst recovery.

Scheme 2



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 4. A mixture of β-naphthol (1 mmol), aldehyde (1 mmol), 4-hydroxycoumarin (1 mmol), and $Zr(HSO_4)_4$ (0.05 mmol) was heated at 110°C for an appropriate time (thin layer chromatography). After completion, the reaction mixture was washed with water (15 mL), and residue was recrystallized from EtOH to afford the pure product **4**. Aqueous washings were collected and evaporated under reduced pressure. After removal of the water, $Zr(HSO_4)_4$ was recovered.

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°C; ^{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°C; ^{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 $C_{26}H_{15}ClO_3$: 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°C; ^{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 $C_{26}H_{15}FO_3$: 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°C; ^{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°C; ^{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°C; ^{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°C; ^{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 $C_{26}H_{14}Cl_2O_3$: 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°C; ^{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 $C_{26}H_{14}Cl_2O_3$: 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°C; ^{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°C; ^{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.

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