Research Article

$\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$ as an Efficient and Recyclable Catalyst for the Synthesis of Dihydropyrano[3,2-*b*]chromenediones

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Received 13 March 2012; Accepted 1 May 2012

Academic Editor: Andrea Penoni

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Dihydropyrano[3,2-*b*]chromenedione derivatives were synthesized in moderate-to-high yields in one-pot three-component reaction from the corresponding aromatic aldehydes, kojic acid, and dimedone, in the presence of catalytic amount of $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$ as a nontoxic, reusable, inexpensive, and easily available reagent, under solvent-free conditions.

1. Introduction

Molecules containing a kojic acid core have been shown to have a broad range of important biological activities including antifungal [1, 2], antineoplastic [3], antiproliferative [4], anti-HIV [5], anticonvulsant [6], anti-inflammatory [7], antioxidative [8], antibacterial [9], and tyrosinase inhibitory activities [10]. Consequently, the synthesis of this heterocycle has attracted considerable attention and a wide variety of methods have been used for its assembly. Recently InCl_3 has been used for the synthesis of dihydropyrano[3,2-*b*]chromenediones [11]. However, the methods are associated with one or more disadvantages such as use of expensive, toxic, and nonrecyclable catalyst and harsh reaction conditions. Thus, there is still need of a simple and general procedure for one-pot synthesis of dihydropyrano[3,2-*b*]chromenedione under mild conditions.

The development of promoted organic reactions using air-stable and water-tolerant lanthanide salts as Lewis acid catalyst is one of the important and challenging subjects in synthesis organic chemistry. In the past few years, Cerium(III) chloride heptahydrate adsorbed on silica gel ($\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$) has emerged as a potential catalyst in affecting various organic transformations due to its high catalytic ability, water tolerance, economic viability, and reusability [12, 13]. Herein we report an efficient, green,

and simple method for the synthesis of dihydropyrano[3,2-*b*]chromenediones via the condensation of aromatic aldehydes, kojic acid, and dimedone, in the presence of catalytic amount of $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$ at 110°C under solvent-free conditions (Scheme 1).

2. Experimental

^1H NMR and ^{13}C NMR spectra were determined on Bruker AV-400 spectrometer (100 MHz for ^{13}C NMR) at room temperature using tetramethylsilane (TMS) as an internal standard (CDCl_3 solution); coupling constants (J) were measured in Hz; IR spectra were determined on FTS-40 infrared spectrometer; elemental analyses were performed by a Vario-III elemental analyzer; mass spectra were taken on a macromass spectrometer (Waters) by electrospray method (ES); melting points were determined on a XT-4 binocular microscope and were uncorrected; $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$ was prepared according to the literature [12]; commercially available reagents were used throughout without further purification unless otherwise stated.

General Procedure for the Preparation of 4. A mixture of kojic acid (1 mmol), aldehyde (1 mmol), dimedone (1 mmol), and $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}/\text{SiO}_2$ (0.05 mmol) was heated at 110°C for an appropriate time (TLC). After completion, ethyl acetate (2

SCHEME 1

TABLE 1: Synthesis of 2-(hydroxymethyl)-7,7-dimethyl-10-phenyl-7,8-dihydropyrano[3,2-*b*]chromene-4,9(6*H*, 10*H*)-dione in the presence of a variety of catalysts^a.

Entry	Catalysts	Time/min	Yield/% ^b
1	—	240	0
2	I ₂ (5 mol%)	60	19
3	<i>p</i> -TsOH (5 mol%)	60	29
4	H ₂ SO ₄ (5 mol%)	90	11
5	HOAc (5 mol%)	90	32
6	CeCl ₃ (5 mol%)	60	84
7	ZnCl ₂ (5 mol%)	60	80
8	CeCl ₃ ·7H ₂ O/SiO ₂ (1 mol%)	90	63
9	CeCl ₃ ·7H ₂ O/SiO ₂ (2 mol%)	60	70
10	CeCl ₃ ·7H ₂ O/SiO ₂ (3 mol%)	60	79
11	CeCl ₃ ·7H ₂ O/SiO ₂ (4 mol%)	45	89
12	CeCl ₃ ·7H ₂ O/SiO ₂ (5 mol%)	45	95
13	CeCl ₃ ·7H ₂ O/SiO ₂ (6 mol%)	45	94
14	CeCl ₃ ·7H ₂ O/SiO ₂ (7 mol%)	45	93

^a Reaction conditions: kojic acid (1 mmol); benzaldehyde (1 mmol); dimerone (1 mmol); neat; 110°C.^b Isolated yield.

× 20 mL) was added, and the solid catalyst was removed by filtration. The solvent was evaporated and the crude product was purified by silica gel column chromatography using ethyl acetate-hexane (7 : 3) as eluents.

2-(Hydroxymethyl)-7,7-dimethyl-10-phenyl-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6*H*, 10*H*)-dione (4a). ¹H NMR (CDCl₃, 400 MHz) δ: 7.32–7.21 (m, 5H), 6.50 (s, 1H), 4.87 (s, 1H), 4.43–4.32 (m, 2H), 2.71–2.59 (m, 2H), 2.29–2.19 (m, 2H), 1.11 (s, 3H), 1.04 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.2, 171.3, 167.3, 163.9, 151.7, 140.5, 137.5, 128.8, 128.1, 127.8, 112.3, 112.2, 60.6, 50.4, 40.9, 38.4, 32.3, 29.0, 27.4; IR (KBr) ν: 3362, 3080, 2952, 2890, 1667, 1637, 1441, 1378, 1219, 1193, 1078, 990, 950, 712; MS (ESI): m/z 353 [M+H]⁺; Anal. calcd for C₂₁H₂₀O₅: C 71.58, H 5.72; found: C 71.65, H 5.68.

2-(Hydroxymethyl)-7,7-dimethyl-10-(4-chlorophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6*H*, 10*H*)-dione (4b). ¹H NMR (CDCl₃, 400 MHz) δ: 7.28–7.18 (m, 4H), 6.52 (s, 1H), 4.87 (s, 1H), 4.41–4.37 (m, 2H), 2.66–2.60 (m, 2H), 2.30–2.21 (m, 2H), 1.11 (s, 3H), 1.03 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.3, 171.4, 168.3, 164.1, 151.2, 138.9, 137.5, 133.6, 129.4, 129.0, 112.0, 111.9, 60.4, 50.3, 40.8, 37.8, 32.3, 28.9, 27.4; IR (KBr) ν: 3325, 2961, 2930, 2870, 1672, 1640, 1600, 1490, 1442, 1377, 1218, 1190, 1076, 1014, 952, 850; MS (ESI): m/z 387 [M+H]⁺; Anal. calcd for C₂₁H₁₉ClO₅: C 65.20, H 4.95; found: C 65.12, H 4.88.

2-(Hydroxymethyl)-7,7-dimethyl-10-(4-florophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6*H*, 10*H*)-dione (4c). ¹H NMR (CDCl₃, 400 MHz) δ: 7.24–7.20 (m, 2H), 6.99–6.95 (m, 2H), 6.52 (s, 1H), 4.87 (s, 1H), 4.40–4.36 (m, 2H), 2.65–2.61 (m, 2H), 2.25–2.22 (m, 2H), 1.10 (s, 3H), 1.03 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.4, 171.5, 168.3, 164.0, 160.9, 151.5, 137.4, 136.2, 129.7, 129.6, 115.8, 115.6, 112.2, 112.0, 60.4, 50.3, 40.8, 37.7, 32.3, 28.9, 27.4; IR (KBr) ν: 3363, 2953, 2930, 2853, 1675, 1638, 1601, 1509, 1443, 1378, 1219, 1192, 1157, 1075, 962, 845, 683; MS (ESI): m/z 371 [M+H]⁺; Anal. calcd for C₂₁H₁₉FO₅: C 68.10, H 5.17; found: C 68.20, H 5.12.

2-(Hydroxymethyl)-7,7-dimethyl-10-(4-nitrophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6*H*, 10*H*)-dione (4d). ¹H NMR (CDCl₃, 400 MHz) δ: 8.18 (d, 2H, *J* = 8.8 Hz), 7.47 (d, 2H, *J* = 8.4 Hz), 6.53 (s, 1H), 5.03 (s, 1H), 4.42–4.37 (m, 2H), 2.69–2.65 (m, 2H), 2.27–2.23 (m, 2H), 1.13 (s, 3H), 1.04 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.0, 170.8, 167.0, 164.4, 150.0, 147.4, 137.9, 129.1, 124.1, 112.6, 111.5, 60.6, 50.3, 40.9, 38.4, 32.3, 28.9, 27.5; IR (KBr) ν: 3334, 2960, 2928, 2855, 1675, 1633, 1596, 1520, 1375, 1347, 1216, 1123, 1058, 1003, 946, 867, 622; MS (ESI): m/z 398 [M+H]⁺; Anal. calcd for C₂₁H₁₉NO₇: C 63.47, H 4.82, N 3.52; found: C 63.35, H 4.850, N 3.57.

2-(Hydroxymethyl)-7,7-dimethyl-10-(3-nitrophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6*H*, 10*H*)-dione (4e). ¹H NMR (CDCl₃, 400 MHz) δ: 8.11–8.09 (m, 2H), 7.63 (d, 1H,

TABLE 2: Preparation of dihydropyrano[3,2-*b*]chromenes^a.

Entry	R	Time/min	Product	Yield/% ^b	m.p./°C (Lit.)
1	C ₆ H ₅	40	4a	95 (93, 89) ^c	184-185 (186-188) ¹¹
2	4-Cl-C ₆ H ₄	40	4b	96	205-206
3	4-F-C ₆ H ₄	40	4c	93	161-162 (160-164) ¹¹
4	4-NO ₂ -C ₆ H ₄	30	4d	95	229-230
5	3-NO ₂ -C ₆ H ₄	30	4e	96	212-213
6	2-Cl-C ₆ H ₄	40	4f	92	216-217
7	2,4-Cl ₂ -C ₆ H ₃	40	4g	90	166-167
8	3,4,5-(MeO) ₃ -C ₆ H ₂	60	4h	86	174-175
9	2,5-(MeO) ₂ -C ₆ H ₃	60	4i	86	192-193
10	4-Me-C ₆ H ₄	45	4j	89	214-215
11	4-MeO-C ₆ H ₄	45	4k	88	178-179 (180-182) ¹¹

^a Reaction conditions: kojic acid (1 mmol); aldehyde (1 mmol); dimedone (1 mmol); CeCl₃·7H₂O/SiO₂ (0.05 mmol); neat; 110°C.

^b Isolated yield.

^c The catalyst was reused for three runs.

¹¹ It is m.p. of compound in Literature 11.

J = 8.0 Hz), 7.50 (t, 1H, *J* = 8.0 Hz), 6.53 (s, 1H), 5.03 (s, 1H), 4.45–4.33 (m, 2H), 2.74–2.61 (m, 2H), 2.30–2.20 (m, 2H), 1.12 (s, 3H), 1.05 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.3, 171.3, 168.2, 164.7, 150.2, 148.4, 142.4, 137.7, 134.3, 129.8, 123.4, 122.9, 112.2, 111.3, 60.4, 50.3, 40.8, 38.3, 32.3, 28.9, 27.5; IR (KBr) ν: 3393, 2955, 2922, 2851, 1669, 1637, 1599, 1530, 1448, 1377, 1350, 1213, 1143, 1080, 678; MS (ESI): *m/z* 398 [M+H]⁺; Anal. calcd for C₂₁H₁₉NO₇: C 63.47, H 4.82, N 3.52; found: C 63.39, H 4.80, N 3.55.

2-(Hydroxymethyl)-7,7-dimethyl-10-(2-chlorophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4f**). ¹H NMR (CDCl₃, 400 MHz) δ: 7.36 (dd, 1H, *J* = 0.8, 8.0 Hz), 7.20–7.16 (m, 3H), 6.49 (s, 1H), 5.44 (s, 1H), 4.40–4.34 (m, 2H), 2.67–2.64 (m, 2H), 2.26–2.22 (m, 2H), 1.12 (s, 3H), 1.08 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.1, 171.3, 168.0, 164.7, 150.9, 137.8, 137.5, 133.7, 130.1, 129.0, 127.3, 112.0, 111.5, 60.4, 50.3, 40.8, 35.7, 32.2, 29.0, 27.5; IR (KBr) ν: 3291, 2956, 2931, 2860, 1673, 1634, 1600, 1468, 1445, 1378, 1221, 1116, 1080, 758; MS (ESI): *m/z* 387 [M+H]⁺; Anal. calcd for C₂₁H₁₉ClO₅: C 65.20, H 4.95; found: C 65.30, H 4.82.

2-(Hydroxymethyl)-7,7-dimethyl-10-(2,4-dichlorophenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4g**). ¹H NMR (CDCl₃, 400 MHz) δ: 7.39 (s, 1H), 7.21–7.18 (m, 1H), 7.14–7.12 (m, 1H), 6.50 (s, 1H), 5.39 (s, 1H), 4.41–4.37 (m, 2H), 2.71–2.65 (m, 2H), 2.26–2.22 (m, 2H), 1.13 (s, 3H), 1.08 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.0, 171.1, 167.6, 164.8, 150.3, 137.7, 134.4, 134.2, 130.9, 129.9, 127.7, 112.2, 111.2, 60.5, 50.3, 40.8, 37.5, 32.2, 28.9, 27.6; IR (KBr) ν: 3296, 2952, 2863, 1676, 1637, 1600, 1445, 1376, 1221, 1101, 1079, 854; MS (ESI): *m/z* 422 [M+H]⁺; Anal. calcd for C₂₁H₁₈Cl₂O₅: C 59.87, H 4.31; found: C 59.90, H 4.28.

2-(Hydroxymethyl)-7,7-dimethyl-10-(3,4,5-trimethoxyphenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4h**). ¹H NMR (CDCl₃, 400 MHz) δ: 6.50–6.44 (m, 3H), 4.81 (s, 1H), 4.45–4.37 (m, 2H), 3.80 (s, 6H), 3.79 (s, 3H), 2.68–2.63 (m, 2H), 2.28–2.27 (m, 2H), 1.14

(s, 3H), 1.11 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.3, 171.3, 167.7, 164.0, 153.4, 151.5, 137.3, 136.1, 112.1, 105.0, 60.8, 60.6, 56.1, 50.3, 40.9, 38.5, 32.2, 29.3, 27.1; IR (KBr) ν: 3296, 2960, 2923, 2869, 1673, 1637, 1598, 1508, 1422, 1375, 1329, 1220, 1126, 1076, 956; MS (ESI): *m/z* 443 [M+H]⁺; Anal. calcd for C₂₄H₂₆O₈: C 65.15, H 5.92; found: C 65.20, H 5.89.

2-(Hydroxymethyl)-7,7-dimethyl-10-(2,6-dimethoxyphenyl)-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4i**). ¹H NMR (CDCl₃, 400 MHz) δ: 6.77–6.74 (m, 3H), 6.47 (s, 1H), 5.17 (s, 1H), 4.40–4.35 (m, 2H), 3.78 (s, 3H), 3.72 (s, 3H), 2.63–2.61 (m, 2H), 2.24–2.20 (m, 2H), 1.11 (s, 3H), 1.04 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.3, 171.3, 167.3, 164.6, 153.7, 151.7, 151.6, 137.8, 129.4, 116.1, 113.4, 112.7, 112.1, 111.4, 60.6, 56.6, 55.6, 50.4, 40.9, 33.8, 32.2, 29.2, 27.1; IR (KBr) ν: 3284, 2950, 2930, 2854, 1672, 1636, 1594, 1503, 1449, 1379, 1226, 1194, 1148, 1080, 1047, 819, 709; MS (ESI): *m/z* 413 [M+H]⁺; Anal. calcd for C₂₃H₂₄O₇: C 66.98, H 5.87; found: C 67.05, H 5.80.

2-(Hydroxymethyl)-7,7-dimethyl-10-4-methylphenyl-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4j**). ¹H NMR (CDCl₃, 400 MHz) δ: 7.13 (d, 2H, *J* = 8.0 Hz), 7.08 (d, 2H, *J* = 8.0 Hz), 6.50 (s, 1H), 4.83 (s, 1H), 4.38–4.34 (m, 2H), 2.64–2.61 (m, 2H), 2.28 (s, 3H), 2.23–2.20 (m, 2H), 1.10 (s, 3H), 1.03 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.4, 171.5, 168.2, 163.9, 152.0, 137.6, 137.5, 137.3, 129.5, 127.9, 112.4, 111.9, 60.4, 50.4, 40.8, 37.9, 32.2, 29.0, 27.4, 21.1; IR (KBr) ν: 3368, 2953, 2931, 2847, 1668, 1636, 1442, 1376, 1219, 1189, 1120, 1076, 950, 862, 624; MS (ESI): *m/z* 367 [M+H]⁺; Anal. calcd for C₂₂H₂₂O₅: C 72.12, H 6.05; found: C 72.20, H 6.00.

2-(Hydroxymethyl)-7,7-dimethyl-10-4-methoxyphenyl-7,8-dihydropyrano[3,2-*b*]chromene-4,9 (6H, 10H)-dione (**4k**). ¹H NMR (CDCl₃, 400 MHz) δ: 7.18–7.16 (m, 2H), 6.83–6.80 (m, 2H), 6.50 (s, 1H), 4.83 (s, 1H), 4.40–4.35 (m, 2H), 3.75 (s, 3H), 2.65–2.61 (m, 2H), 2.23–2.22 (m, 2H), 1.10 (s, 3H), 1.04 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ: 196.4,

171.5, 168.0, 16.7, 159.0, 152.0, 137.3, 132.7, 129.1, 114.2, 112.5, 129.1, 60.5, 55.2, 50.4, 40.8, 37.5, 32.2, 29.0, 27.4; IR (KBr) ν : 3355, 2958, 2922, 2836, 1672, 1637, 1511, 1443, 1377, 1219, 1193, 1120, 1029, 950, 860; MS (ESI): m/z 383 $[M+H]^+$; Anal. calcd for $C_{22}H_{22}O_6$: C 69.10, H 5.80; found: C 69.20, H 5.72.

3. Results and Discussion

First, to achieve suitable conditions for the synthesis of dihydropyrano[3,2-*b*]chromenediones, we tested the reaction of kojic acid, benzaldehyde, and dimedone as a simple model system with or without the presence of various catalysts (Table 1). As can be seen in Table 1, the best result was obtained with 5 mol% of $CeCl_3 \cdot 7H_2O/SiO_2$ as the catalyst at 110°C under solvent-free conditions (entry 12). Using less catalyst resulted in lower yields, whereas higher amounts of catalyst did not affect reaction times and yields (significantly) (Table 1). When this reaction was carried out without $CeCl_3 \cdot 7H_2O/SiO_2$ or with other catalysts such as *p*-TsOH, HOAc, I_2 , and H_2SO_4 , the yield of the expected product was low (Table 1). In the presence of $CeCl_3$ or $ZnCl_2$ as catalyst, the product was obtained in moderate yields.

Encouraged by this success, a variety of aromatic aldehydes were employed under similar conditions to evaluate the substrate scope of this reaction. When a mixture of aromatic aldehydes, kojic acid, and dimedone, and a catalytic amount of $CeCl_3 \cdot 7H_2O/SiO_2$ was stirred at 110°C under solvent-free conditions, the corresponding dihydropyrano[3,2-*b*]chromenediones were obtained in excellent yield (Table 2). The reactions were completed smoothly within 40 to 60 min, and the product was isolated by filtration and washing the solid residues with ethyl acetate, the remaining catalyst being reused for the fresh reactions. There is little decrease in the better yield of products even though the reaction has also been observed in its absence. When this reaction was carried out with aliphatic aldehyde such as butanal or pentanal, TLC and 1H NMR spectra of the reaction mixture showed a combination of starting materials and numerous products; the yield of the expected product was very poor.

4. Conclusion

In summary, we have described a successful strategy, efficient and convenient green synthesis for the preparation of dihydropyrano[3,2-*b*]chromenediones in valuing cyclocondensation reaction of aromatic aldehydes, kojic acid, and dimedone using the inexpensive, nontoxic, and easily available $CeCl_3 \cdot 7H_2O/SiO_2$ catalyst. The method offers several advantages including high yield of products, recyclable of the catalyst and easy experimental work-up procedure, which makes it a useful process for the synthesis of dihydropyrano[3,2-*b*]chromenediones.

Acknowledgment

The authors are pleased to acknowledge the financial support from Xinxiang Medical University.

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