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Iodine-Catalyzed Mild and Efficient Method for the Synthesis of Chalcones

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Iodine-Catalyzed Mild and Efficient Method for the Synthesis of Chalcones

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Abstract: Molecular iodine is found to catalyze the condensation of acetophenones with various aromatic aldehydes to prepare chalcones.

Keywords: Chalcone synthesis, Claisen–Schmidt reaction, iodine catalyst

INTRODUCTION

For the synthesis of chalcones, the Claisen–Schmidt (CS) condensation of a ketone with an aldehyde had been carried out in the presence of aq. NaOH,[1,2] Potassium hydroxide (KOH),[3,4] $Ba(OH)_2$[5] hydrotalcites, zeolites,[6] LiHDMS,[7] and calcined $NaNO_3$/natural phosphates.[8,9] The acid-catalyzed methods include the use of $AlCl_3$,[10] dry HCl,[11] $Zn(bpy)(OAc)_2$,[12] $TiCl_4$,[13] $Cp_2ZrH_2/NiCl_2$,[14] and $RuCl_3$.[15] Recently, $BF_3 \cdot Et_2O$,[16,17] $SOCl_2$/EtOH,[18] and Brønsted acidic ionic liquid catalysts[19] have been used to effect this reaction.

Because of its mild Lewis acidity, molecular I_2 has recently received considerable attention as an inexpensive, nontoxic, readily available catalyst for various organic transformations.[20–31]

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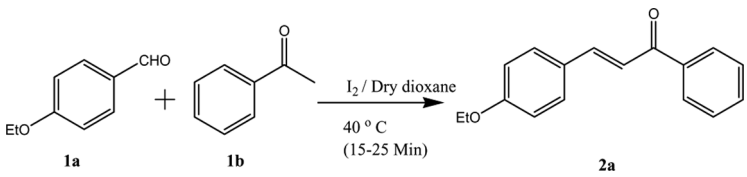


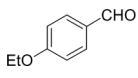
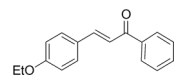
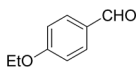
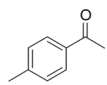
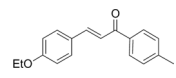
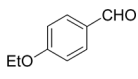
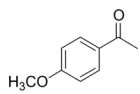
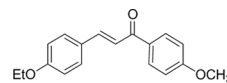
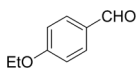
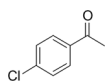
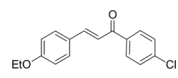
Scheme 1. Iodine-catalyzed synthesis of chalcones.

RESULTS AND DISCUSSION

As a preliminary study, we treated 4-ethoxy benzaldehyde **1a** with acetophenone **1b** in dry dioxane in the presence of I_2 (5 mol %) at 40°C to form chalcone **2a** in 85% yield (Scheme 1) and characterized the products through NMR, mass, and infrared (IR) spectroscopic studies.

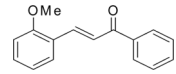
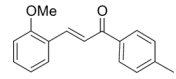
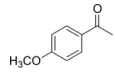
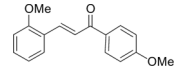
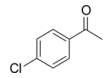
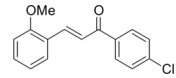
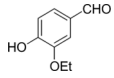
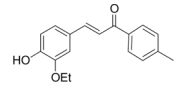
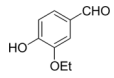
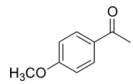
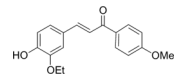
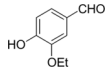
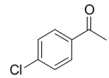
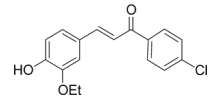
To optimize the iodine requirements, 1 mol%, 5 mol%, 10 mol%, and 20 mol % were employed, and the best results were obtained with 5 mol% of iodine in terms of yield and duration. In the absence of iodine, the reaction did not proceed. We next investigated the scope and generality of the reaction in which several chalcones were successfully synthesized **2b** to **2k** (Table 1) in high yields, illustrating this novel and rapid procedure. In all cases, the reactions proceeded rapidly

Table 1. Chalcones and yields

Entry	Substrate: Aldehyde	Substrate: Ketone	Product[a]	Time (min)	Isolated yield (%)	Mp (°C)
2a				20	85	52–53
2b				25	90	95–96
2c				15	84	105–106
2d				15	86	125–126

(*Continued*)

Table 1. Continued

Entry	Substrate: Aldehyde	Substrate: Ketone	Product[a]	Time (min)	Isolated yield (%)	Mp (°C)
2e				20	88	53–54
2f				15	90	Liquid
2g				25	86	Liquid
2h				25	85	75–76
2i				20	84	124–125
2j				15	86	114–115
2k				15	82	104–105

[a]All the products were characterized ^{1}H NMR, ^{13}C NMR, and mass spectroscopy.
[b]Isolated yields after column chromatography.

under mild conditions at 40°C. Most of the products were formed within 15–25 min.

To our knowledge, there are no reports of I_2-catalyzed Claisen–Schmidt reaction for the synthesis of chalcones. Other researchers[32,33] have recently reported that iodine catalyzed the two-component condensation of cycloalkanones and aryladehydes to afford the corresponding α,α^1-bis (arylmethylidene) cycloalkanones.

CONCLUSION

In conclusion, we have developed a simple and efficient synthesis of substituted chalcones via Claisen–Schmidt condensation using molecular I_2 as a catalyst.

EXPERIMENTAL

Melting points were recorded on a Buchi-530 capillary melting-point apparatus and are uncorrected. IR spectra of the compounds were recorded on a Perkin-Elmer AC-1 spectrometer. ^{1}H NMR spectra were run on Bruker Avance DPX 300-MHz spectrometer in $CDCl_3$, and tetramethylsilane (TMS) was used as the internal standard. Electrospray ionization (ESI) mass spectra were recorded on a Jeol S X 102/DA-6000 instrument. Silica gel (60–120 mesh) was used as a stationary phase to isolate the compounds.

General Procedure for the Preparation of 3-(4-Ethoxy-phenyl)-1-phenyl-propenone 2a

A mixture of a 4-ethoxy benzaldehyde **1a** (150 mg, 1.0 mmol), acetophenone **1b** (120 mg, 1.0 mmole), and iodine (5 mol%) in dry dioxane was stirred at room temperature (40°C). The reaction was monitored by thin-layer chromatography (TLC), and on completion, excess iodine was quenched by adding saturated sodium thiosulfate solution (5 mL) and extracted with chloroform. The organic phase was washed with saturated $NaHCO_3$ solution, dried over anhydrous Na_2SO_4, and then concentrated to give **2a** (214 mg, 85%).

Selected Data

3-(4-Ethoxyphenyl)-1-phenylpropenone (**2a**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.01 (d, $J = 7.2$ Hz, 2H), 7.79 (d, $J = 15.9$ Hz, 1H), 7.57–7.38 (m, 6H), 6.89 (d, $J = 7.9$ Hz, 2H), 4.01 (q, $J = 7.5$, 6.8 Hz, 2H), 1.40 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (75 MHz) δ 190.48, 161.20, 144.74, 138.52, 132.66, 130.30, 128.59, 128.43, 127.37, 119.50, 114.93, 63.52, 14.54; ESI (m/z) 252 $[M + H]^+$; IR (KBr cm^{-1}): 3021, 2927, 1656, 1596, 1216, 760.

3-(4-Ethoxyphenyl)-1-*p*-tolylpropenone (**2b**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.94 (d, $J = 7.6$ Hz, 2H), 7.79 (d, $J = 15.2$ Hz, 1H), 7.57 (d, $J = 8.7$ Hz, 2H), 7.41 (d, $J = 15.2$ Hz, 1H), 7.27 (d, $J = 7.6$ Hz, 2H), 6.90 (d, $J = 8.1$ Hz, 2H), 4.02 (q, $J = 7.5$,

6.8 Hz, 2H), 1.40 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (75 MHz) δ 189.87, 161.11, 144.33, 143.33, 135.90, 130.18, 129.28, 128.61, 127.47, 119.46, 114.84, 63.70, 21.71, 14.85; ESI (m/z) 267 $[M + H]^+$; IR (KBr cm^{-1}): 3030, 2977, 1648, 1563, 1217.

3-(4-Ethoxyphenyl)-1-(4-methoxyphenyl)-propenone (**2c**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.05 (d, $J = 9.0$ Hz, 2H), 7.79 (d, $J = 15.4$ Hz, 1H), 7.59 (d, $J = 7.9$ Hz, 2H), 7.43 (d, $J = 15.4$ Hz, 1H), 7.0–6.91 (m, 4H), 4.08 (q, $J = 7.5$, 6.8 Hz, 2H), 3.89 (s, 3H), 1.44 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (75 MHz) δ 188.88, 163.30, 160.91, 143.93, 131.42, 130.71, 130.16, 127.62, 119.42, 114.88, 113.81, 63.52, 55.62, 14.54; ESI (m/z) 283 $[M + H]^+$; IR (KBr cm^{-1}): 3019, 1657, 1601, 1216, 1028, 759.

1-(4-Chlorophenyl)-3-(4-ethoxyphenyl)-propenone (**2d**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.95 (d, $J = 9.0$ Hz, 2H), 7.77 (d, $J = 15.7$ Hz, 1H), 7.56 (d, $J = 7.9$ Hz, 2H), 7.44 (d, $J = 15.8$ Hz, 2H), 7.35 (d, $J = 15.8$ Hz, 1H), 4.05 (q, $J = 7.5$, 6.8 Hz, 2H), 1.42 (t, $J = 6.7$ Hz, 3H); ^{13}C NMR (50 MHz) δ 189.79, 162.06, 146.03, 139.32, 137.26, 130.8, 130.29, 129.3, 127.65, 119.31, 115.35, 64.15, 15.17; ESI (m/z) 287 $[M + H]^+$; IR (KBr cm^{-1}): 3019, 1657, 1601, 1216, 1028, 759.

3-(2-Methoxyphenyl)-1-phenylpropenone (**2e**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.17 (d, $J = 15.4$ Hz, 1H), 8.04 (d, $J = 7.7$ Hz, 2H), 7.67–7.45 (m, 5H), 7.34 (t, $J = 7.7$ Hz, 1H), 6.99–6.94 (t, $J = 7.7$ Hz, 1H), 6.89 (d, $J = 9.0$ Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (75 MHz) δ 191.01, 158.96, 140.51, 138.55, 132.79, 132.01, 129.26, 128.61, 128.53, 123.90, 122.72, 120.89, 111.34, 55.62; ESI (m/z) 239 $[M + H]^+$; IR (KBr cm^{-1}): 3061, 2956, 2837, 1659, 1597, 1340, 1248, 1209, 1018, 753.

3-(2-Methoxyphenyl)-1-*p*-tolylpropenone (**2f**)

^{1}H NMR ($CDCl_3$, 200 MHz) δ 8.14 (d, $J = 16.5$ Hz, 1H), 7.91 (d, $J = 8.0$ Hz, 2H), 7.63–7.55 (m, 2H), 7.32–7.18 (m, 3H), 6.94–6.80 (m, 2H), 3.77 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (50 MHz) δ 190.66, 159.18, 143.72, 140.12, 136.29, 132.11, 129.72, 129.41, 129.05, 124.32, 122.97,

121.17, 111.65, 55.81, 22.12; ESI (m/z) 253 $[M+H]^+$; IR (KBr, cm^{-1}): 3019, 2932, 1654, 1601, 1216, 1026, 760.

3-(2-Methoxyphenyl)-1-(4-methoxyphenyl)-propenone (**2g**)

^1H NMR ($CDCl_3$, 300 MHz) δ 8.13 (d, $J = 16.1$ Hz, 1H), 8.05 (d, $J = 9.3$ Hz, 2H), 7.68–7.62 (m, 2H), 7.38 (t, $J = 8.1$ Hz, 1H), 7.0–6.94 (m, 4H), 3.92 (s, 3H), 3.89 (s, 3H); ^{13}C NMR (75 MHz) δ 189.40, 163.29, 158.77, 139.55, 131.60, 131.41, 130.85, 129.20, 124.16, 122.70, 120.77, 113.76, 111.27, 55.55, 55.48; ESI (m/z) 268 $[M+H]^+$; IR (KBr, cm^{-1}): 3017, 2935, 1655, 1599, 1501, 1218, 1168, 1026, 758.

1-(4-Chlorophenyl)-3-(2-methoxyphenyl)-propenone (**2h**)

^1H NMR ($CDCl_3$, 300 MHz) δ 8.13 (d, $J = 14.8$ Hz, 1H), 7.94 (d, $J = 9.2$ Hz, 2H), 7.61 (d, $J = 8.3$ Hz, 1H), 7.54 (s, 1H), 7.42 (d, $J = 7.3$ Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 1H), 6.99–6.89 (m, 2H), 3.88 (s, 3H); ^{13}C NMR (50 MHz) δ 190.06, 159.44, 141.23, 139.29, 137.18, 132.47, 130.4, 129.69, 129.25, 124.06, 122.56, 121.24, 111.68, 55.93; ESI (m/z) 273 $[M+H]^+$; IR (KBr, cm^{-1}): 3017, 2935, 1655, 1599, 1501, 1218, 1168, 1026, 758.

3-(3-Ethoxy-4-hydroxyphenyl)-1-*p*-tolylpropenone (**2i**)

^1H NMR ($CDCl_3$, 300 MHz) δ 7.94 (d, $J = 8.4$ Hz, 2H), 7.74 (d, $J = 15.4$ Hz, 1H), 7.40 (d, $J = 4.2$ Hz, 1H), 7.28 (d, $J = 8.4$ Hz, 2H), 7.20 (d, $J = 7.7$ Hz, 1H), 7.11 (s, 1H), 6.96 (d, $J = 7.7$ Hz, 1H), 4.16 (q, $J = 6.9$, 6.9 Hz, 2H), 2.43 (s, 3H), 1.47 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (50 MHz) δ 190.24, 148.44, 146.18, 145.06, 143.44, 135.86, 129.34, 128.62, 127.46, 123.17, 119.61, 114.90, 111.04, 65.02, 22.08, 14.82; ESI (m/z) 283 $[M+H]^+$; IR (KBr, cm^{-1}): 3524, 3017, 1588, 1512, 1441.

3-(3-Ethoxy-4-hydroxyphenyl)-1-(4-methoxyphenyl)-propenone (**2j**)

^1H NMR ($CDCl_3$, 300 MHz) δ 8.03 (d, $J = 9.4$ Hz, 2H), 7.74 (d, $J = 15.9$ Hz, 1H), 7.39 (d, $J = 15.9$ Hz, 1H), 7.20 (d, $J = 8.7$ Hz, 1H), 7.11 (s, 1H), 6.98–6.95 (m, 3H), 4.15 (q, $J = 6.9$, 6.9 Hz, 2H), 3.87 (s, 3H), 1.46 (t, $J = 6.8$ Hz, 3H);^{13}C NMR (50 MHz) δ 189.34, 163.73, 148.82, 146.72, 145.07, 131.82, 131.27, 127.96, 123.54, 119.79, 115.31, 114.21, 111.49, 65.1, 55.93, 15.48; ESI (m/z) 299 $[M+H]^+$; IR (KBr, cm^{-1}): 3012, 2925, 1675, 1594, 1218, 1166, 1022.

1-(4-Chlorophenyl)-3-(3-ethoxy-4-hydroxyphenyl)-propenone (**2k**)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.95 (d, $J = 7.9$ Hz, 2H), 7.76 (d, $J = 15.9$ Hz, 1H), 7.47 (d, $J = 7.9$ Hz, 2H), 7.40–7.29 (m, 2H), 7.21 (d, $J = 7.9$ Hz, 1H), 7.12 (s, 1H), 6.95 (d, $J = 7.1$ Hz, 1H), 6.21 (s, 1H), 4.18 (q, $J = 6.9$, 6.9 Hz, 2H), 1.49 (t, $J = 6.8$ Hz, 3H); ESI (m/z) 303 $[M + H]^+$; ; IR (KBr, cm^{-1}): 3016, 2932, 1665, 1594, 1531, 1218, 1026, 748.

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