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A novel approach for the synthesis of β-keto esters: one-pot reaction of carboxylic acids with chlorosulfonyl isocyanate

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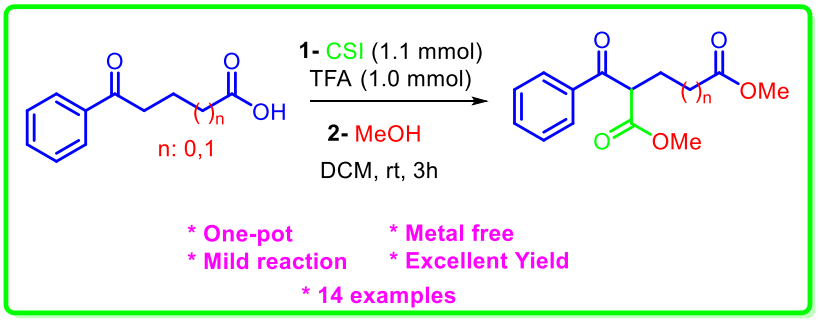
Abstract

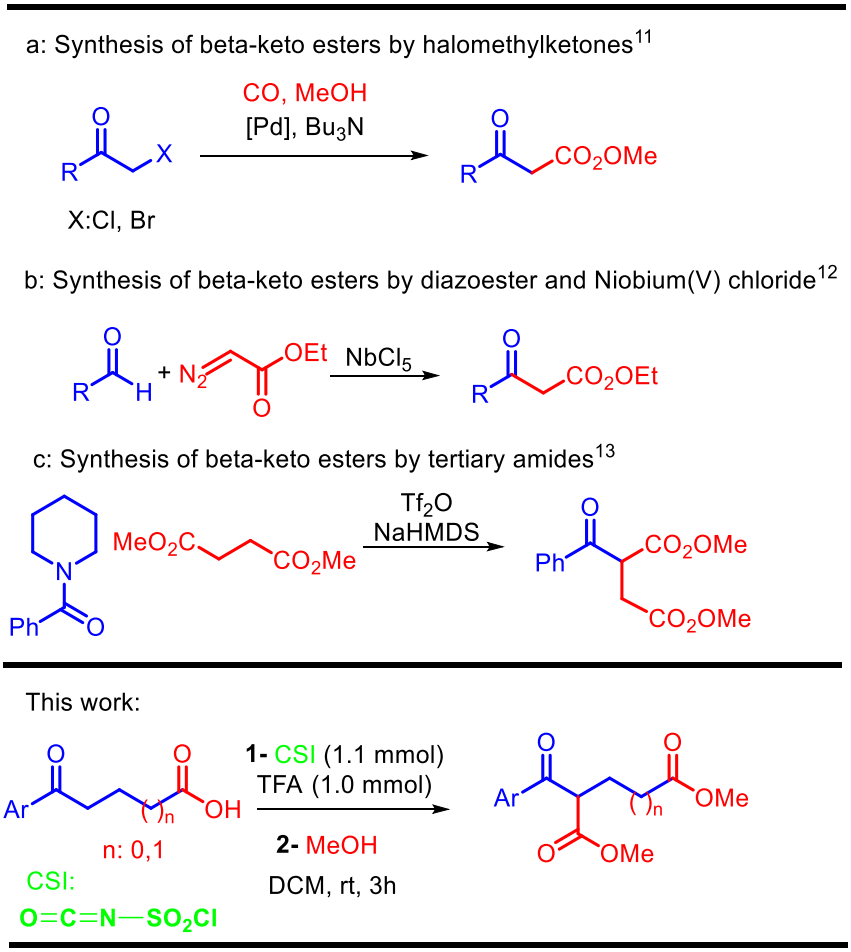
β-Keto esters were synthesized by direct carboxylation of various 4- and 5-oxo-carboxylic acid derivatives in the presence of chlorosulfonyl isocyanate in excellent yield under mild conditions. Additionally, optimization conditions were examined for synthesis β-keto esters. Finally, it has been found that trifluoroacetic acid is efficient in DCM under optimized conditions. This efficient one-pot novel method is very usefull, fair price and easy to handle of β-keto esters.



Keywords: β-Keto ester, chlorosulfonyl isocyanate, carboxylic acid, trifluoroacetic acid, one-pot

Introduction

β-Keto esters are multi-coupling reagents that have both a nucleophilic carbon and an electrophilic carbonyl group. These molecules are very significant intermediates for the synthesis of a wide range of bioactive and natural products such as 1,4-dihydropyridines, arylcoumarins, and 3,4-dihydropyrimidinones,[1] chokol,[2] syncarpic acid,[3] and polyoxamic acid.[4] Furthermore, they are also used preparation of catalysis,[5] polymer stabilization,[6] synthesis of metal microfilms impregnation and liquid crystal preparation technology.[7] Therefore, various methods have been investigated for the synthesis of their derivatives.[1] Common methods for the synthesis of β-keto esters mostly rely on Claisen reaction[8] and related condensations of enolates with alkyl carbonates and oxalates followed by decarbonylation.[9,10]



Scheme 1. Methods for the synthesis of β-keto ester derivatives

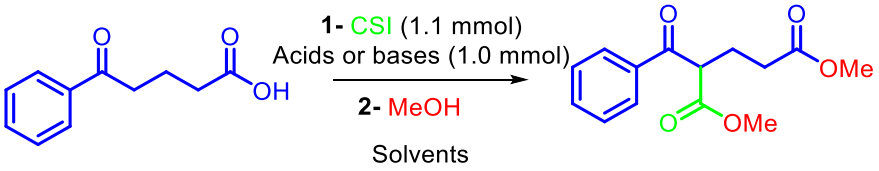
In recent years, with the increasing demands to β-keto esters, synthesis of these molecules has been reported alkoxycarboxylation of halomethylketones with Pd-catalyzed (Scheme 1a).[11] Meanwhile, synthesis of β-keto esters by C-H insertion reactions of diazoesters with Niobium(V) chloride catalyzed were reported by the Yadav group (Scheme 1b).[12] Synthesis of β-keto esters by aza-Knoevenagel reactions of tertiary amides have been also developed (Scheme 1c).[13] Recent developments in the synthetic use of CSI are known in the literature.[14] Previously, we completed reaction of a range of benzoic acid derivatives with CSI that were yielded sulfamate derivatives.[15]

In this work, we reported novel and efficient one-pot synthetic method for the synthesis of β-keto esters (**2a-n**) from carboxylic acid derivatives (**1a-n**) under mild conditions in good yields (Scheme 1).

Results and Discussion

Initially, the reaction of 5-oxo-5-phenylpentanoic acid with CSI (**1a**) was chosen that as the model reaction which was carried out in various solvents, acids, bases. The solvent effect was investigated with dichloromethane (DCM) and acetonitrile in the absence of acid and base at room temperature. In these reactions, 2-benzoylpentanedioate (**2a**) was observed in 54% and 42% isolated yield, respectively. The structure of dimethyl 2-benzoylpentanedioate (**2a**) was verified with ^{1}H- and ^{13}C-NMR, IR, and HRMS. Then, acid or base effect was examined with the triflic acid (TfOH), trifluoroacetic acid (TFA), triethylamine (NEt_3), and pyridine. The use of TfOH gave the dimethyl 2-benzoylpentanedioate (**2a**) in good yield (entry 2 and 7). The desired product **2a** yielded the use of NEt_3, or pyridine in poor yield (entry 4, 5, 9, and 10). The desired product dimethyl 2-benzoylpentanedioate (**2a**) was obtained in 92% yield, when the reaction was performed in DCM with TFA (entry 8).

Table 1. Model reaction for novel synthesis of dimethyl 2-benzoylpentanedioate (**2a**)

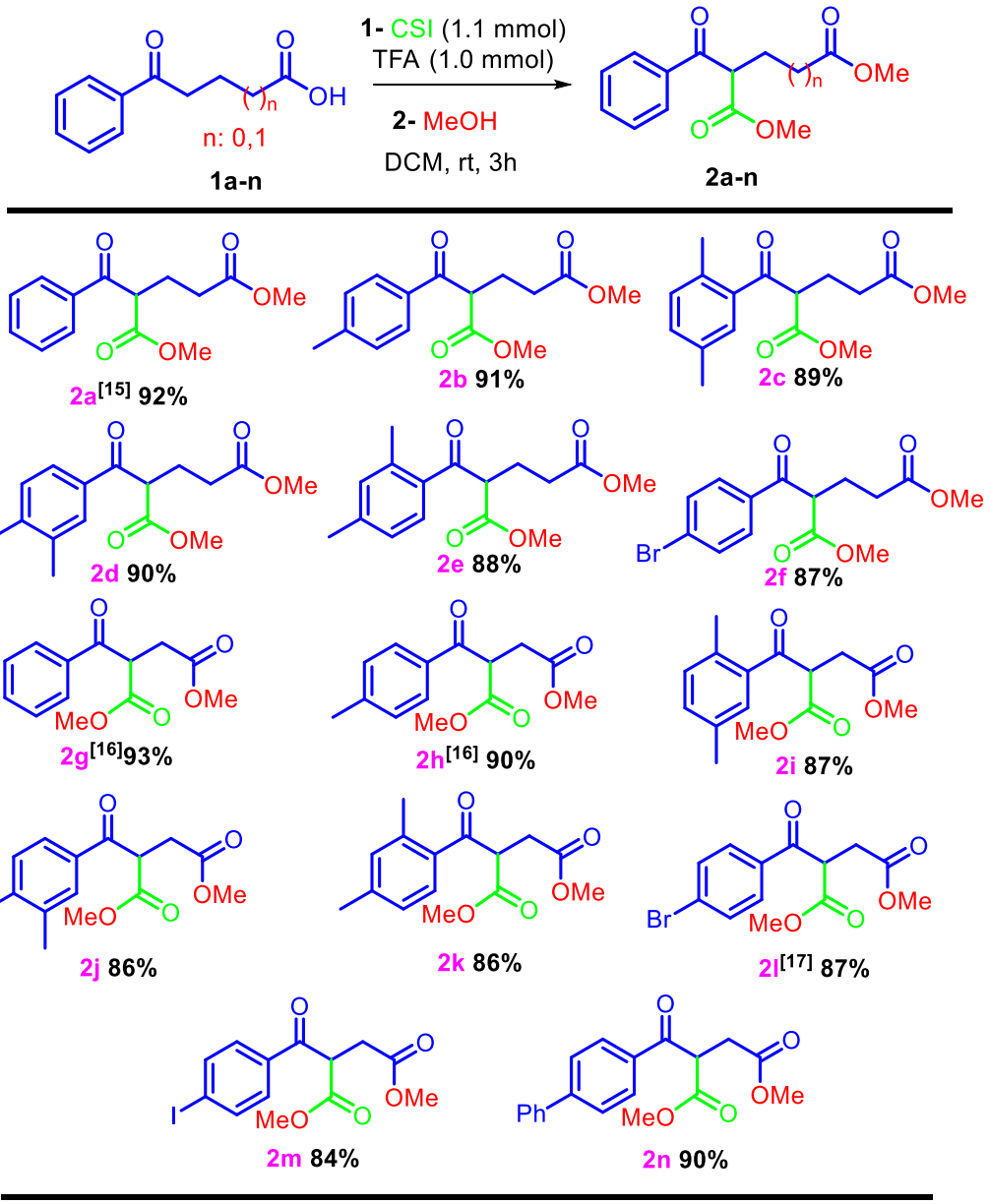


entry	Solvents	Acid or Base[a]	Yield[b] (%)
1	Acetonitrile	-	42
2		TfOH	53
3		TFA	62
4		NEt_3	28
5		Pyridine	17
6	**CH_2Cl_2**	-	54
7		TfOH	69
8		**TFA**	**92**
9		NEt_3	29
10		Pyridine	23

[a] 1 equivalent, [b]Isolated yield

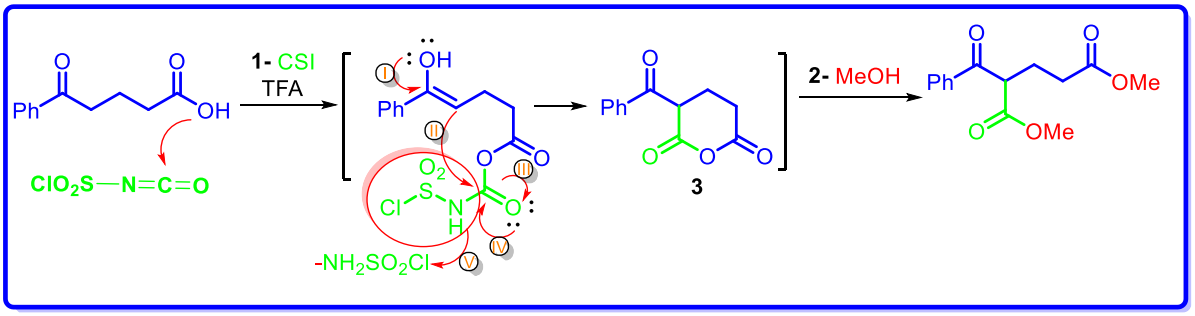
Thus, the optimized reaction condition for alternative and novel intramolecular synthesis of β-keto esters from 4- and 5-oxocarboxylic acid derivatives was concluded finally to be the use DCM as solvent with TFA as additive under mild conditions at room temperature for 3 h.

Table 2. Novel synthesis of β-keto esters from carboxylic acids using TFA[a,b,c]



[a]Reaction conditions: 1.0 mmol of Carboxylic acid (**1a-n**), 1.0 mmol TFA, 1.1 mmol of CSI, 10 mL DCM, reaction was stirred for 2h and added 2 mL MeOH for 1h, [b]Isolated yield, [c]Lit.

With the above optimal reaction conditions in hand, we examined the scope of 4- and 5-oxo carboxylic acid derivatives (**1a-n**) as the reaction partners. As shown in Table 2, the reactions of 4- and 5-oxo carboxylic acid derivatives bearing electron donating groups (4−methyl, 2,5−dimethyl, 3,4−dimethyl, 2,4−dimethyl and, 4-phenyl) with CSI proceeded smoothly to furnish **2a**[16] (92%), **2b** (91%), **2c** (89%), **2d** (90%), **2e** (88%), 2g[16] (93%), **2h**[17] (90%), **2i** (87%), **2j** (86%), **2k** (86%), and **2n** (90%), respectively. The halogen substituted 4- and 5-oxo-carboxylic acid derivatives (4−Br, 4−I) were observed also good reactivities, and the novel β-keto ester derivative **2f** was obtained in 87% yield. **2l**[18] and **2m** were isolated in 87% and 84% yield, respectively, when 4-(4-bromophenyl)-4-oxobutanoic acid (**1l**) and 4-(4-iodophenyl)-4-oxobutanoic acid (**1m**) were subjected to the reaction with CSI.



Scheme 2. Possible mechanism for synthesis of β-keto ester

In the first step, OH group of carboxylic acid attacks to carbonyl group of isocyanate as nucleophilic. Intermadiate carbamate is being tautomerization (**I**) and following the intramolecular attack of *α*-carbon to carbonyl carbamate (**II, III and IV**). Then, sulfamoyl chloride (NH_2SO_2Cl) leave from relating molecule to form cyclic anhydride (**V**). Finally, β-keto ester was occured by ring opening reaction of cyclic anhydride (**3**).

Conclusions

A pratical novel alternative method, one-pot synthesis β-keto esters with chlorosulfonyl isocyanate from 4- and 5-oxocarboxylic acid derivatives, were developed in high yields under mild conditions. This novel method highly useful environmentally and benign for organic chemists and pharmaceutical industry without any metal or oxidations, and easy to handle, under mild conditions, short reaction time. Moreover, a wide variety of β-keto esters can be obtained in good yields.

Experimental Section

General. Solvents are commercially available and used without further purification. 4- and 5-oxo-carboxylic acid derivatives were synthesized as in the literature.[19] ^{1}H and ^{13}C NMR spectra were recorded a Bruker 400 MHz in $CDCl_3$ with and NMR shifts are presented as δ in ppm. FTIR spectras were mesaured with a Perkin Elmer spectrophotometers in CH_2Cl_2 and by solutions in 0.1mm cells. High resolution mass spectra (HRMS) were obtained with a AB-Sciex 4600 QTOF MS spectrometer.

General procedure Synthesis of β-Keto ester. Carboxylic acid (**1a-n**) (1.0 eq) was dissolved in 10 mL DCM. The reaction mixture was added CSI (1.1 eq) and TFA (1.0 eq) and stirred for 2 h at room temperature. Then, it was added 2 mL MeOH and stirred for 1h. The reaction mixture was extracted with dichloromethane. The organic extract was dried over sodium sulfate, filtrate and evaporation in vacuo. The resulting residue was purified by thin-layer chromatography (TLC) on silica gel.

Dimethyl 2-benzoylpentanedioate (2a). Yellowish oil (316 mg, yield 92%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.30-2.35 (m, 2H), 2.46-2.50 (m, 2H), 3.69 (s, 3H), 3.71 (s, 3H), 4.55 (t, *J* 7.2 Hz, 1H), 7.50-7.53 (m, 2H), 7.60-7.64 (m, 1H), 8.04-8.06 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 24.0, 31.2, 51.7, 52.5, 52.6, 128.7, 128.8, 133.7, 135.9, 170.0, 173.2, 194.9; IR (CH_2Cl_2, cm^{-1}): 3003, 2955, 2849, 1737, 1689, 1596, 1436, 1333, 1272, 1161; HRMS (ESI) calcd. for $C_{14}H_{15}O_5$ $[M - H]^-$ 263.0925; found: 263.0916.

Dimethyl 2-(4-methylbenzoyl)pentanedioate (2b). Yellowish oil (305 mg, yield 91%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.28-2.32 (m, 2H), 2.42-2.47 (m, 5H), 3.67 (s, 3H), 3.68 (s, 3H), 4.50 (t, *J* 7.2 Hz, 1H), 7.28 (d, *J* 8.0, 2H), 7.92 (d, *J* 8.0 Hz, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.7, 24.0, 51.7, 52.4, 52.5, 128.9, 129.5, 133.4, 144.7, 170.1, 173.2, 194.4; IR (CH_2Cl_2, cm^{-1}): 3010, 2942, 2855, 1748, 1690, 1592, 1440, 1328, 128, 1153; HRMS (ESI−) calcd. for $C_{14}H_{15}O_5$ $[M - H]^-$ 263,0925; found: 263,0916; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1088.

Dimethyl 2-(2,5-dimethylbenzoyl)pentanedioate (2c). Yellowish oil (294 mg, yield 89%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.25-2.28 (m, 2H), 2.35-2.44 (m, 8H), 3.67 (s, 6H), 4.40 (t, *J* 7.1 Hz, 1H), 7.12-7.15 (m, 1H), 7.19-7.21 (m, 1H), 7.49-7.51 (m, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 20.6, 23.9, 31.3, 33.1, 51.7, 52.4, 54.9, 129.2, 132.0, 132.6, 135.3, 135.9, 136.7, 170.1, 173.1, 198.4; IR (CH_2Cl_2, cm^{-1}): 2953, 1739, 1652, 1437, 1302, 1203, 1118; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ $[M - H]^-$ 291.1238; found: 291.1240.

Dimethyl 2-(3,4-dimethylbenzoyl)pentanedioate (2d). Yellowish oil (297 mg, yield 90%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.26-2.34 (m, 8H), 2.42-2.46 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.50 (t, *J* 7.2 Hz, 1H), 7.23 (d, *J* 7.8 Hz, 1H), 7.75 (d, *J* 7.8 Hz, 1H), 7.79 (s, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 19.8, 20.1, 24.1, 31.3, 51.7, 52.3, 52.5, 126.5, 129.8, 130.0, 133.8, 137.3, 143.5, 170.2, 173.2, 194.7; IR (CH_2Cl_2, cm^{-1}): 2956, 1740, 1686, 1611, 1439, 1286, 1169; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ $[M - H]^-$ 291.1238; found: 291.1224.

Dimethyl 2-(2,4-dimethylbenzoyl)pentanedioate (2e). Yellowish oil (292 mg, yield 88%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.25-2.29 (m, 2H), 2.33-2.48 (m, 8H), 3.66 (s, 3H), 3.68 (s, 3H), 4.41 (t, *J* 7.2 Hz, 1H), 7.08 (s, 1H), 7.10 (s, 1H), 7.66 (d, *J* 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.4, 24.0, 51.7, 52.4, 54.6, 126.5, 129.3, 133.1, 133.7, 139.7, 142.7, 170.2, 142.7, 170.3, 173.2, 197.5; IR (CH_2Cl_2, cm^{-1}): 2953, 1742, 1663, 1442, 1328, 1243, 1136; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ $[M - H]^-$ 291.1238; found: 291.1215.

Dimethyl 2-(4-bromobenzoyl)pentanedioate (2f). Yellowish oil (275 mg, yield 87%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.26-2.30 (m, 2H), 2.43-2.47 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.49 (t, *J* 7.2 Hz, 1H), 7.63 (d, *J* 8.5 Hz, 2H), 7.90 (d, *J* 8.5 Hz, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 23.4, 31.1, 33.0, 51.7, 52.4, 129.1, 130.2, 132.2, 134.6, 169.8, 173.2, 193.9; IR (CH_2Cl_2, cm^{-1}): 2952, 1735, 1686, 1583, 1435, 1329, 1272, 1171; HRMS (ESI) calcd. for $C_{14}H_{14}BrO_5$ $[M - H]^-$ 341.0030; found: 341.0035.

Dimethyl 2-benzoylsuccinate (2g). Yellowish oil (327 mg, yield 93%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.03-3.14 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.89 (t, *J* 7.2 Hz, 1H), 7.48 (d, *J* 7.8 Hz, 2H), 7.58-7.61 (m, 1H), 8.04 (d, *J* 7.3 Hz, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.1, 49.3, 52.1, 52.9, 128.8, 128.9, 133.8, 135.8, 169.2, 171.7, 194.0; IR (CH_2Cl_2, cm^{-1}): 3003, 2955, 2849, 1737, 1689, 1596, 1436, 1333, 1272, 1161; HRMS (ESI) calcd. for $C_{13}H_{14}O_5$ $[M - H]^-$ 250.0847; found: 250.0832.

Dimethyl 2-(4-methylbenzoyl)succinate (2h). Yellowish oil (309 mg, yield 90%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.42 (s, 3H), 3.05-3.08 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.87 (t, *J* 7.2 Hz, 1H), 7.29 (d, *J* 8.2 Hz, 2H), 7.93 (d, *J* 8.2 Hz, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 28.9, 33.1, 49.2, 52.1, 52.8, 129.1, 129.5, 133.3, 144.8, 169.3, 171.8, 193.5; IR (CH_2Cl_2, cm^{-1}): 2953, 2826, 1737, 682, 1607, 1436, 1255, 1165; HRMS (ESI) calcd. for $C_{14}H_{15}O_5$ $[M - H]^-$ 263.0925; found: 263.0904.

Dimethyl 2-(2,5-dimethylbenzoyl)succinate (2i). Yellowish oil (293 mg, yield 87%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.37 (s, 3H), 2.41 (s, 3H), 3.01-3.11 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.76 (t, *J* 7.2 Hz, 1H), 7.23-7.25 (m, 1H), 7.76-7.80 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 20.4, 21.0, 32.9, 51.9, 52.1, 52.7, 129.3, 131.9, 132.6, 135.3, 135.9, 136.7, 169.3, 171.8, 197.5; IR (CH_2Cl_2, cm^{-1}): 2951, 1734, 1680, 1432, 1272, 1170; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1090.

Dimethyl 2-(3,4-dimethylbenzoyl)succinate (2j). Yellowish oil (290 mg, yield 86%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.32 (s, 6H), 2.98-3.11 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.87 (t, *J* 7.2 Hz, 1H), 7.11-7.22 (m, 2H), 7.58 (s, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 19.8, 20.1, 33.1, 49.1, 52.1, 52.8, 126.7, 129.9, 130.0, 133.6, 137.2,

143.6, 169.4, 171.8, 193.8; IR (CH_2Cl_2, cm^{-1}): 2954, 1738, 1684, 1605, 1438, 1276, 1174; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1054.

Dimethyl 2-(2,4-dimethylbenzoyl)succinate (2k). Yellowish oil (291 mg, yield 86%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.35 (s, 3H), 2.46 (s, 3H), 2.94-3.11 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.77 (t, *J* 6.6 Hz, 1H), 7.08-7.11 (m, 2H), 7.74 (d, *J* 7.8, Hz, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.3, 21.4, 33.1, 51.6, 52.1, 52.7, 126.4, 129.4, 133.0, 133.7, 139.6, 142.7, 169.5, 171.8, 196.6; IR (CH_2Cl_2, cm^{-1}): 2955, 2893, 1741, 1683, 1439, 1203, 1173; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1074.

Dimethyl 2-(4-bromobenzoyl)succinate (2l). Yellowish oil (279 mg, yield 87%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.01-3.17 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.82 (dd, *J* 6.2, 8.1 Hz, 1H), 7.63-7.65 (m, 2H), 7.89-7.92 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.0, 49.1, 52.2, 53.0, 129.1, 130.4, 132.1, 134.6, 168.8, 171.7, 193.1; IR (CH_2Cl_2, cm^{-1}): 2954, 1737, 1688, 1585, 1437, 1331, 1274, 1173; HRMS (ESI) calcd. for $C_{13}H_{12}BrO_5$ $[M - H]^-$ 326.9874; found: 326.9863.

Dimethyl 2-(4-iodobenzoyl)succinate (2m). Yellowish oil (260 mg, yield 84%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.99-3.16 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.81 (dd, *J* 6.2 , 8.2 Hz, 1H), 7.73-7.76 (m, 2H), 7.85-7.88 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.0, 49.1, 52.2, 53.0, 102.1, 130.2, 135.2, 138.1, 168.8, 171.7, 193.5; IR (CH_2Cl_2, cm^{-1}): 2952, 2833, 1737, 1687, 1581, 1437, 1272, 1071; HRMS (ESI) calcd. for $C_{13}H_{12}IO_5$ $[M - H]^-$ 374.9735; found: 374.9732.

Dimethyl 2-([1,1'-biphenyl]-4-carbonyl)succinate (2n). Yellow solid (289 mg, yield 90%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.03-3.18 (m, 2H), 3.68 (s, 3H), 3.70 (s, 3H), 4.93 (t, *J* 7.2 Hz, 1H), 7.41-7.50 (m, 3H), 7.62-7.73 (m, 4H), 8.11-8.14 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.1, 49.2, 52.2, 52.9, 127.3, 127.4, 128.4, 129.0, 129.6, 134.5, 139.7, 146.5, 169.2, 171.8, 193.6; IR (CH_2Cl_2, cm^{-1}): 2953, 1736, 682, 603, 1436, 1274, 1172; HRMS (ESI) calcd. for $C_{19}H_{17}O_5$ $[M - H]^-$ 325.1081; found: 325.1071.

3-Benzoyldihydro-2H-pyran-2,6(3H)-dione (3). Reaction mixture ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.31-2.47 (m, 2H), 2.93-2.96 (m, 2H), 4.92 (t, *J* 5.3 Hz, 1H), 7.53-7.59 (m, 2H), 7.67-7.73 (m, 1H), 7.96-7.98 (m, 2H).

Acknowledgements

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Supplementary Material

Experimental details, ^{1}H- and ^{13}C-NMR spectra for the products are provided in Supporting Information File.

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