Synthesis W. Ding et al. Paper

One-Pot Synthesis of α,β-Unsaturated Esters, Ketones, and Nitriles from Alcohols and Phosphonium Salts

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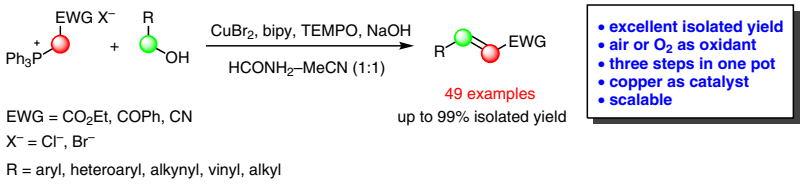


Received: 17.07.2017
Accepted after revision: 10.08.2017
Published online: 12.09.2017
DOI: 10.1055/s-0036-1590904; Art ID: ss-2017-f0451-op

Abstract A general method for the synthesis of α,β-unsaturated esters, ketones, and nitriles is successfully achieved by a one-pot copper-catalyzed oxidation with O_2 in air as oxidant. The solvent mixture of acetonitrile and formamide (1:1) is optimized to ensure the oxidation of alcohols, deprotonation of phosphonium salt, and Wittig reaction occur efficiently in one pot. A broad range of substrates has been explored for this process, including three electron-withdrawing group (CO_2Et, COPh, CN) functionalized phosphonium salts. They reacted not only with benzylic and heteroaromatic alcohols, but also with aliphatic alcohols, forming the corresponding α,β-unsaturated esters, ketones, and nitriles in moderate to excellent yields.

Key words oxidation of alcohols, phosphonium salts, tandem reaction, copper catalyst, Wittig reaction, carbon–carbon double bond

The Wittig olefination reaction[1] is a strategic route for the construction of C=C bonds in organic synthesis and it has had an enormous impact on the sophistication of the total synthesis of organic molecules.[2] However, the method has a great limitation when applied to aldehydes due to their instability, volatility, or unavailability. To circumvent the use of unstable or unavailable aldehydes, the direct one-pot oxidation–Wittig reaction of alcohols with ylides is an attractive approach for the construction of C=C bonds. Since the one-pot Swern oxidation–Wittig reaction reported by Ireland and Norbeck,[3] several methods for the one-pot reaction of alcohols with ylides have been devised. These studies involved a large amount or a full equivalent of the oxidant, such as Dess–Martin, periodinane,[4] $BaMnO_4$,[5] MnO_2,[6] PCC,[7] SO_3·Py,[8] or $PhI(OAc)_2$, combined with TEMPO (cat.),[9] or NMO with catalytic amount of TPAP.[10] Some cases may result in many problems, such as the formation of hazardous/toxic byproducts. Certain oxidants themselves are not only hazardous but also difficult to handle and this further limits their large-scale applications.

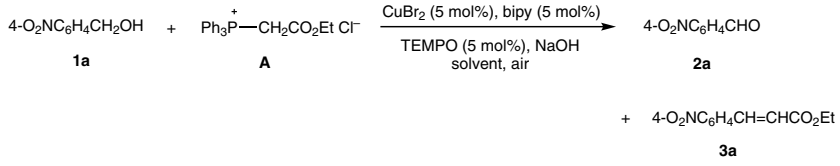
Oxygen is a more desirable oxidant from the standpoint of green chemistry, and the use of O_2 in the tandem oxidation–Wittig reaction has attracted increasing attention. Several methods have been reported, but drawbacks still remain. For example, Chang and co-workers[11] and Park and co-workers[12] both reported the ruthenium-catalyzed oxidation–Wittig reaction, where noble metal catalyst Ru was used and certain target products had low to moderate yields. Williams and co-workers reported that *N*,*N*,*N*′,*N*′-tetramethylenediamine dioxide ($TMEDAO_2$) could be used as co-oxidant for the tandem Ley–Griffith–Wittig reaction with stabilized ylides, where in some cases the sequential addition protocol was needed in order to increase reaction yields with the use of 2 equivalents of the ylide.[13] Recently, expensive metal catalyst gold/palladium bimetallic nanoparticles were utilized to catalyze the tandem process of aerobic oxidation and Horner–Wadsworth–Emmons olefination,[14] where 1.5 equivalents of triethyl phosphonoacetate and 3 equivalents of base were added to promote the reaction smoothly. Bera and co-workers reported that a diruthenium complex catalyzed the dehydrogenative coupling reactions of alcohols with Wittig reagents,[15] where the starting material for making the catalyst was commercially unavailable, and its preparation procedure was not easy. Therefore, developing a facile, efficient, and environmentally friendly synthesis of the C=C bond using the method of oxidation–Wittig tandem reaction is still highly desired.

Copper/TEMPO catalyst was reported by Semmelhack and co-workers in 1984 to be effective for the oxidation of benzylic and allylic alcohols.[16] Copper-based catalysts,[17] particularly those employing TEMPO[18] as the redox-active co-catalysts, have emerged as one of the most effective catalysts. However, an efficient and practical Cu-catalyzed oxi-

dation–Wittig tandem method using directly alcohols and ylide has yet to be developed. Meanwhile, the ylide, another starting material for the Wittig reaction, is usually provided from the deprotonation of a phosphonium salt with base. Whether we could use the phosphonium salt instead of the ylide as the starting material and therefore invoke three types of reaction (deprotonation of phosphonium salt, catalytic oxidation of alcohol, and Wittig reaction) in one pot inspired us to carry out this research. Herein, we report a facile one-pot synthesis of α,β-unsaturated esters, ketones, and nitriles directly from alcohols and a phosphonium salt.

Initially, in the model reaction of (2-ethoxy-2-oxoethyl)triphenylphosphonium chloride (**A**) and (4-nitrophenyl)methanol (**1a**) the catalyst was chosen as $CuBr_2$ (5 mol%) with bipyridine (5 mol%) and TEMPO (5 mol%) as the co-catalyst for the one-pot catalytic oxidation of the alcohol.[18] When we used *t*-BuOK or NaOH as a base to abstract proton from phosphonium salt **A**, and MeCN/H_2O (1:1) as the solvent, a poor (GC) yield of α,β-unsaturated ester **3a** was obtained with full conversion of **1a**, while 69% or 56% of 4-nitrobenzaldehyde (**2a**) remained (Table 1, entries 1 and 2), respectively. These results indicate that under these conditions the oxidation reaction occurred efficiently, but deprotonation or Wittig reaction was inefficient. Then we focused on the optimization of solvent based on the anticipation that the key to our idea is to find a suitable solvent which supports all three reactions in one pot. Results of the solvent screening are summarized in Table 1. The various solvents tested, such as MeCN, DMF, DMSO, 1,4-dioxane, THF, toluene, MeOH, and $HCONH_2$ (entries 3–10), all gave

Table 1 Solvent Screening for One-Pot Reaction[a]



Entry	Solvent	Yield (%)			Ratio *Z*/*E* of **3a**[b]
		1a	**2a**	**3a**	
1[c]	MeCN/H_2O (1:1)	–	69	31	29:71
2	MeCN/H_2O (1:1)	–	56	44	27:73
3	MeCN	57	–	43	21:79
4	DMF	3	54	43	0:100
5	DMSO	14	13	73	22:78
6	1,4-dioxane	67	33	–	–
7	THF	23	77	–	–
8	toluene	25	46	29	14:86
9	MeOH	7	93	–	–
10	$HCONH_2$	–	47	53	30:70
11	DMF/MeCN (1:1)	32	3	65	12:88
12	DMSO/MeCN (1:1)	13	5	82	11:89
13	dioxane/MeCN (1:1)	57	12	31	13:87
14	THF/MeCN (1:1)	3	53	44	14:86
15	toluene/MeCN (1:1)	14	46	40	13:87
16	$HCONH_2$/MeCN (1:1)	–	1	99	21:79
17	$HCONH_2$/MeCN (1:2)	9	31	60	32:68
18	$HCONH_2$/MeCN (2:1)	–	36	64	30:70
19[d]	$HCONH_2$/MeCN (1:1)	–	58	42	31:69
20[e]	$HCONH_2$/MeCN (1:1)	2	25	73	38:62

[a] Reaction conditions: 100 mL Schlenk tube, alcohol **1a** (1.0 mmol), **A** (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipyridine (bipy; 5 mol%), TEMPO (5 mol%), NaOH (1.1 mmol, 1.1 equiv), solvent (2 mL), 65 °C, stirring, air balloon, 24 h.
[b] The *Z*/*E* ratio was determined by GC-MS.
[c] *t*-BuOK was used instead of NaOH.
[d] At 25 °C.
[e] At 40 °C.

the product of **3a** in an unsatisfactory yield. Then we examined the use of mixed solvents (entries 11–18). We found that the mixed solvent of MeCN/$HCONH_2$ (1:1) has the best performance. Increasing or decreasing the amount of MeCN or $HCONH_2$ all caused the yield of the reaction to decrease. The reaction temperature had no significant effect on the oxidation of alcohol which was converted into aldehyde with full conversion even at room temperature; however, it had a great influence on the Wittig reaction. With the decrease of the temperature, the yield of **3a** was reduced (entries 19 and 20).

Then, reactions with different NaOH loading were carried out; the results are shown in Table 2. It is clear that without the addition of NaOH, only a low yield of aldehyde was obtained, and no Wittig product was formed. Increasing the amount of NaOH resulted in higher yields of the target product **3a** with more conversion of alcohol. This result reveals that NaOH is very important for the one-pot reaction which abstracts a proton from the phosphonium salt yielding the ylide, and promotes the conversion of the alcohol into the aldehyde; 1.1 equivalents of NaOH are required to ensure all three reactions occur efficiently.

Table 2 NaOH Loading Effect on the One-Pot Reaction[a]

Entry	NaOH (equiv)	Yield (%)			Ratio *Z*/*E* of **3a**[b]
		1a	**2a**	**3a**	
1	–	92	8	–	–
2	0.1	72	19	9	22:78
3	0.5	32	28	40	35:65
4	0.8	20	–	80	25:75
5	1.0	7	–	93	26:74
6	1.1	–	–	100	21:79

[a] Reaction conditions: 100 mL Schlenk tube, alcohol **1a** (1.0 mmol), **A** (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipyridine (5 mol%), TEMPO (5 mol%), NaOH in solvent (2 mL), 65 °C, stirring, air balloon, 24 h.
[b] The *Z*/*E* ratio was determined by GC-MS.

The optimized reaction conditions were then applied to various primary alcohols; the results are shown in Table 3. The investigation indicates that those with reactive functional groups such as bromo, chloro, and fluoro were compatible with the optimized reaction conditions, and no dehalogenated product was detected by GC-MS. The substituent electronic effect of benzylic alcohols was not significant in the reactions. All the electron-rich (4-Me, and 4-MeO) and electron-deficient (4-NO_2, 4-F, 4-Cl, and 4-Br) benzylic alcohols reacted efficiently to give α,β-unsaturated esters in high yield. Moreover, substituent steric effect was not significant either, as more hindered *ortho*-substituted benzylic alcohols, such as 2-MeO and 2-Cl, reacted smoothly and formed the target products in high yield. In contrast to benzyl alcohols, heterobenzyl alcohols did not react efficiently (entries 16–20) and the products were isolated only in moderate yield. The only exception is pyridin-3-ylmethanol, which reacted efficiently in an excellent yield (entry 17).

Table 3 Synthesis of α,β-Unsaturated Esters from **A** with Alcohols[a]

A **3**

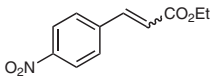
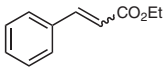
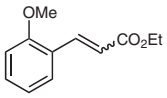
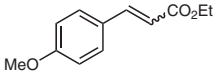
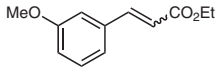
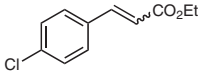
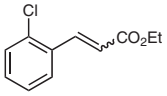
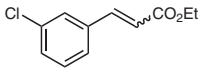
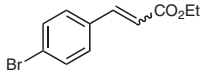
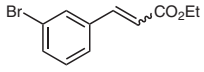
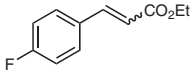
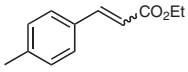
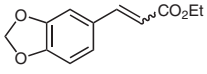
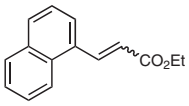
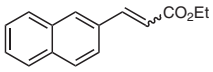
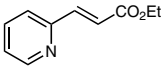
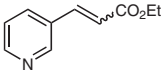
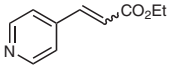
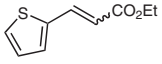
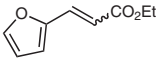
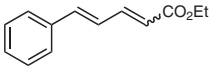
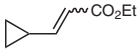
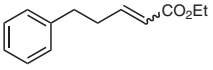
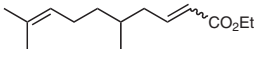
Entry	Product **3**	Yield (%)	Ratio *E*/*Z*[b]
1		97 (**3a**)	79:21
2		96 (**3b**)	86:14
3		82 (**3c**)	64:36
4		92 (**3d**)	85:15
5		95 (**3e**)	84:16
6		98 (**3f**)	85:15
7		90 (**3g**)	77:23
8		85 (**3h**)	81:19
9		91 (**3i**)	83:17
10		95 (**3j**)	81:19
11		99 (**3k**)	85:15
12		93 (**3l**)	86:14
13		96 (**3m**)	96:4

Table 3 (continued)

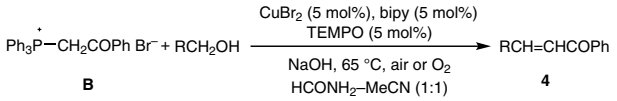
Entry	Product **3**	Yield (%)	Ratio *E*/*Z*[b]
14		95 (**3n**)	93:7
15		92 (**3o**)	93:7
16		56 (**3p**)	100:0
17		99 (**3q**)	86:14
18		69 (**3r**)	86:14
19		61 (**3s**)	95:5
20		71 (**3t**)	77:23
21[c]		69 (**3u**)	83:17
22[c]		84 (**3v**)	75:25
23[c]		87 (**3w**)	86:14
24[c]	$Me(CH_2)_6CH{=}CHCO_2Et$	72 (**3x**)	73:27
25[c]		76 (**3y**)	68:32

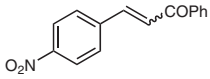
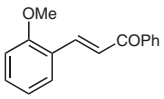
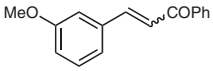
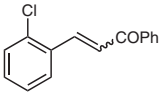
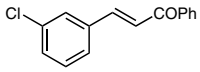
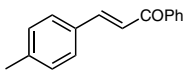
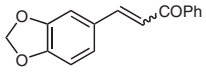
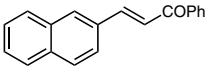
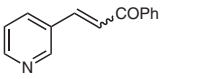
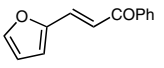
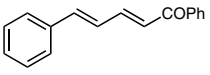
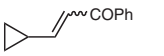
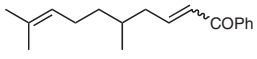
[a] Reaction conditions: 100 mL Schlenk tube, alcohol **1a** (1.0 mmol), **A** (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipyridine (5 mol%), TEMPO (5 mol%), NaOH (1.1 mmol, 1.1 equiv), solvent (2 mL), 65 °C, stirring, air balloon, 24 h.
[b] The *E*/*Z* ratio was determined by ^1H NMR [GC (**3u** and **3v**)].
[c] Under O_2 with 10 mol% $CuBr_2$.

It should be mentioned that aliphatic alcohols are also suitable substrates (Table 3, entries 21–25). When the tandem reactions were conducted under optimized reaction condition, the conversion of aliphatic alcohol into aldehyde was very low. However, the reactions with 10 mol% of $CuBr_2$ under an O_2 atmosphere were efficient and the corresponding products were obtained in moderate yields.

This method was then applied to the reaction of (2-oxo-2-phenylethyl)triphenylphosphonium bromide (**B**) and alcohols to synthesize the corresponding α,β-unsaturated ketones. As shown in Table 4, under optimized conditions the reaction took place smoothly and gave the unsaturated ketones in good to excellent yields. Similarly, the reactions of **B** with aliphatic alcohols conducted under O_2 conditions with 10 mol% of $CuBr_2$ formed the corresponding products in very good yields. It is remarkable that a highly *E*-selective olefination occurs with benzyl and heterobenzyl alcohols (**4a–i**), 3-phenylprop-2-en-1-ol (**4k**), and cyclopropylmeth-

Table 4 Synthesis of α,β-Unsaturated Ketones from **B** with Alcohols[a]



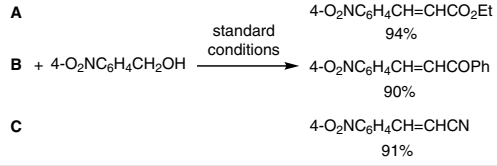
Entry	Product **4**	Yield (%)	Ratio *E*/*Z*[b]
1		95 (**4a**)	96:4
2		93 (**4b**)	93:7
3		95 (**4c**)	93:7
4		93 (**4d**)	97:3
5		93 (**4e**)	100:0
6		92 (**4f**)	100:0
7		96 (**4g**)	96:4
8		94 (**4h**)	100:0
9		95 (**4i**)	98:2
10		95 (**4j**)	100:0
11		89 (**4k**)	100:0
12[c]		86 (**4l**)	95:5
13[c]		82 (**4m**)	86:14

[a] Reaction conditions: 100 mL Schlenk tube, alcohol **1a** (1.0 mmol), **B** (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipyridine (5 mol%), TEMPO (5 mol%), NaOH (1.1 mmol, 1.1 equiv), solvent (2 mL), 65 °C, stirring, air balloon, 24 h.
[b] The *E*/*Z* ratio was determined by ^1H NMR or GC-MS.
[c] Under O_2 with 10 mol% $CuBr_2$.

anol (**4l**) producing *E*-vinyl ketones. Aliphatic alcohols participated in the reaction with **B** to give *E*-vinyl ketones with moderate selectivity (**4m**).

We have also examined the reaction of (cyanomethyl)triphenylphosphonium chloride (**C**) and alcohols to prepare the corresponding α,β-unsaturated nitriles. As shown in Table 5, the target products were obtained in excellent yield. Unfortunately, aliphatic alcohols were not suitable, and they could not be oxidized into aldehydes in the presence of **C** even under O_2 conditions and in the presence of a large amount of $CuBr_2$. It should be noted that the selectivity of isomers was not good. Different from the result obtained with **A** and **B** in Tables 3 and 4, *Z*-isomers were dominant in the two isomers products of α,β-unsaturated nitriles with moderate *Z*/*E* selectivity. 3-Methoxybenzylic alcohol was an exception as compound **5c** was formed as a mixture of two isomers (*Z*/*E* 40:60). It remains unclear why *Z*-isomer formation for the reaction of **C** with alcohols is the dominant product.

Finally, gram-scale experiments for the one-pot reaction for three kinds of phosphonium salts **A–C** were carried out. Thus, as shown in Scheme 1, in the cases of 5-mmol scale reactions under the standard conditions **3a**, **4a**, and **5a** were afforded in 94%, 90%, and 91% isolated yields, respectively, after 72 hours. The result clearly revealed the generality, practicality, and synthetic potential of this method.

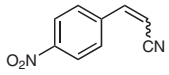
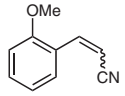
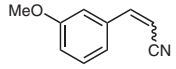
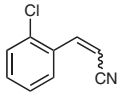
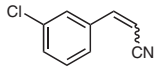
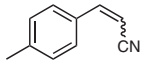
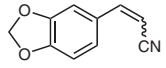
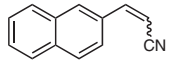
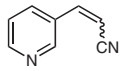
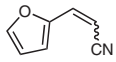
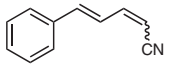


Scheme 1 Gram-scale for one-pot reaction for three kinds of phosphonium salts

A one-pot copper-catalyzed synthesis which includes aerobic oxidation of alcohols, deprotonation of phosphonium salts, and Wittig reaction was developed in this study, leading to high-yield production of α,β-unsaturated esters, ketones, and nitriles. Besides the mild reaction conditions and high isolated yields, this new procedure utilizes readily available starting materials and is valid for a broad range of substrates, making it very practical and convenient. Further studies of this catalytic aerobic synthesis using other phosphonium salts are underway.

Phosphonium salts were prepared through the reaction of Ph_3P with the corresponding halides, and other reagents were purchased from commercial suppliers and used without further purification. Column chromatography was carried out with silica gel (300–400 mesh). NMR spectra were collected on 500 MHz spectrometers at r.t. in $CDCl_3$ referenced to TMS (δ = 0). IR spectra were recorded on Perkin Elmer TG-IR-GC/MS.

Table 5 Synthesis of α,β-Unsaturated Nitriles from **C** with Alcohols[a]

Entry	Product **5**	Yield (%)	Ratio *Z*/*E*[b]
1		97 (**5a**)	60:40
2		96 (**5b**)	85:15
3		95 (**5c**)	40:60
4		94 (**5d**)	76:24
5		91 (**5e**)	60:40
6		98 (**5f**)	61:39
7		95 (**5g**)	48:52
8		95 (**5h**)	56:44
9		93 (**5i**)	61:39
10		88 (**5j**)	56:44
11		90 (**5k**)	56:44

[a] Reaction conditions: 100 mL Schlenk tube, alcohol **1a** (1.0 mmol), **C** (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipyridine (5 mol%), TEMPO (5 mol%), NaOH (1.1 mmol, 1.1 equiv), solvent (2 mL), 65 °C, stirring, air balloon, 24 h.
[b] The *Z*/*E* ratio was determined by ^1H NMR or GC-MS.

α,β-Unsaturated Esters 3, α,β-Unsaturated Ketones 4, and α,β-Unsaturated Nitriles 5; General Procedure

Alcohol (1 mmol), phosphonium salt (1.1 mmol), NaOH (1.1 mmol), $CuBr_2$ (5 mol%), 2,2′-bipy (5 mol%), and TEMPO (5 mol%) were mixed in MeCN and $HCONH_2$ (1:1, 2 mL) in a 100-mL Schlenk tube with an air balloon, and the mixture was stirred at 65 °C for 24 h (monitoring by TLC and/or GC-MS). After completion, product was purified by column chromatography (EtOAc and petroleum ether).

Ethyl 3-(4-Nitrophenyl)acrylate (3a)[19]

White solid; yield: 214 mg (97%); mp 134–136 °C (Lit.[20] 138 °C).

IR: 3110.2, 3077.3, 1713.1, 1642.7, 1595.3, 1517.8, 1344.6, 1187.9, 1026.8, 977.9, 843.8, 757.8, 717.1 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.24 (d, *J* = 8.5 Hz, 2 H), 7.70–7.69 (m, 3 H), 6.57 (d, *J* = 16.0 Hz, 1 H), 4.30 (q, *J* = 7.0 Hz, 2 H), 1.36 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.8, 148.3, 141.4, 140.4, 128.5, 124.0, 122.4, 60.8, 14.1.

MS (EI): *m*/*z* (%) = 221 (22), 204 (12), 193 (51), 192 (10), 191 (10), 177 (12), 176 (100), 146 (18), 130 (50), 118 (13), 102 (30), 91 (11), 84 (11), 82 (16), 76 (16).

Ethyl 3-Phenylacrylate (3b)[19]

Colorless liquid; yield: 169 mg (96%).

IR: 3107.1, 3074.2, 2981.9, 1711.5, 1538.2, 1450.3, 1366.8, 1310.6, 1269.6, 1173.9, 1037.9, 980.7, 855.8, 767.9, 712.1, 685.6 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.68 (d, *J* = 16.0 Hz, 1 H), 7.50–7.48 (m, 2 H), 7.35–7.34 (m, 3 H), 6.43 (d, *J* = 16.0 Hz, 1 H), 4.25 (q, *J* = 7.5 Hz, 2 H), 1.31 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.6, 144.2, 134.3, 129.9, 128.6, 127.8, 118.0, 60.1, 14.0.

MS (EI): *m*/*z* (%) = 176 (32), 148 (15), 147 (14), 132 (11), 131 (100), 104 (13), 103 (43), 102 (10), 77 (23), 51 (9).

Ethyl 3-(2-Methoxyphenyl)acrylate (3c)[13]

Colorless liquid; yield: 169 mg (82%).

IR: 2979.5, 2907.1, 2834.4, 1707.3, 1631.3, 1438.8, 1453.8, 1316.2, 1247.9, 1160.1, 1027.1, 999.5, 751.8 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.99 (d, *J* = 16.0 Hz, 1 H), 7.49 (d, *J* = 8.0 Hz, 1 H), 7.34–7.23 (m, 1 H), 6.95–6.84 (m, 2 H), 6.52 (d, *J* = 16.0 Hz, 1 H), 4.25 (q, *J* = 7.0 Hz, 2 H), 3.85 (s, 3 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.6, 157.5, 139.1, 130.6, 128.1, 122.6, 119.9, 117.9, 110.3, 59.5, 54.6, 13.6.

MS (EI): *m*/*z* (%) = 206 (55), 176 (11), 175 (100), 162 (11), 161 (97), 147 (95), 146 (19), 133 (10), 132 (16), 131 (27), 119 (22), 118 (35), 105 (38), 103 (23), 91 (24), 90 (15), 89 (18), 79 (14), 77 (29), 63 (10), 51 (10).

Ethyl 3-(4-Methoxyphenyl)acrylate (3d)[14]

Colorless liquid; yield: 190 mg (92%).

IR: 2980.4, 2839.8, 1705.3, 1533.8, 1508.7, 1250.4, 1160.7, 1029.8, 983.0, 827.9 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.63 (d, *J* = 16.0 Hz, 1 H), 7.45 (d, *J* = 8.5 Hz, 2 H), 6.88 (d, *J* = 8.5 Hz, 2 H), 6.30 (d, *J* = 16.0 Hz, 1 H), 4.24 (q, *J* = 7.0 Hz, 2 H), 3.80 (s, 3 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0, 161.1, 144.0, 129.4, 126.9, 115.5, 114.1, 60.0, 55.1, 14.1.

MS (EI): *m*/*z* (%) = 206 (63), 178 (12), 162 (11), 161 (100), 134 (58), 133 (7), 118 (12), 90 (10), 89 (11), 77 (11).

Ethyl 3-(3-Methoxyphenyl)acrylate (3e)[5b]

Yellow liquid; yield: 196 mg (95%).

IR: 2981.2, 2837.3, 1709.3, 1638.0, 1580.9, 1255.7, 1164.1, 1087.4, 981.6, 856.8, 783.6, 679.8 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 16.0 Hz, 1 H), 7.28 (t, *J* = 8.0 Hz, 1 H), 7.11 (d, *J* = 8.0 Hz, 1 H), 7.03 (s, 1 H), 6.92 (dd, *J* = 2.5, 8.0 Hz, 1 H), 6.42 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 3.81 (s, 3 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.8, 159.8, 144.4, 135.7, 129.7, 120.6, 118.4, 116.0, 112.8, 60.4, 55.1, 14.2.

MS (EI): *m*/*z* (%) = 206 (65), 178 (13), 177 (13), 162 (17), 161 (100), 134 (20), 133 (26), 118 (21), 90 (12), 77 (10).

Ethyl 3-(4-Chlorophenyl)acrylate (3f)[19]

Colorless liquid; yield: 206 mg (98%).

IR: 2982.5, 2952.1, 1713.3, 1639.1, 1491.7, 1310.5, 1171.4, 1090.3, 1035.2, 821.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 16.0 Hz, 1 H), 7.41 (d, *J* = 8.5 Hz, 2 H), 7.32 (d, *J* = 8.5 Hz, 2 H), 6.38 (d, *J* = 16.0 Hz, 1 H), 4.25 (q, *J* = 7.0 Hz, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.4, 142.8, 135.8, 132.8, 129.0, 128.9, 118.7, 60.3, 14.1.

MS (EI): *m*/*z* (%) = 210 (34), 182 (22), 167 (33), 166 (11), 165 (100), 139 (14), 138 (20), 137 (35), 131 (10), 102 (36), 101 (31), 75 (17), 51 (10).

Ethyl 3-(2-Chlorophenyl)acrylate (3g)[5b]

Colorless liquid; yield: 189 mg (90%).

IR: 3057.0, 2982.7, 1714.3, 1635.2, 1471.5, 1442.9, 1355.9, 1314.6, 1258.3, 1177.6, 1035.9, 980.0, 760.3 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.08 (d, *J* = 7.0 Hz, 1 H), 7.59 (dd, *J* = 7.5, 2.0 Hz, 1 H), 7.39 (dd, *J* = 7.5, 2.0 Hz, 1 H), 7.30–7.21 (m, 2 H), 6.42 (d, *J* = 16.0 Hz, 1 H), 4.27 (q, *J* = 7.0 Hz, 2 H), 1.34 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.2, 140.1, 134.7, 132.6, 130.8, 130.0, 127.4, 126.9, 120.8, 60.5, 14.1.

MS (EI): *m*/*z* (%) = 210 (12), 175 (52), 167 (10), 165 (31), 148 (9), 147 (100), 137 (22), 103 (18), 102 (20), 101 (27), 75 (15), 51 (8).

Ethyl 3-(3-Chlorophenyl)acrylate (3h)[5b]

Colorless liquid; yield: 179 mg (85%).

IR: 3055.0, 2982.3, 1712.6, 1640.7, 1556.3, 1311.9, 1269.9, 1199.9, 1177.2, 880.8, 860.3, 786.7, 673.6 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.59 (d, *J* = 16.0 Hz, 1 H), 7.48 (s, 1 H), 7.43–7.27 (m, 3 H), 6.42 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.3, 142.7, 136.1, 134.7, 129.9, 129.8, 127.6, 126.0, 119.6, 60.5, 14.1.

MS (EI): *m*/*z* (%) = 212 (10), 210 (34), 182 (20), 181 (11), 167 (33), 166 (11), 165 (100), 139 (12), 138 (14), 137 (33), 131 (12), 102 (39), 101 (28), 75 (16), 51 (10).

Ethyl 3-(4-Bromophenyl)acrylate (3i)[13]

Colorless liquid; yield: 231 mg (91%).

IR: 2981.0, 2930.2, 1712.5, 1637.7, 1587.7, 1488.2, 1310.2, 1270.3, 1171.8, 1072.2, 818.3, 743.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 16.0 Hz, 1 H), 7.51–7.47 (m, 2 H), 7.36 (d, *J* = 8.5 Hz, 2 H), 6.41 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.5, 143.0, 132.3, 132.0, 129.3, 124.3, 118.9, 60.5, 14.2.

MS (EI): *m*/*z* (%) = 210 (8), 175 (52), 148 (10), 147 (100), 137 (23), 103 (23), 102 (25), 101 (32), 75 (22), 51 (13).

Ethyl 3-(3-Bromophenyl)acrylate (3j)[5b]

Yellow liquid; yield: 241 mg (95%).

IR: 3037.2, 2981.3, 1712.0, 1539.9, 1551.0, 1311.2, 1176.8, 1085.3, 980.1, 880.0, 784.7, 669.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.65 (s, 1 H), 7.59 (d, *J* = 16.0 Hz, 1 H), 7.49–7.41 (m, 2 H), 7.25–7.21 (m, 1 H), 6.42 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.3, 142.6, 136.4, 132.8, 130.5, 130.2, 126.4, 122.8, 119.6, 60.5, 14.1.

MS (EI): *m*/*z* (%) = 255 (35), 254 (37), 227 (22), 225 (23), 210 (69), 208 (70), 182 (19), 181 (17), 147 (15), 131 (17), 130 (24), 129 (26), 103 (16), 102 (100), 101 (17), 76 (19), 75 (20), 51 (17).

Ethyl 3-(4-Fluorophenyl)acrylate (3k)[19]

Colorless liquid; yield: 192 mg (99%).

IR: 3028.7, 2983.6, 1712.0, 1639.7, 1600.6, 1509.8, 1314.1, 1231.7, 1160.4, 1096.1, 1035.8, 982.4, 831.5 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.64 (d, *J* = 16.0 Hz, 1 H), 7.51–7.48 (m, 2 H), 7.27–7.02 (m, 2 H), 6.35 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.6, 164.7, 162.7 (t, J_{C-F} = 241.8 Hz), 143.0, 130.6, 130.5 (d, J_{C-F} = 8.5 Hz), 117.9, 115.8 (d, J_{C-F} = 21.8 Hz), 60.3, 14.1.

MS (EI): *m*/*z* (%) = 194 (27), 166 (20), 150 (11), 149 (100), 122 (15), 121 (37), 101 (34), 75 (11).

Ethyl 3-(4-Tolyl)acrylate (3l)[14]

Colorless liquid; yield: 177 mg (93%).

IR: 2981.1, 2910.4, 1711.4, 1636.1, 1310.3, 1260.4, 1154.4, 1037.7, 984.0, 813.1 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 16.0 Hz, 1 H), 7.39 (d, *J* = 8.5 Hz, 2 H), 7.16 (d, *J* = 8.5 Hz, 2 H), 6.38 (d, *J* = 16.0 Hz, 1 H), 4.24 (q, *J* = 7.5 Hz, 2 H), 2.34 (s, 3 H), 1.32 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0, 144.4, 140.4, 131.6, 129.4, 127.9, 117.0, 60.2, 21.2, 14.2.

MS (EI): *m*/*z* (%) = 190 (46), 162 (15), 146 (11), 145 (100), 131 (8), 118 (21), 117 (32), 116 (11), 115 (34), 65 (7).

Ethyl 3-(1,3-Benzodioxol-5-yl)acrylate (3m)[19]

White solid; yield: 211 mg (96%); mp 57–60 °C (Lit.[13] 51–53 °C).

IR: 2985.9, 2908.6, 1702.4, 1631.8, 1604.0, 1492.1, 1447.1, 1236.6, 1175.3, 1032.6, 982.3, 928.2, 851.5, 806.2 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.59 (d, *J* = 16.0 Hz, 1 H), 7.03–6.99 (m, 2 H), 6.81 (d, *J* = 8.0 Hz, 1 H), 6.26 (d, *J* = 16.0 Hz, 1 H), 6.00 (s, 2 H), 4.25 (q, *J* = 7.5 Hz, 2 H), 1.33 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.2, 149.5, 148.3, 144.3, 128.9, 124.3, 116.2, 108.5, 106.5, 101.5, 60.4, 14.3.

MS (EI): *m*/*z* (%) = 218 (100), 174 (87), 145 (82), 147 (60), 175 (60), 148 (45), 144 (44), 146 (34), 173 (27), 220 (24).

Ethyl 3-(Naphthalen-1-yl)acrylate (3n)[5b]

Colorless liquid; yield: 215 mg (95%).

IR: 3059.9, 2980.9, 1710.2, 1633.3, 1304.9, 1262.9, 1166.8, 1040.7, 977.6, 799.5, 775.4 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.50 (d, *J* = 15.5 Hz, 1 H), 8.15 (d, *J* = 8.5 Hz, 1 H), 7.81 (t, *J* = 2.5 Hz, 2 H), 7.68 (d, *J* = 7.0 Hz, 1 H), 7.53–7.39 (m, 3 H), 6.49 (d, *J* = 16.0 Hz, 1 H), 4.29 (q, *J* = 7.0 Hz, 2 H), 1.35 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.7, 141.4, 133.5, 131.6, 131.2, 130.3, 126.7, 126.0, 125.3, 124.8, 123.2, 120.7, 60.4, 14.2.

MS (EI): *m*/*z* (%) = 226 (27), 181 (18), 154 (14), 153 (100), 152 (68), 151 (11), 76 (9).

Ethyl 3-(Naphthalen-2-yl)acrylate (3o)[21]

White solid; yield: 208 mg (92%); mp 70–72 °C (Lit.[22] 70–72 °C).

IR: 3064.1, 2982.0, 1707.7, 1633.9, 1174.4, 989.9, 822.3, 752.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.81–7.70 (m, 5 H), 7.55–7.54 (m, 1 H), 7.43–7.41 (m, 2 H), 6.49 (d, *J* = 16.0 Hz, 1 H), 4.26 (q, *J* = 7.0 Hz, 2 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0, 144.6, 134.2, 133.3, 132.0, 129.9, 128.7, 128.6, 127.8, 127.2, 126.7, 123.5, 118.5, 60.5, 14.4.

MS (EI): *m*/*z* (%) = 227 (13), 226 (75), 198 (15), 197 (13), 182 (16), 181 (100), 154 (45), 153 (63), 152 (76), 151 (18), 127 (13), 126 (9), 91 (9), 77 (12), 76 (26).

Ethyl (*E*)-3-(Pyridin-2-yl)acrylate (3p)[14]

Colorless liquid; yield: 99 mg (56%).

IR: 2982.3, 2908.0, 1711.6, 1545.5, 1582.1, 1317.1, 1297.6, 1260.6, 1203.6, 1161.1, 981.4, 785.3, 744.8, 599.5 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.64 (d, *J* = 4 Hz, 1 H), 7.73–7.67 (m, 2 H), 7.43 (d, *J* = 7.5 Hz, 1 H), 7.28–7.25 (m, 1 H), 6.92 (d, *J* = 16.0 Hz, 1 H), 4.28 (q, *J* = 7.0 Hz, 2 H), 1.34 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.5, 152.8, 149.9, 143.1, 136.6, 124.0, 123.9, 122.3, 60.5, 14.1.

MS (EI): *m*/*z* (%) = 177 (21), 148 (9), 133 (36), 132 (100), 105 (45), 104 (44), 79 (13), 78 (29), 51 (15).

Ethyl 3-(Pyridin-3-yl)acrylate (3q)[23]

Yellow liquid; yield: 175 mg (99%).

IR: 2982.5, 2905.2, 1711.7, 1541.3, 1310.9, 1254.8, 1170.7, 1025.7, 982.0, 805.5, 697.9, 629.9 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.75 (d, *J* = 2.5 Hz, 1 H), 8.60 (dd, *J* = 1.5, 5.0 Hz, 1 H), 7.85 (dt, *J* = 1.5, 8.0 Hz, 1 H), 7.68 (d, *J* = 16.0 Hz, 1 H), 7.33 (dd, *J* = 5.0, 7.5 Hz, 1 H), 6.53 (d, *J* = 16.0 Hz, 1 H), 4.28 (q, *J* = 7.5 Hz, 2 H), 1.35 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.9, 150.6, 149.3, 140.5, 133.8, 129.9, 122.4, 120.1, 60.4, 13.9.

MS (EI): *m*/*z* (%) = 177 (21), 148 (16), 133 (32), 132 (100), 131 (22), 120 (9), 105 (9), 104 (36), 78 (13), 77 (16), 51 (20).

Ethyl 3-(Pyridin-4-yl)acrylate (3r)

White solid; yield: 122 mg (69%); mp 64–65 °C.

IR: 3054.6, 2984.7, 2902.1, 1705.1, 1642.5, 1598.2, 1324.1, 1300.5, 1182.4, 1033.3, 992.9, 818.2, 558.2 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.65 (d, *J* = 6.5 Hz, 2 H), 7.60 (d, *J* = 16.0 Hz, 1 H), 7.37 (d, *J* = 6.5 Hz, 2 H), 6.61 (d, *J* = 16.0 Hz, 1 H), 4.29 (q, *J* = 7.5 Hz, 2 H), 1.35 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.7, 150.3, 141.4, 122.7, 121.5, 60.7, 14.0.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{10}H_{12}NO_2$: 178.0868; found: 178.0873.

Ethyl 3-(Thiophen-2-yl)acrylate (3s)[14]

Yellow liquid; yield: 111 mg (61%).

IR: 3105.5, 2981.6, 1705.7, 1525.8, 1369.5, 1305.2, 1251.5, 1160.8, 1039.7, 969.7, 855.0, 704.9 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 15.0 Hz, 1 H), 7.35 (d, *J* =5.0 Hz, 1 H), 7.23 (d, *J* = 3.5 Hz, 1 H), 7.03 (t, *J* = 4.5 Hz, 1 H), 6.23 (d, *J* = 16.0 Hz, 1 H), 4.24 (q, *J* = 7.5 Hz, 2 H), 1.31 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.7, 139.5, 136.9, 130.7, 128.3, 128.0, 126.4, 117.0, 60.4, 14.3.

MS (EI): *m*/*z* (%) = 182 (49), 154 (11), 138 (10), 137 (100), 110 (30), 109 (32), 65 (17).

Ethyl 3-(Furan-2-yl)acrylate (3t)[13]

Yellow liquid; yield: 118 mg (71%).

IR: 2982.8, 2911.4, 1711.1, 1538.4, 1368.7, 1304.3, 1260.7, 1209.9, 1163.3, 1018.5, 973.3, 752.0 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46 (s, 1 H), 7.42 (d, *J* = 15.5 Hz, 1 H), 6.59 (d, *J* = 3.0 Hz, 1 H), 6.46–6.45 (m, 1 H), 6.30 (d, *J* = 15.5 Hz, 1 H), 4.23 (q, *J* = 7.5 Hz, 2 H), 1.31 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0, 150.9, 144.6, 130.9, 115.9, 114.5, 112.2, 60.4, 14.3.

MS (EI): *m*/*z* (%) = 166 (51), 138 (39), 122 (8), 121 (100), 110 (12), 94 (37), 93 (9), 92 (7), 66 (10), 65 (37), 63 (7).

Ethyl 5-Phenylpenta-2,4-dienoate (3u)[19]

Colorless liquid; yield: 139 mg (69%).

IR: 2982.6, 2923.5, 1727.6, 1450.9, 1370.4, 1244.3, 1179.5, 1027.4, 859.5, 752.6, 699.1 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.47–7.43 (m, 3 H), 7.37–7.34 (m, 2 H), 7.32–7.31 (m, 1 H), 6.92–6.84 (m, 2 H), 5.99 (d, *J* = 15.5 Hz, 1 H), 4.23 (q, *J* = 7.0 Hz, 2 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0, 144.5, 140.3, 136.0, 129.0, 128.8, 127.1, 126.2, 121.3, 60.3, 14.3.

MS (EI): *m*/*z* (%) = 202 (17), 157 (18), 130 (13), 129 (100), 128 (56), 127 (16), 115 (6), 77 (7), 64 (5), 51 (6).

Ethyl 3-Cyclopropylacrylate (3v)[24]

Colorless liquid; yield: 118 mg (84%).

IR: 2830.2, 1722.0, 1672.7, 1063.4, 1041.9, 1010.4, 857.8 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.41 (dd, *J* = 10.0, 15.5 Hz, 1 H), 5.88 (d, *J* = 15.5 Hz, 1 H), 4.16 (q, *J* = 7.0 Hz, 2 H), 1.59–1.54 (m, 1 H), 1.27 (t, *J* = 7.0 Hz, 3 H), 0.95–0.91 (m, 2 H), 0.64–0.61 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.7, 152.9, 117.2, 59.0, 13.3, 13.2, 7.6.

MS (EI): *m*/*z* (%) = 140 (21), 125 (17), 112 (100), 111 (16), 97 (74), 95 (84), 84 (40), 67 (70), 66 (14), 65 (13).

Ethyl 5-Phenylpent-2-enoate (3w)[19]

Colorless liquid; yield: 177 mg (87%).

IR: 2981.8, 2934.0, 1718.0, 1653.5, 1453.6, 1313.7, 1255.3, 1192.4, 1057.4, 974.8, 747.7, 698.9 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32–7.26 (m, 2 H), 7.23–7.18 (m, 3 H), 7.05–6.99 (m, 1 H), 5.86 (d, *J* = 16.0 Hz, 1 H), 4.19 (q, *J* = 7.0 Hz, 2 H), 2.78 (t, *J* = 7.5 Hz, 2 H), 2.55–2.51 (m, 2 H), 1.29 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.5, 148.0, 140.8, 128.4, 128.3, 126.1, 121.8, 60.1, 34.3, 33.8, 14.2.

MS (EI): *m*/*z* (%) = 158 (7), 157 (6), 131 (7), 130 (24), 129 (5), 116 (4), 92 (8), 91 (100), 70 (5), 65 (7).

Ethyl Dec-2-enoate (3x)[25]

Yellow liquid; yield: 142 mg (72%).

IR: 2891.1, 1870.0, 1718.8, 1656.3, 1261.5, 1042.0, 858.7 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.99–6.93 (m, 1 H), 5.82–5.79 (m, 1 H), 4.17 (q, *J* = 7.5 Hz, 2 H), 2.21–2.16 (m, 2 H), 1.29–1.27 (m, 13 H), 0.87 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.8, 149.5, 121.2, 60.1, 32.2, 31.7, 29.1, 29.0, 28.0, 22.6, 14.3.

MS (EI): *m*/*z* (%) = 110 (40), 101 (82), 99 (39), 96 (33), 88 (39), 84 (43), 73 (68), 69 (56), 56 (31), 55 (100).

Ethyl 5,9-Dimethyldeca-2,8-dienoate (3y)[13]

Colorless liquid; yield: 181 mg (76%).

IR: 2963.9, 2923.8, 1721.0, 1656.6, 1455.3, 1377.3, 1253.3, 1181.5, 1043.5, 982.5, 808.3 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.96–6.90 (m, 1 H), 5.81–5.78 (m, 1 H), 5.07–5.06 (m, 1 H), 4.19–4.13 (m, 2 H), 2.66–2.53 (m, 1 H), 2.23–2.17 (m, 1 H), 2.06–1.92 (m, 2 H), 1.67 (s, 3 H), 1.64–1.61 (m, 1 H), 1.59 (s, 3 H), 1.36–1.31 (m, 2 H), 1.29–1.26 (m, 3 H), 0.91–0.89 (m, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.6, 148.1, 131.4, 124.4, 122.4, 60.1, 39.6, 36.7, 32.0, 25.7, 25.5, 19.5, 17.6.

MS (EI): *m*/*z* (%) = 136 (26), 109 (39), 95 (69), 93 (33), 86 (22), 81 (46), 74 (22), 69 (100), 67 (35), 55 (45).

3-(4-Nitrophenyl)-1-phenylprop-2-en-1-one (4a)[26]

Yellow solid; yield: 240 mg (95%); mp 163–164 °C (Lit.[27] 164.5 °C).

IR: 3110.2, 2932.6, 2853.6, 1577.3, 1528.0, 1347.0, 1110.2, 856.9, 745.1, 689.1 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.27 (d, *J* = 8.5 Hz, 2 H), 8.04 (d, *J* = 8.5 Hz, 2 H), 7.83–7.78 (m, 3 H), 7.66–7.61 (m, 2 H), 7.55–7.52 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 189.6, 148.6, 141.5, 141.0, 137.5, 133.3, 128.9, 128.8, 128.6, 125.8, 124.2.

MS (EI): *m*/*z* (%) = 251 (100), 250 (44), 234 (60), 204 (51), 178 (56), 177 (63), 176 (52), 175 (38), 105 (68), 77 (40).

3-(2-Methoxyphenyl)-1-phenylprop-2-en-1-one (4b)[26]

Yellow liquid; yield: 221 mg (93%).

IR: 3052.2, 2938.8, 2838.6, 1659.1, 1596.2, 1573.5, 1488.3, 1463.4, 1315.9, 1246.9, 1212.7, 1016.5, 988.8, 816.1, 751.2, 733.1, 693.2, 586.6 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 15.5 Hz, 1 H), 8.01 (d, *J* = 7.5 Hz, 2 H), 7.64–7.61 (m, 2 H), 7.56 (t, *J* = 7.0 Hz, 1 H), 7.48 (t, *J* = 7.5 Hz, 2 H), 7.36 (t, *J* = 7.5 Hz, 1 H), 6.98 (t, *J* = 7.5 Hz, 1 H), 6.92 (d, *J* = 7.5 Hz, 1 H), 3.89 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.2, 158.0, 139.5, 137.7, 131.7, 131.0, 128.4, 127.7, 127.6, 123.1, 122.0, 119.9, 110.5, 54.8.

MS (EI): *m*/*z* (%) = 236 (6), 207 (28), 206 (27), 205 (100), 204 (16), 177 (5), 161 (10), 160 (7), 105 (13), 77 (11).

3-(3-Methoxyphenyl)-1-phenylprop-2-en-1-one (4c)[28]

Yellow liquid; yield: 226 mg (95%).

IR: 3060.4, 2937.1, 2835.9, 1662.9, 1603.0, 1578.5, 1489.4, 1450.6, 1433.7, 1254.0, 1213.8, 1158.5, 1017.0, 981.8, 850.3, 770.2, 687.4, 574.4 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 7.0 Hz, 2 H), 7.78 (d, *J* = 15.5 Hz, 1 H), 7.59 (t, *J* = 7.5 Hz, 1 H), 7.51 (t, *J* = 7.5 Hz, 3 H), 7.36–7.32 (m, 1 H), 7.25 (d, *J* = 7.5 Hz, 1 H), 7.16 (t, *J* = 2.0 Hz, 1 H), 6.98–6.95 (m, 1 H), 3.86 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.6, 160.0, 144.8, 138.2, 136.3, 132.8, 129.9, 128.6, 128.5, 122.5, 121.1, 116.3, 113.5, 55.4.

MS (EI): *m*/*z* (%) = 237 (29), 236 (70), 235 (98), 234 (44), 207 (37), 206 (28), 205 (100), 161 (36), 105 (24), 77 (24).

3-(2-Chlorophenyl)-1-phenylprop-2-en-1-one (4d)[29]

Yellow liquid; yield: 225 mg (93%).

IR: 3063.0, 2928.4, 1654.2, 1605.2, 1469.7, 1444.9, 1314.8, 1271.7, 1214.5, 1014.1, 977.8, 750.5, 691.0, 656.6, 630.3 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.18 (d, *J* = 15.5 Hz, 1 H), 8.02 (d, *J* = 8.0 Hz, 2 H), 7.76–7.74 (m, 1 H), 7.59 (t, *J* = 7.5 Hz, 1 H), 7.52–7.47 (m, 3 H), 7.44–7.42 (m, 1 H), 7.34–7.29 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.3, 140.5, 137.8, 135.4, 133.2, 132.8, 131.1, 130.2, 128.6, 128.5, 127.7, 127.0, 124.7.

MS (EI): *m*/*z* (%) = 242 (14), 208 (17), 207 (100), 105 (15), 102 (10), 101 (13), 89 (8), 77 (24), 75 (6), 51 (10).

(*E*)-3-(3-Chlorophenyl)-1-phenylprop-2-en-1-one (4e)[28]

Yellow solid; yield: 225 mg (93%); mp 75–76 °C (Lit.[30] 75 °C).

IR: 3054.3, 2930.5, 1664.3, 1604.9, 1564.7, 1476.9, 1448.2, 1309.0, 1215.5, 1015.5, 974.8, 771.2, 685.3, 652.8, 577.8 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 8.5 Hz, 2 H), 7.72 (d, *J* = 15.5 Hz, 1 H), 7.61–7.60 (m, 2 H), 7.53–7.47 (m, 4 H), 7.37–7.32 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 189.9, 142.9, 137.9, 136.7, 134.9, 132.9, 130.2, 130.1, 128.6, 128.5, 127.8, 126.7, 123.2.

MS (EI): *m*/*z* (%) = 243 (28), 242 (73), 241 (51), 207 (100), 179 (30), 105 (76), 102 (48), 101 (33), 77 (86), 51 (32).

(*E*)-1-Phenyl-3-(4-tolyl)prop-2-en-1-one (4f)[26]

White solid; yield: 204 mg (92%); mp 95–97 °C (Lit.[27] 99 °C).

IR: 3024.7, 2922.7, 1656.3, 1597.3, 1340.5, 1222.0, 991.7, 817.4, 774.6, 689.1 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 7.0 Hz, 2 H), 7.81 (d, *J* = 16.0 Hz, 1 H), 7.60–7.56 (m, 3 H), 7.54–7.49 (m, 3 H), 7.23 (d, *J* = 7.5 Hz, 2 H), 2.40 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.6, 144.9, 141.0, 138.4, 132.6, 132.2, 129.7, 128.5, 128.4, 128.3, 121.1, 21.5.

MS (EI): *m*/*z* (%) = 220 (36), 219 (45), 207 (30), 206 (27), 205 (100), 178 (27), 177 (19), 145 (33), 144 (19), 115 (22).

3-(1,3-Benzodioxol-5-yl)-1-phenylprop-2-en-1-one (4g)[28]

Yellow solid; yield: 242 mg (96%); mp 120–122 °C (Lit.[31] 122 °C).

IR: 2924.5, 2857.0, 1657.5, 1588.0, 1487.9, 1447.0, 1307.8, 1251.8, 1215.5, 1105.5, 1018.5, 984.2, 918.4, 775.9, 698.8, 654.6, 598.6 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.00 (d, *J* = 7.0 Hz, 2 H), 7.73 (d, *J* = 15.5 Hz, 1 H), 7.57 (t, *J* = 8.0 Hz, 1 H), 7.49 (t, *J* = 8.0 Hz, 2 H), 7.37 (d, *J* = 15.5 Hz, 1 H), 7.16 (d, *J* = 1.5 Hz, 1 H), 7.12 (dd, *J* = 1.0, 8.0 Hz, 1 H), 6.84 (d, *J* = 8.0 Hz, 1 H), 6.02 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.3, 149.9, 148.4, 144.6, 138.4, 132.6, 129.4, 128.5, 128.4, 125.1, 120.1, 108.6, 106.6, 101.6.

MS (EI): *m*/*z* (%) = 252 (22), 251 (27), 250 (61), 249 (100), 248 (47), 174 (25), 165 (40), 164 (27), 145 (41), 122 (36).

(*E*)-3-(Naphthalen-2-yl)-1-phenylprop-2-en-1-one (4h)[32]

Yellow solid; yield: 242 mg (94%); mp 157–158 °C (Lit.[33] 157–158 °C).

IR: 3059.3, 2945.1, 1658.1, 1587.5, 1477.0, 1350.9, 1295.8, 1209.9, 1012.7, 991.7, 823.5, 780.9, 691.1, 657.5 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.07 (d, *J* = 7.5 Hz, 2 H), 8.03 (s, 1 H), 7.99 (d, *J* = 15.5 Hz, 1 H), 7.89–7.84 (m, 3 H), 7.80 (d, *J* = 8.5 Hz, 1 H), 7.67 (s, 1 H), 7.64–7.59 (m, 1 H), 7.55–7.52 (m, 4 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.5, 144.9, 138.3, 134.4, 133.3, 132.7, 132.4, 130.6, 128.7, 128.6, 128.5, 127.8, 127.3, 126.7, 123.6, 123.6, 122.2.

MS (EI): *m*/*z* (%) = 259 (19), 258 (93), 257 (100), 229 (19), 181 (15), 153 (15), 152 (22), 128 (17), 105 (31), 77 (37).

1-Phenyl-3-(pyridin-3-yl)prop-2-en-1-one (4i)[32]

Yellow solid; yield: 198 mg (95%); mp 101–102 °C (Lit.[34] 101–102 °C).

IR: 3062.2, 2915.6, 1650.0, 1601.4, 1309.3, 1221.5, 1014.2, 980.6, 755.8, 712.6, 680.5, 625.3, 573.4 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.83 (s, 1 H), 8.60 (d, *J* = 3.5 Hz, 1 H), 7.80 (d, *J* = 7.0 Hz, 2 H), 7.93–7.91 (m, 1 H), 7.75 (d, *J* = 16.0 Hz, 1 H), 7.60–7.56 (m, 2 H), 7.48 (t, *J* = 7.5 Hz, 2 H), 7.35–7.32 (m, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 189.7, 150.9, 149.8, 140.7, 137.6, 134.6, 133.0, 130.6, 128.6, 128.4, 123.8, 123.7.

MS (EI): *m*/*z* (%) = 209 (100), 208 (64), 181 (20), 180 (54), 132 (22), 105 (52), 104 (27), 78 (17), 77 (74), 51 (39).

(*E*)-3-(Furan-2-yl)-1-phenylprop-2-en-1-one (4j)[28]

Reddish liquid; yield: 188 mg (95%).

IR: 3052.7, 2933.4, 1652.5, 1600.0, 1551.1, 1283.0, 1222.8, 1012.2, 971.1, 748.6, 699.1, 638.4, 592.2 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.04 (d, *J* = 7.5 Hz, 2 H), 7.62–7.56 (m, 2 H), 7.53–7.46 (m, 4 H), 6.72 (d, *J* = 3.0 Hz, 1 H), 6.52 (s, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 189.5, 151.4, 144.7, 137.9, 132.5, 130.4, 128.3, 128.1, 119.1, 115.9, 112.4.

MS (EI): *m*/*z* (%) = 199 (8), 198 (55), 144 (8), 141 (11), 121 (18), 115 (10), 105 (100), 77 (43), 65 (29), 51 (18).

(2*E*,4*E*)-1,5-Diphenylpenta-2,4-dien-1-one (4k)[35]

Yellow solid; yield: 208 mg (89%); mp 102–103 °C (Lit.[35] 102–103 °C).

IR: 3060.1, 3034.5, 1667.9, 1598.1, 1448.5, 1221.5, 971.1, 748.4, 692.0 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.99 (d, *J* = 7.0 Hz, 2 H), 7.64–7.55 (m, 2 H), 7.51–7.48 (m, 4 H), 7.39–7.36 (m, 2 H), 7.34–7.31 (m, 1 H), 7.10 (d, *J* = 14.5 Hz, 1 H), 7.06–6.99 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.4, 144.7, 141.8, 138.2, 136.1, 132.6, 129.1, 128.8, 128.5, 128.3, 127.2, 126.9, 125.4.

MS (EI): *m*/*z* (%) = 235 (18), 234 (100), 233 (38), 157 (22), 129 (20), 128 (34), 105 (53), 91 (32), 77 (60), 51 (24).

3-Cyclopropyl-1-phenylprop-2-en-1-one (4l)[29]

Yellow liquid; yield: 148 mg (86%).

IR: 3064.1, 3008.1, 1663.5, 1607.5, 1447.9, 1379.2, 1271.5, 1229.9, 1180.5, 936.3, 889.7, 771.4, 696.1, 657.5 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.93 (d, *J* = 8.5 Hz, 2 H), 7.54 (t , *J* = 7.5 Hz, 1 H), 7.46 (t, *J* = 7.5 Hz, 2 H), 7.02 (d, *J* = 15.5 Hz, 1 H), 6.55 (dd, *J* = 10.5, 15.5 Hz, 1 H), 1.74–1.67 (m, 1 H), 1.04–1.00 (m, 2 H), 0.75–0.72 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 189.9, 155.2, 138.2, 132.4, 128.4, 128.3, 122.8, 15.3, 9.2.

MS (EI): *m*/*z* (%) = 157 (21), 144 (89), 129 (11), 115 (14), 105 (100), 95 (15), 77 (86), 67 (28), 65 (11), 51 (33).

5,9-Dimethyl-1-phenyldeca-2,8-dien-1-one (4m)[35]

Yellow liquid; yield: 221 mg (82%).

IR: 2965.5, 2925.5, 2873.4, 1670.7, 1620.4, 1449.0, 1378.5, 1278.8, 982.0, 758.2, 694.1 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.93 (d, *J* = 8.0 Hz, 2 H), 7.55 (t, *J* = 8.0 Hz, 1 H), 7.47 (t, *J* = 8.0 Hz, 2 H), 7.08–7.02 (m, 1 H), 6.88 (d, *J* = 15.5 Hz, 1 H), 5.09 (t, *J* = 6.0 Hz, 1 H), 2.36–2.31 (m, 1 H), 2.19–2.13 (m, 1 H), 2.07–1.97 (m, 2 H), 1.68 (s, 3 H), 1.60 (s, 3 H), 1.43–1.37 (m, 1 H), 1.26–1.20 (m, 1 H), 1.18–1.16 (m, 1 H), 0.95 (d, *J* = 7.0 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 190.8, 148.9, 138.0, 132.5, 131.5, 128.5, 128.4, 127.1, 124.4, 40.3, 36.8, 32.2, 25.7, 25.5, 19.6, 17.6.

MS (EI): *m*/*z* (%) = 254 (68), 253 (30), 137 (53), 136 (100), 135 (56), 133 (74), 132 (28), 145 (19), 121 (26), 105 (96).

3-(4-Nitrophenyl)acrylonitrile (5a)[13]

Colorless liquid; yield: 169 mg (97%).

IR: 3072.9, 2925.4, 2854.3, 2217.6, 1597.0, 1514.8, 1107.9, 971.9, 857.2 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.31 (d, *J* = 8.5 Hz, 2 H), 7.96 (d, *J* = 8.5 Hz, 2 H), 7.23 (d, *J* = 12.0 Hz, 1 H), 5.70 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 149.1, 147.7, 139.2, 128.1, 124.4, 116.9, 101.0.

MS (EI): *m*/*z* (%) = 174 (75), 173 (100), 172 (43), 144 (40), 143 (18), 128 (45), 127 (18), 116 (36), 101 (42), 77 (12).

3-(2-Methoxyphenyl)acrylonitrile (5b)[36]

Yellow liquid; yield: 153 mg (96%).

IR: 3055.1, 2942.5, 2840.8, 2213.5, 1500.5, 1487.1, 1463.6, 1251.8, 1110.8, 1024.9, 972.3, 751.4, 582.2 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.09 (d, *J* = 7.5 Hz, 1 H), 7.58 (d, *J* = 12.0 Hz, 1 H), 7.41 (t, *J* = 7.5 Hz, 1 H), 7.03 (t, *J* = 7.5 Hz, 1 H), 6.92 (d, *J* = 8.5 Hz, 1 H), 5.42 (d, *J* = 12.0 Hz, 1 H), 3.85 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 157.4, 143.6, 132.2, 128.2, 122.7, 120.6, 117.5, 110.8, 94.6, 55.5.

MS (EI): *m*/*z* (%) = 159 (100), 158 (61), 132 (34), 131 (87), 130 (61), 119 (14), 116 (21), 91 (13), 89 (20).

3-(3-Methoxyphenyl)acrylonitrile (5c)[36]

Yellow liquid; yield: 151 mg (95%).

IR: 3063.6, 3008.2, 2838.5, 2216.7, 1619.1, 1578.2, 1480.9, 1451.1, 1433.2, 1260.1, 1159.6, 1040.5, 955.7, 779.9, 684.3 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.34–7.30 (m, 2 H), 7.04 (d, *J* = 8.0 Hz, 1 H), 7.00–6.97 (m, 1 H), 6.94 (s, 1 H), 5.86 (d, *J* = 16.5 Hz, 1 H), 3.83 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 160.0, 150.4, 134.8, 130.1, 119.9, 118.0, 116.8, 112.5, 96.6, 55.3.

MS (EI): *m*/*z* (%) = 159 (42), 158 (100), 157 (53), 131 (20), 130 (33), 128 (21), 116 (15), 102 (14), 89 (17).

3-(2-Chlorophenyl)acrylonitrile (5d)[37]

Yellow liquid; yield: 153 mg (94%).

IR: 3064.3, 2890.4, 2219.1, 1615.2, 1470.8, 1439.5, 1051.7, 1039.3, 965.8, 771.6, 744.1, 700.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.09–8.07 (m, 1 H), 7.56 (d, *J* = 12.0 Hz, 1 H), 7.46–7.44 (m, 1 H), 7.39–7.35 (m, 2 H), 5.60 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 145.2, 134.4, 131.7, 129.9, 129.0, 127.2, 116.5, 98.1.

MS (EI): *m*/*z* (%) = 165 (18), 163 (59), 128 (100), 101 (34), 100 (15), 77 (14), 75 (19), 64 (13), 51 (6), 50 (19).

3-(3-Chlorophenyl)acrylonitrile (5e)[38]

Yellow liquid; yield: 148 mg (91%).

IR: 3055.3, 2218.1, 1619.6, 1563.5, 1476.9, 1428.2, 1079.9, 794.5, 697.4 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.74 (d, *J* = 7.0 Hz, 1 H), 7.70 (s, 1 H), 7.4–7.38 (m, 1 H), 7.34–7.32 (m, 1 H), 7.07 (d, *J* = 12.0 Hz, 1 H), 5.51 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 147.0, 135.2, 134.9, 130.8, 130.2, 129.0, 126.7, 116.7, 96.7.

MS (EI): *m*/*z* (%) = 165 (24), 163 (74), 128 (100), 101 (41), 100 (16), 77 (17), 75 (27), 64 (19), 51 (23), 50 (29).

3-(4-Tolyl)acrylonitrile (5f)[39]

Yellow liquid; yield: 140 mg (98%).

IR: 3035.7, 2924.3, 2214.9, 1608.3, 1511.7, 1181.9, 1041.4, 970.0, 825.3, 797.2, 704.1, 575.8 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.71 (d, *J* = 8.5 Hz, 2 H), 7.24 (d, *J* = 8.5 Hz, 2 H), 7.08 (d, *J* = 12.0 Hz, 1 H), 5.37 (d, *J* = 12.0 Hz, 1 H), 2.39 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 148.5, 141.5, 130.9, 129.5, 129.0, 117.5, 93.7, 21.5.

MS (EI): *m*/*z* (%) = 144 (8), 143 (74), 142 (100), 141 (43), 140 (6), 139 (5), 117 (6), 116 (29), 115 (41), 114 (20).

3-(1,3-Benzodioxol-5-yl)acrylonitrile (5g)[36]

White solid; yield: 164 mg (95%); mp 90–91 °C (Lit.[40] 94 °C).

IR: 3058.9, 2911.9, 2204.1, 1620.5, 1594.3, 1499.8, 1443.1, 1257.5, 1099.9, 1035.1, 931.7, 810.0, 626.5 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.48 (d, *J* = 1.5 Hz, 1 H), 7.19 (dd, *J* = 1.5, 8.0 Hz, 1 H), 6.91 (d, *J* = 12.0 Hz, 1 H), 6.84 (d, *J* = 8.0 Hz, 1 H), 6.02 (s, 2 H), 5.29 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 149.9, 148.2, 148.0, 128.0, 125.5, 117.6, 108.5, 107.9, 92.4.

MS (EI): *m*/*z* (%) = 174 (8), 173 (73), 172 (100), 171 (81), 170 (13), 116 (5), 115 (4), 114 (7), 89 (4), 88 (5).

3-(Naphthalen-2-yl)acrylonitrile (5h)[36]

Colorless liquid; yield: 170 mg (95%).

IR: 3050.1, 2213.9, 1615.9, 1363.2, 1159.9, 965.7, 858.0, 812.6, 750.7 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.14 (s, 1 H), 8.03 (d, *J* = 8.5 Hz, 1 H), 7.90–7.79 (m, 3 H), 7.57–7.48 (m, 2 H), 7.23 (d, *J* = 12.0 Hz, 1 H), 5.49 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 148.5, 134.2, 132.8, 131.0, 130.3, 128.7, 128.6, 127.7, 126.7, 124.6, 117.5, 94.9.

MS (EI): *m*/*z* (%) = 180 (14), 179 (100), 178 (30), 177 (8), 152 (12), 151 (15), 89 (13), 76 (22), 75 (12), 63 (14).

3-(Pyridin-3-yl)acrylonitrile (5i)

Yellow liquid; yield: 121 mg (93%).

IR: 3061.5, 2219.0, 1619.8, 1587.5, 1479.9, 1418.5, 1181.2, 1119.8, 722.3, 700.3 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.74 (s, 1 H), 8.62 (d, *J* = 9.0 Hz, 1 H), 8.35 (d, *J* = 9.0 Hz, 1 H), 7.40–7.37 (m, 1 H), 7.14 (d, *J* = 12.0 Hz, 1 H), 5.59 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 151.5, 150.8, 145.0, 134.6, 129.3, 123.6, 116.6, 97.8.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for C_8H_7N: 131.0609; found: 131.0606.

3-(Furan-2-yl)acrylonitrile (5j)[41]

Colorless liquid; yield: 105 mg (88%).

IR: 3062.3, 2935.1, 2214.7, 1672.3, 1625.2, 1474.1, 1390.0, 1019.7, 957.0, 748.4, 592.4 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.61 (s, 1 H), 7.07 (d, *J* = 3.5 Hz, 1 H), 6.98 (d, *J* = 12.0 Hz, 1 H), 6.57 (d, *J* = 3.5 Hz, 1 H), 5.26 (d, *J* = 12.0 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 149.3, 144.4, 134.2, 116.7, 114.9, 111.8, 90.6.

MS (EI): *m*/*z* (%) = 119 (100), 92 (26), 91 (42), 90 (60), 65 (12), 64 (60), 63 (40), 62 (10), 52 (14), 51 (9).

5-Phenylpenta-2,4-dienenitrile (5k)[41]

Yellow liquid; yield: 140 mg (90%).

IR: 3052.9, 3031.3, 2211.9, 1621.3, 1589.5, 1493.1, 1449.6, 988.5, 945.8, 801.7, 735.2, 689.0 cm^{-1}.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.45 (d, *J* = 8.5 Hz, 2 H), 7.33–7.28 (m, 3 H), 7.19–7.13 (m, 1 H), 6.93–6.85 (m, 2 H), 5.19 (d, *J* = 10.5 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 149.2, 141.6, 135.2, 129.6, 128.8, 127.5, 124.1, 116.6, 96.5.

MS (EI): *m*/*z* (%) = 155 (100), 154 (84), 140 (32), 128 (41), 127 (54), 115 (79), 102 (20), 77 (35), 63 (29), 51 (43).

Funding Information

Financial support from the National Natural Science Foundation of China (No. 21302143 and 51572198) and Natural Science Foundation of Zhejiang Province (No. LY13B020006 and LZ17E020002) is greatly appreciated.

Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0036-1590904.

References

(1) (a) Wittig, G.; Geissler, G. *Justus Liebigs Ann. Chem.* **1953**, *580*, 44. (b) Wittig, G.; Schollkopf, U. *Chem. Ber.* **1954**, *87*, 1318.
(2) (a) Takeda, T. *Modern Carbonyl Olefination*; Wiley-VCH: Weinheim, **2004**. (b) Maryanoff, B. E.; Reitz, A. B. *Chem. Rev.* **1989**, *89*, 863. (c) Shen, Y. *Acc. Chem. Res.* **1998**, *31*, 584.
(3) Ireland, R. E.; Norbeck, D. W. *J. Org. Chem.* **1985**, *50*, 2198.
(4) Barrett, A. G. M.; Hamprecht, D.; Ohkubo, M. *J. Org. Chem.* **1997**, *62*, 9376.
(5) (a) Shuto, S.; Niizuma, S.; Matsuda, A. *J. Org. Chem.* **1998**, *63*, 4489. (b) Gholinejad, M.; Firouzabadi, H.; Bahrami, M.; Nájera, C. *Tetrahedron Lett.* **2016**, *57*, 3773.
(6) (a) Pillips, D. J.; Graham, A. E. *Synlett* **2008**, 649. (b) Taylor, R. J. K.; Reid, M.; Foot, J.; Raw, S. A. *Acc. Chem. Res.* **2005**, *38*, 851. (c) Nakamura, M.; Mori, Y.; Okuyama, K.; Tanikawa, K.; Yasuda, S.; Hanada, K.; Kobayashi, S. *Org. Biomol. Chem.* **2003**, *1*, 3362. (d) Blackburn, L.; Wei, X.; Taylor, R. J. K. *Chem. Commun.* **1999**, 1337.
(7) Bressette, A. R.; Glover, L. C. IV. *Synlett* **2004**, 738.
(8) Crisóstomo, F. R. P.; Carrillo, R.; Martín, T.; García-Tellado, F.; Martín, V. S. *J. Org. Chem.* **2005**, *70*, 10099.
(9) Vatèle, J. M. *Tetrahedron Lett.* **2006**, *47*, 715.
(10) MacCoss, R. N.; Blaskus, E. P.; Ley, S. V. *Tetrahedron Lett.* **2003**, *44*, 7779.
(11) Kim, G.; Lee, D. G.; Chang, S. *Bull. Korean Chem. Soc.* **2001**, *22*, 943.
(12) Lee, E. Y.; Kim, Y.; Lee, J. S.; Park, J. *Eur. J. Org. Chem.* **2009**, 2943.
(13) Read, C. D. G.; Moore, P. W.; Williams, C. M. *Green Chem.* **2015**, *17*, 4537.
(14) Miyamura, H.; Suzuki, A.; Yasukawa, T.; Kobayashi, S. *Adv. Synth. Catal.* **2015**, *357*, 3815.
(15) Dutta, I. D.; Sarbajna, A.; Pandey, P.; Rahaman, S. M. W.; Singh, K.; Bera, J. K. *Organometallics* **2016**, *35*, 1505.
(16) Semmelhack, M. F.; Schmid, C. R.; Cortes, D. A.; Chou, C. S. *J. Am. Chem. Soc.* **1984**, *106*, 3374.
(17) For reviews on the copper-catalyzed oxidation of alcohols, see: (a) Ryland, B. L.; Stahl, S. S. *Angew. Chem. Int. Ed.* **2014**, *53*, 8824. (b) Cao, Q.; Dornan, L. M.; Rogan, L.; Hughes, N. L.; Muldoon, M. L. *Chem. Commun.* **2014**, *50*, 4524; and references therein.
(18) Cu/TEMPO catalyst systems: (a) Gamez, P.; Arends, I.; Reedijk, J.; Sheldon, R. A. *Chem. Commun.* **2003**, 2414. (b) Gamez, E.; Arends, I.; Sheldon, R. A.; Reedijk, J. *Adv. Synth. Catal.* **2004**, *346*, 805. (c) Hoover, J. M.; Stahl, S. S. *J. Am. Chem. Soc.* **2011**, *133*, 16901. (d) Hoover, J. M.; Ryland, B. L.; Stahl, S. S. *J. Am. Chem. Soc.* **2013**, *135*, 2357. (e) Ryland, B. L.; McCann, S. D.; Brunold, T. C.; Stahl, S. S. *J. Am. Chem. Soc.* **2014**, *136*, 12166; and references therein.
(19) Leung, P. S. W.; Teng, Y.; Toy, P. H. *Org. Lett.* **2010**, *12*, 4996.
(20) Zhou, Y.; Zhou, M. X.; Chen, M.; Su, J. H.; Du, J. F.; Song, Q. L. *RSC Adv.* **2015**, *5*, 103977.
(21) Wang, Y.; Du, G.; Gu, C.; Xing, F.; Dai, B.; He, L. *Tetrahedron* **2016**, *72*, 472.
(22) Jiang, X. Y.; Hartwig, J. F. *Angew. Chem. Int. Ed.* **2017**, *56*, 8887.
(23) Xia, X.; Toy, P. H. *Synlett* **2015**, *26*, 1737.
(24) Arai, S.; Koike, Y.; Hada, H.; Nishida, A. *J. Org. Chem.* **2010**, *75*, 7573.

(25) Webb, D.; Jamison, T. F. *Org. Lett.* **2012**, *14*, 2465.
(26) Yang, W.; Miao, T.; Li, P.; Wang, L. *RSC Adv.* **2015**, *5*, 95833.
(27) Kim, N.; David, W. L. *Aust. J. Chem.* **2017**, *70*, 436.
(28) Zhang, L.; Wang, A.; Wang, W.; Huang, Y.; Liu, X.; Miao, S.; Liu, J.; Zhang, T. *ACS Catal.* **2015**, *5*, 6563.
(29) Liang, D.; Li, X.; Lan, Q.; Huang, W.; Yuan, L.; Ma, Y. *Tetrahedron Lett.* **2016**, *57*, 2207.
(30) Zhu, Y. L.; Li, C.; Zhang, J. D.; She, M. Y.; Sun, W.; Wan, K.; Wang, Y. Q.; Yin, B.; Liu, P.; Li, J. L. *Org. Lett.* **2015**, *17*, 3872.
(31) Avik, K. B.; Alakananda, H. J. *RSC Adv.* **2014**, *4*, 23287.
(32) Jiang, Q.; Jia, J.; Xu, B.; Zhao, A.; Guo, C.-C. *J. Org. Chem.* **2015**, *80*, 3586.
(33) Xu, L. T.; Zhang, C.; He, Y. P.; Tan, L. S.; Ma, D. W. *Angew. Chem. Int. Ed.*. **2016**, *55*, 331.
(34) Yang, X. Y.; Jia, Y. X.; Tay, W. S.; Li, Y. X.; Pullarkata, S. A.; Leung, P. H. *Dalton Trans.*. **2016**, *45*, 13449.
(35) Chen, S.-J.; Lu, G.-P.; Cai, C. *RSC Adv.* **2015**, *5*, 13208.
(36) Han, Y.; Song, X.; Qiu, Y.; Hao, X.; Wang, J.; Wu, X.; Liu, X.; Liang, Y. *J. Org. Chem.* **2015**, *80*, 9200.
(37) Li, L.; Stimac, J. C.; Geary, L. M. *Tetrahedron Lett.* **2017**, *58*, 1379.
(38) Wang, Z.; Chang, S. *Org. Lett.* **2013**, *15*, 1990.
(39) Gu, Z.; Wang, Y.; Yao, Y.; Xia, X.; Wang, H.; Li, W. *Catal. Lett.* **2015**, *145*, 2046.
(40) Stephen, A. D.; Bruce, A. L.; Anthony, H.; Raymond, A. W.; George, W. G. *J. Org. Chem.* **1979**, *44*, 4640.
(41) Lanari, D.; Alonzi, M.; Ferlin, F.; Santoro, S.; Vaccaro, L. *Org. Lett.* **2016**, *18*, 2680.