The Surfactant-Promoted Cross-Coupling Reactions of Arylboronic Acids with Carboxylic Anhydrides or Acyl Chlorides in Water

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Abstract: The palladium(II) chloride catalyzed cross-coupling of arylboronic acids with carboxylic anhydrides or acyl chlorides in water in the presence of various surfactants is described. The inexpensive and industrially widely used sodium dodecyl sulfate (SDS) was found to be a good promoter of the coupling reaction and aryl ketones were obtained in good yields without the use of phosphine ligands. The reactions were unaffected by the presence of electron-releasing and electron-withdrawing substituents in both the arylboronic acids and carboxylic derivatives and a variety of aryl ketones were obtained under mild conditions in air.

Key words: aryl ketone, boronic acid, carboxylic anhydride, acyl chloride, palladium catalysis

Aryl ketones are important structural motifs that are present in a vast number of natural and unnatural products of biological and medicinal interest.[1] Friedel–Crafts acylation is a traditional method for the preparation of aryl ketones,[2] however, it has major drawbacks, such as drastic reaction conditions, low regioselectivity, and the formation of large amounts of byproducts.[3] Aryl ketones can also be prepared by the nucleophilic addition of organometallic reagents to carboxylic acid derivatives,[4–6] but low yields are often obtained because of the formation of tertiary alcohols.[7] Recently, palladium-catalyzed cross-coupling processes have been developed for the preparation of aryl ketones using carboxylic derivatives and boronic acids.[8–11] These methods are superior to the previous methods in terms of mildness of reaction conditions, efficiency, selectivity, and functional group compatibility,[12] and the boronic acids are nontoxic and they are thermally, air-, and moisture-stable.[13] Generally, acyl chlorides can be used for cross-coupling reactions in the presence or absence of phosphine ligands,[14] but the less active carboxylic derivatives, usually carboxylic anhydrides or carboxylic acids, require activation by phosphine ligands.[15]

Recently, aqueous transition metal catalysis has gained popularity as a method for generating complex organic molecules with reduced environmental impact.[16] The benefits involved with aqueous catalytic systems include easier product separation, decreased cost, and increased reactivity.[17] Although the utility of a variety of transition-metal catalysts in water has been studied,[18] little attention has been paid to the palladium-catalyzed synthesis of aryl ketones in an aqueous medium. Our previous research revealed that the cross-coupling reactions of arylboronic acids with carboxylic anhydrides or acyl chlorides could be promoted by using the aqueous phase as the reaction medium.[19] Herein, we wish to report the surfactant-improved palladium-catalyzed cross-coupling reaction of arylboronic acids with carboxylic anhydrides or acyl chlorides in water in the presence of surfactants. Surfactants have considerably extend organic chemistry in water and have a notable effect on the reaction rates.[20–22] Our results show that the surfactants have a significant effect on the activity of the cross-coupling reaction in water in the absence of phosphine ligands.

We initially studied the effect of various surfactants on the coupling reaction as shown in Table 1. The coupling reaction of benzoic anhydride and phenylboronic acid to give benzophenone (**1a**) was chosen as the model reaction using potassium carbonate as the base in the presence of palladium(II) chloride (1.7 mol%) as the catalyst at 60 °C for six hours. The reaction was carried out on a 0.1 M aqueous solution of surfactant. The separation of the products was easily performed by the extraction with diethyl ether. The results showed that the coupling reaction was less active in pure water and surfactants could improve the reaction markedly. The addition tetramethylammonium bromide (TBAB) clearly increased the yield of desired product, but a good yield was obtained when the surfactant was changed to cetyltrimethylammonium bromide (CTAB), which is in possession of a long alkyl chain (Table 1, entries 2, 3). This result should be attributed to the properties of amphiphiles.[23,24] The long alkyl chain in CTAB is advantageous for the formation of micelles, which might cause the acceleration of the coupling reaction in the aqueous medium. TBAB with short alkyl chain does not form micelles, but it can influence the hydrogen-bonding structure of water and it is termed a hydrotrope; hydrotropes can also form associates, but these have a much smaller size than micelles.[20a] Among the anionic surfactants tested, sodium stearate (SS) and sodium dodecylbenzenesulfonate (SDBS) delivered a moderate yield (Table 1, entries 4, 5), while sodium dodecyl sulfate (SDS) and sodium dodecane-1-sulfonate (DSASS) exhibited much better efficiency on the coupling reaction (Table 1, entries 6, 7). Since it is inexpensive and widely

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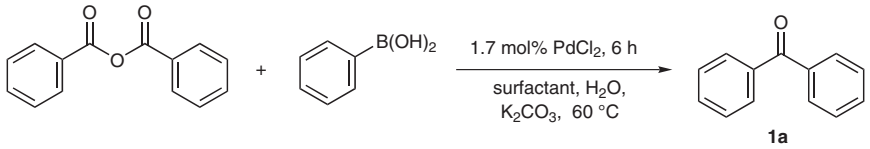
Table 1 The Effect of Surfactant on the Coupling Reaction in Water[a]



1a

Entry	Surfactant (CMC)	Yield[b] (%)
1	–	31
2	TBAB	57
3	CTAB (9.2×10^{-4} mol/L)	82
4	SS (4.2×10^{-4} mol/L)	67
5	SDBS (1.6×10^{-3} mol/L)	77
6	SDS (8.1×10^{-3} mol/L)	95
7	DSASS (9.8×10^{-3} mol/L)	94

[a] Reaction conditions: Bz_2O (1.0 mmol), $PhB(OH)_2$ (1.5 mmol), K_2CO_3 (1.6 mmol), surfactant (0.5 mmol), $PdCl_2$ (1.7 mol%), H_2O (5 mL), 6 h, 60 °C.
[b] GC yield; determined using pentane-2,4-dione as internal standard.

used in industry,[25] SDS was chosen as the additive in all of our further experiments.

The rate of the coupling reaction was found to be dependent on the concentration of SDS.[26] When the concentration of SDS was lower than its critical micelle concentration (CMC) (8.1×10^{-3} mol/L at 25 °C),[20a] the effect of SDS on the coupling reaction was poor (Table 2, entries 1, 2). Under the same conditions, other surfactants have little influence on the reactivity of the coupling reaction. However, the reaction rate increased rapidly when the concentration was higher than the CMC (Table 2, entries 3–5) and an optimum yield was obtained at 0.1 M SDS solution (Table 2, entry 6). In addition, the formation of the byproduct biphenyl, which is generated by self-coupling of phenylboronic acid, at this concentration was also obviously depressed. Further increases in the concentration of SDS led to a decrease in the yields (Table 2, entries 7–9), and heavy hydrolysis of benzoic anhydride was observed.

The effect of various bases was screened and the results are presented in Table 3. Sodium carbonate, potassium carbonate, sodium hydroxide, and potassium hydroxide showed good efficiency and the best yield was obtained using potassium carbonate (Table 3, entries 1, 2, 7, 8). Potassium phosphate afforded a good yield (Table 3, entry 6), but sodium acetate, potassium acetate, potassium fluoride, and triethylamine gave the desired product with low yields (Table 3, entries 3, 4, 9, 10). Palladium(II) chloride was shown to be an excellent catalyst in this water–SDS system (Table 3, entry 2), while palladium(II) acetate and bis(acetonitrile)dichloropalladium(II) gave moderate yields (Table 3, entries 11, 12). Increasing the temperature from 40 °C to 80 °C had a positive effect on the acylation reaction (Table 3, entries 13, 14), but the rate of self-coupling of phenylboronic acid was also enhanced. It should be noted that the reaction rate in this aqueous condition was faster than that in organic solvent with the activation of phosphine ligands.[27]

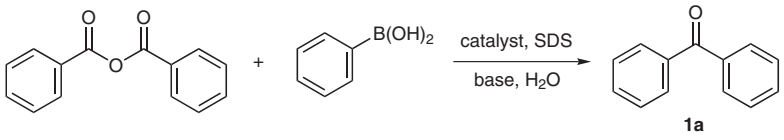
Table 2 The Effect of the Concentration of SDS on the Coupling Reaction[a]

Entry	SDS (M)	Ratio of Bz_2O/SDS	Yield[b] (%)
1	0	–	31
2	0.004	50	33
3	0.008	25	57
4	0.016	12.5	62
5	0.04	5	78
6	0.10	2	95
7	0.15	1.3	90
8	0.20	1	84
9	0.4	0.5	77

[a] Reaction conditions: Bz_2O (1.0 mmol), $PhB(OH)_2$ (1.5 mmol), K_2CO_3 (1.6 mmol), $PdCl_2$ (1.7 mol%), H_2O (5 mL), SDS, 6 h, 60 °C.
[b] GC yield; determined using pentane-2,4-dione as internal standard.

To further understand the scope and limitations of the cross-coupling reaction in palladium(II) chloride/water/SDS, a variety of arylboronic acids were applied to the coupling reaction and the results are presented in Table 4. The electron-rich arylboronic acids showed the excellent reactivity and furnished the products **1** in high yields (Table 4, entries 1–3). A moderate yield was obtained for the electron-deficient arylboronic acid (Table 4, entry 5). 2-Naphthylboronic acid afforded the desired product **1f** in good yield (Table 4, entry 6), but the yield decreased for

Table 3 The Effect of Base and Catalysts on the Cross-Coupling Reaction[a]



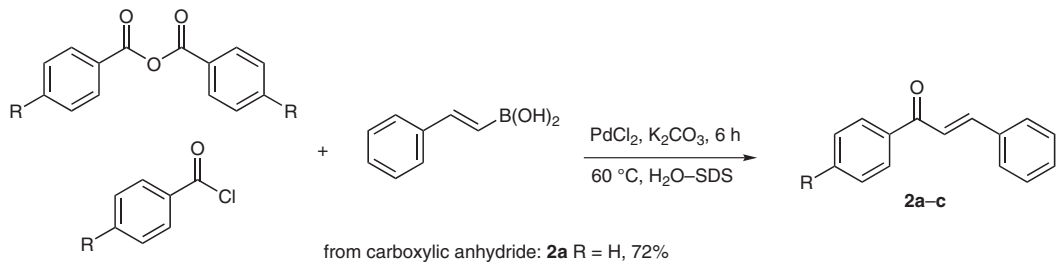
1a

Entry	Base	Catalyst (1.7 mol%)	Yield[b] (%)
1	Na_2CO_3	$PdCl_2$	88
2	K_2CO_3	$PdCl_2$	95
3	NaOAc	$PdCl_2$	53
4	KOAc	$PdCl_2$	50
5	Na_3PO_4	$PdCl_2$	70
6	K_3PO_4	$PdCl_2$	80
7	NaOH	$PdCl_2$	87
8	KOH	$PdCl_2$	89
9	KF	$PdCl_2$	54
10	Et_3N	$PdCl_2$	66
11	K_2CO_3	$Pd(OAc)_2$	63
12	K_2CO_3	$PdCl_2(MeCN)_2$	71
13	K_2CO_3	$PdCl_2$[c]	50
14	K_2CO_3	$PdCl_2$[c]	95
15	K_2CO_3	$PdCl_2$[d]	80
16	K_2CO_3	$PdCl_2$[e]	95

[a] Reaction conditions: Bz_2O (1.0 mmol), $PhB(OH)_2$ (1.5 mmol), base (1.6 mmol), SDS (0.5 mmol), H_2O (5 mL), 60 °C, 6 h.
[b] Conversion was determined by GC, using pentane-2,4-dione as internal standard.
[c] Reaction temperature of entries 13 and 14 was 40 °C and 80 °C, respectively.
[d] 1 mol% $PdCl_2$ was used.
[e] 3 mol% $PdCl_2$ was used.

the more sterically hindered 1-naphthylboronic acid (Table 4, entry 7). 2-Methylboronic acid gave the analogous result (Table 4, entry 4). The catalytic systems were compatible with heteroarylboronic acids as exemplified in the reaction of 2-thienylboronic acid and 3-thienylboronic acid with benzoic anhydride to give the ketones in good yields (Table 4, entries 8, 9). Vinylboronic acid showed excellent reactivity and furnished the products **2** in high yields (Scheme 1).

Similar with the palladium(II) acetate/water/PEG and palladium(II) acetate/water/[bmim][PF_6] system, low yields were obtained for the reactions involving the aromatic carboxylic anhydrides bearing strong electron-withdrawing groups in benzoic anhydride, which were sensitive to

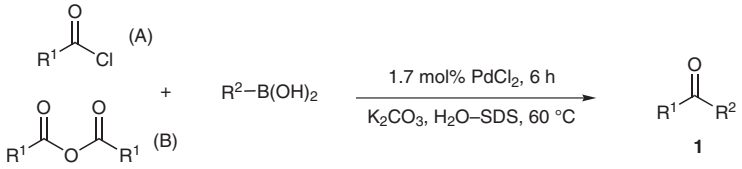


2a–c

from carboxylic anhydride: **2a** R = H, 72%
2b R = Me, 80%
from acyl chloride: **2a** R = H, 69%
2b R = Me, 81%
2c R = CN, 74%

Scheme 1

Table 4 Cross-Coupling Reactions of Arylboronic Acids with Acyl Chlorides (A) or Carboxylic Anhydrides (B)



Entry	R^1COCl or $(R^1CO)_2O$	$R^2B(OH)_2$	Product	Yield[a] (%) from A	Yield[a] (%) from B
1	Ph	Ph	**1a**	76	89
2	Ph	4-MeC_6H_4	**1b**	80	88
3	Ph	4-$MeOC_6H_4$	**1c**	82	93
4	Ph	2-MeC_6H_4	**1d**	56	63
5	Ph	4-$F_3CC_6H_4$	**1e**	65	76
6	Ph	2-naphthyl	**1f**	78	89
7	Ph	1-naphthyl	**1g**	75	74
8	Ph	2-thienyl	**1h**	63	80
9	Ph	3-thienyl	**1i**	78	93
10	4-$O_2NC_6H_4$	Ph	**1j**	71	71
11	4-NCC_6H_4	Ph	**1k**	55	55
12	4-ClC_6H_4	Ph	**1l**	77	81
13	4-ClC_6H_4	4-$MeOC_6H_4$	**1m**	73	87
14	4-ClC_6H_4	4-$F_3CC_6H_4$	**1n**	74	78
15	4-ClC_6H_4	2-naphthyl	**1o**	81	83
16	3-ClC_6H_4	Ph	**1p**	82	85
17	3-ClC_6H_4	4-$MeOC_6H_4$	**1q**	74	81
18	3-ClC_6H_4	4-$F_3CC_6H_4$	**1r**	76	80
19	2-ClC_6H_4	Ph	**1s**	40	68
20	2-ClC_6H_4	4-$MeOC_6H_4$	**1t**	52	65
21	4-MeC_6H_4	Ph	**1b**	64	87
22	4-MeC_6H_4	4-$MeOC_6H_4$	**1u**	73	93
23	4-MeC_6H_4	4-$F_3CC_6H_4$	**1v**	74	79
24	4-MeC_6H_4	2-naphthyl	**1w**	80	85
25	4-$MeOC_6H_4$	Ph	**1c**	83	85
26	2-$MeOC_6H_4$	Ph	**1x**	53	51
27	2-furyl	Ph	**1y**	88	94
28	2-furyl	4-$MeOC_6H_4$	**1z**	76	95
29	2-furyl	4-$F_3CC_6H_4$	**1aa**	43	82
30	2-furyl	2-naphthyl	**1ab**	79	90
31	2-furyl	2-thienyl	**1ac**	72	79
32	*n*-C_5H_{11}	Ph	**1ad**	trace	<5
33	*n*-C_5H_{11}	4-$MeOC_6H_4$	**1ae**	23	30
34	*n*-C_5H_{11}	Ph	**1af**	11	16

[a] Isolated yields; all the compounds were identified by MS and NMR.

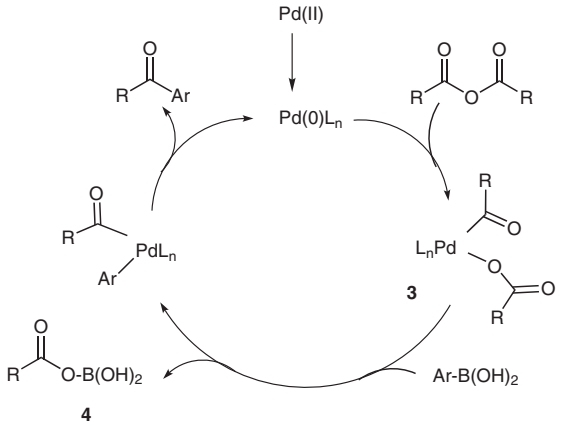
the aqueous phase and easier to hydrolyze (Table 4, entries 10, 11). Except for the sterically demanding 2-chlorobenzoic anhydride (Table 4, entries 19, 20), good yields were obtained for various chlorobenzoic anhydrides (Table 4, entries 12–18). The aromatic carboxylic anhydride with electron-donating substituents in benzoic anhydride showed better reactivity and delivered the corresponding ketones in good to excellent yields (Table 4, entries 21–25). The yield of sterically demanding 2-methoxybenzoic anhydride was slightly decreased (Table 4, entry 26). The method was applicable to furan-2-carboxylic anhydride and excellent yields were obtained (Table 4, entries 27–31). The reactions of aliphatic carboxylic anhydrides were sluggish under the reaction conditions (Table 4, entries 32–34).

Although the acyl chlorides were sensitive to moisture, moderate to good yields were obtained for the coupling reaction of acyl chloride with arylboronic acid in this system (Table 4). Under the same reaction conditions, a variety of ketones were prepared in moderate to good yields and the competitive hydrolysis for most of the acyl chlorides appears to be minimized. The electronic properties of the substituents in both arylboronic acid and aryl acyl chloride have little effect on the reactivity (Table 4, entries 1–18, 21–25, 27–31), but steric effects indeed influenced the reaction (Table 4, entries 19, 20, 26). The hydrolysis of the aliphatic acyl chlorides was heavy and led to the very low yields of desired cross-coupling product (Table 4, entries 32–34).

The reusability of the palladium(II) chloride/water/SDS was studied on the model coupling reaction of benzoic anhydride with phenylboronic acid. The product was easily isolated by extraction with diethyl ether. The residue after the extraction of the product with diethyl ether was recycled and a 78% yield was obtained. But the reactivity rapidly decreased and only a trace of the cross-coupling product was isolated on the third run.

The acylation in the absence of phosphine ligands in water is thought to proceed through the mechanism previously described by Yamamoto[28] and Gooßen[29] (Scheme 2). The oxidative addition of carboxylic anhydride onto palladium gives the palladium complex of **3**, which then forms the (acyl)(aryl)palladium(II) intermediate and **4**. Reductive elimination leads to the coupling product and active palladium(0) species to complete the catalytic cycle. The research of Yamamoto and Gooßen showed that the successful oxidative addition of anhydride onto palladium in organic solvent required the activation of phosphine ligands. In the aqueous media, the surfactant promotes the solubility of the substrates in water through the formation of micelles, which processes the low dielectric constant and facilitates the coupling reaction. On the other hand, the reactants are concentrated relative to the surrounding water phase, thus leading to an increased rate of coupling reaction.[30]

In conclusion, we have developed a surfactant-promoted method for the preparation of aryl ketones in aqueous media. The results demonstrated that the cross-coupling reactions of arylboronic acids with carboxylic anhydride or acyl chlorides in water were significantly accelerated by the surfactants and the reactions could be carried out smoothly under mild conditions in the absence of expensive and environmentally unfavorable phosphine ligands. The environmentally friendly nature of water and easy isolation of the product made the present method attractive.



Scheme 2

Starting materials and solvents were purchased from common commercial sources and were used without additional purification. Column chromatography was carried out on silica gel (300–400 mesh). ^{1}H NMR spectra were recorded at 500 MHz or 400 MHz, ^{13}C NMR spectra were recorded at 100 MHz, using TMS as internal standard. Mass spectroscopy data of the product of acylation reaction was collected on a MS-EI instrument.

Acylation Reaction in Water and Surfactant; General Procedure

K_2CO_3 (0.221 g, 1.6 mmol) and $PdCl_2$ (3 mg, 1.7 mol%) were added to a mixture of SDS (0.144 g, 0.5 mmol) and H_2O (5 mL) and heated to 60 °C with stirring. Then arylboronic acid (1.2 mmol) and carboxylic anhydride (1.0 mmol) (or 1.0 mmol acyl chloride) were added to the soln and the mixture held at 60 °C for the indicated time. When the reaction was complete, the soln was cooled to r.t. and the resulting suspension was extracted with Et_2O (4 × 5 mL). The combined ether phases were concentrated and further purification of the product was achieved by flash column chromatography on silica gel.

Benzophenone (1a)[9a]

White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.81 (d, *J* = 4.1 Hz, 4 H, H_{phenyl}), 7.61–7.59 (m, 2 H, H_{phenyl}), 7.51–7.48 (t, *J* = 7.7 Hz, 4 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 182 (75) [M$^+$], 105 (100), 77 (56), 51 (15).

4-Methylbenzophenone (1b)[9a]

Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.68–7.66 (d, *J* = 6.2 Hz, 2 H, H_{phenyl}), 7.62–7.60 (d, *J* = 8.1 Hz, 2 H, 4-MeC_6H_4), 7.47–7.44 (t, *J* = 7.4 Hz, 1 H, H_{phenyl}), 7.37–7.34 (t, *J* = 7.6 Hz, 2 H, H_{phenyl}), 7.17–7.15 (d, *J* = 8.0 Hz, 2 H, 4-MeC_6H_4), 2.32 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 196 (60) [M$^+$], 181 (15), 165 (5), 152 (5), 119 (100), 105 (60), 91 (40), 77 (40), 65 (20), 51 (15).

4-Methoxybenzophenone (1c)[29]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 2 H, H_{phenyl}), 7.76–7.74 (d, *J* = 4.2 Hz, 2 H, 4-$MeOC_6H_4$), 7.56–7.54 (t, *J* = 7.5 Hz, 1 H, H_{phenyl}), 7.48–7.45 (t, *J* = 7.6 Hz, 2 H, H_{phenyl}), 6.97–6.95 (d, *J* = 8.8 Hz, 2 H, 4-$MeOC_6H_4$), 3.87 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 212 (70) [M$^+$], 181 (3), 135 (100), 105 (10), 92 (12), 77 (28), 51 (6).

2-Methylbenzophenone (1d)[29]
Colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83–7.80 (q, *J* = 3.1 Hz, 2 H, H_{phenyl}), 7.58–7.54 (t, *J* = 7.2 Hz, 1 H, H_{phenyl}), 7.45–7.43 (m, 3 H, H_{phenyl}), 7.37–7.35 (q, *J* = 2.4 Hz, 1 H, H_{phenyl}), 7.04–6.98 (m, 2 H, H_{phenyl}), 2.42 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 196 (80) [M$^+$], 181 (20), 165 (5), 152 (5), 119 (100), 105 (60), 91 (40), 77 (30), 65 (20), 51 (20).

4-(Trifluoromethyl)benzophenone (1e)[27]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.90–7.89 (d, *J* = 7.3 Hz, 2 H, 4-$F_3CC_6H_4$), 7.81–7.80 (d, *J* = 8.1 Hz, 2 H, 4-$F_3CC_6H_4$), 7.76–7.75 (d, *J* = 8.1 Hz, 2 H, H_{phenyl}), 7.65–7.61 (m, 1 H, H_{phenyl}), 7.52–7.49 (d, *J* = 7.2 Hz, 2 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 250 (60) [M$^+$], 181 (7), 173 (100), 105 (30), 77 (40).

2-Benzoylnaphthalene (1f)[29]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.27 (s, 1 H, $H_{naphthyl}$), 7.96 (s, 2 H, $H_{naphthyl}$), 7.94–7.92 (d, *J* = 7.6 Hz, 2 H, H_{phenyl}), 7.88–7.86 (d, *J* = 6.8 Hz, 2 H, $H_{naphthyl}$), 7.64–7.61 (m, 2 H, $H_{naphthyl}$), 7.59–7.51 (m, 3 H, H_{phenyl}).

MS (EI) = *m*/*z* (%): 233 (10) [M + 1]$^+$, 232 (M$^+$, 60), 155 (100), 127 (80), 105 (40), 77 (75), 51 (20).

1-Benzoylnaphthalene (1g)[2b]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.11–8.09 (d, *J* = 8.0 Hz, 1 H, $H_{naphthyl}$), 8.02–8.00 (d, *J* = 8.0 Hz, 1 H, $H_{naphthyl}$), 7.94–7.92 (d, *J* = 4.8 Hz, 1 H, $H_{naphthyl}$), 7.89–7.86 (d, *J* = 3.3 Hz, 2 H, H_{phenyl}), 7.63–7.56 (m, 2 H, $H_{naphthyl}$), 7.56–7.51 (m, 3 H, H_{phenyl}), 7.51–7.45 (m, 2 H, $H_{naphthyl}$).

MS (EI): *m*/*z* (%) = 233 (12) [M + 1]$^+$, 232 (M$^+$, 60), 155 (100), 127 (60), 105 (25), 77 (40), 51 (20).

2-Benzoylthiophene (1h)[1b]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87–7.85 (d, *J* = 6.8 Hz, 2 H, H_{phenyl}), 7.72–7.71 (d, *J* = 3.6 Hz, 1 H, $H_{thienyl}$), 7.65–7.63 (d, *J* = 4.0 Hz, 1 H, $H_{thienyl}$), 7.61–7.57 (t, *J* = 7.2 Hz, 1 H, H_{phenyl}), 7.51–7.47 (t, *J* = 7.2 Hz, 2 H, H_{phenyl}), 7.17–7.15 (t, *J* = 4.4 Hz, 1 H, $H_{thienyl}$).

MS (EI): *m*/*z* (%) = 188 (90) [M$^+$], 171 (10), 160 (10), 111 (100), 105 (40), 77 (20), 51 (12).

3-Benzoylthiophene (1i)[31]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87–7.85 (d, *J* = 4.2 Hz, 2 H, H_{phenyl}), 7.73–7.71 (m, 1 H, $H_{thienyl}$), 7.65–7.63 (m, 1 H, H_{phenyl}), 7.61–7.57 (m, 1 H, $H_{thienyl}$), 7.51–7.47 (m, 2 H, H_{phenyl}), 7.17–7.14 (m, 1 H, $H_{thienyl}$).

MS (EI): *m*/*z* (%) = 188 (95) [M$^+$], 171 (10), 160 (10), 111 (100), 105 (40), 77 (20), 51 (12), 39 (12).

4-Nitrobenzophenone (1j)[27]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36–8.34 (d, *J* = 2.9 Hz, 2 H, 4-$O_2NC_6H_4$), 7.95–7.93 (d, *J* = 2.9 Hz, 2 H, 4-$O_2NC_6H_4$), 7.81–7.79 (d, *J* = 4.0 Hz, 2 H, H_{phenyl}), 7.66–7.64 (t, *J* = 8.0 Hz, 1 H, H_{phenyl}), 7.55–7.51 (d, *J* = 8.0 Hz, 2 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 227 (75) [M$^+$], 181 (5), 150 (20), 105 (100), 77 (60), 76 (20), 51 (20).

4-Benzoylbenzonitrile (1k)[27]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89–7.87 (d, *J* = 4.2 Hz, 2 H, 4-NCC_6H_4), 7.81–7.80 (m, 2 H, H_{phenyl}), 7.79–7.78 (m, 2 H, 4-NCC_6H_4), 7.67–7.63 (m, 1 H, H_{phenyl}), 7.54–7.50 (d, *J* = 7.6 Hz, 2 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 207 (40) [M$^+$], 130 (20), 105 (100), 77 (35), 51 (18).

4-Chlorobenzophenone (1l)[27]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.78–7.75 (m, 4 H, H_{phenyl}), 7.62–7.59 (m, 1 H, H_{phenyl}), 7.51–7.46 (m, 4 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 218 (10) [M$^+$, ^{37}Cl], 216 (30) [M$^+$, ^{35}Cl], 181 (10), 139 (50), 111 (50), 105 (80), 77 (100), 51 (70).

(4-Chlorophenyl)(4-methoxyphenyl)methanone (1m)[32]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.81–7.79 (d, *J* = 9.0 Hz, 2 H, 4-ClC_6H_4), 7.72–7.70 (d, *J* = 8.5 Hz, 2 H, 4-$MeOC_6H_4$), 7.46–7.44 (d, *J* = 8.4 Hz, 2 H, 4-ClC_6H_4), 6.97–6.96 (d, *J* = 8.8 Hz, 2 H, 4-$MeOC_6H_4$), 3.89 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 248 (10) [M$^+$, ^{37}Cl], 246 (30) [M$^+$, ^{35}Cl], 218 (^{37}Cl, 20), 216 (^{35}Cl, 60), 212 (70) 181 (10), 139 (80), 135 (100), 111 (50), 105 (80), 77 (70), 51 (40).

(4-Chlorophenyl)[4-(trifluoromethyl)phenyl]methanone (1n)[33]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.80–7.79 (d, *J* = 8.2 Hz, 2 H, 4-$F_3CC_6H_4$), 7.70–7.68 (m, 4 H), 7.43–7.41 (d, *J* = 8.5 Hz, 2 H, 4-ClC_6H_4).

MS (EI): *m*/*z* (%) = 286 (10) [M$^+$, ^{37}Cl], 284 (30) [M$^+$, ^{35}Cl], 250 (60), 218 (^{37}Cl, 30), 216 (^{35}Cl, 90), 181 (10), 173 (100), 139 (52), 105 (80), 77 (40), 51 (20).

(4-Chlorophenyl)(2-naphthyl)methanone (1o)[34]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.23 (s, 1 H, $H_{naphthyl}$), 7.97–7.83 (m, 4 H), 7.82–7.80 (m, 2 H, 4-ClC_6H_4), 7.65–7.63 (m, 1 H, $H_{naphthyl}$), 7.62–7.57 (m, 1 H, $H_{naphthyl}$), 7.56–7.48 (m, 2 H, $H_{naphthyl}$).

MS (EI): *m*/*z* (%) = 268 (30) [M$^+$, ^{37}Cl], 266 (90) [M$^+$, ^{35}Cl], 231 (30), 155 (100), 139 (30), 127 (50), 111 (20), 51 (5).

3-Chlorobenzophenone (1p)[10]
Yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.81–7.79 (m, 3 H), 7.68–7.63 (t, *J* = 9.3 Hz, 1 H, 3-ClC_6H_4), 7.62–7.56 (m, 2 H), 7.52–7.49 (t, *J* = 7.7 Hz, 2 H, H_{phenyl}), 7.45–7.41 (t, *J* = 7.8 Hz, 1 H, 3-ClC_6H_4).

MS (EI): *m*/*z* (%) = 218 (15) [M$^+$, ^{37}Cl], 216 (45) [M$^+$, ^{35}Cl], 181 (20), 139 (50), 111 (100), 105 (30), 77 (80), 51 (50).

(3-Chlorophenyl)(4-methoxyphenyl)methanone (1q)[35]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.81 (d, *J* = 2.0 Hz, 2 H, 4-$MeOC_6H_4$), 7.80 (s, 1 H, 3-ClC_6H_4), 7.72–7.71 (d, *J* = 2.5 Hz, 1 H, 3-ClC_6H_4), 7.61 (s, 1 H, 3-ClC_6H_4), 7.41 (s, 1 H, 3-ClC_6H_4), 6.99–6.97 (d, *J* = 3.8 Hz, 2 H, 4-$MeOC_6H_4$), 3.89 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 248 (10) [M$^+$, ^{37}Cl], 246 (30) [M$^+$, ^{35}Cl], 218 (^{37}Cl, 15), 216 (^{35}Cl, 45), 212 (60) 181 (20), 139 (80), 135 (100), 111 (60), 105 (80), 77 (50), 51 (10).

(3-Chlorophenyl)[4-(trifluoromethyl)phenyl]methanone (1r)[36]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89–7.88 (d, *J* = 8.1 Hz, 2 H, 4-$F_3CC_6H_4$), 7.78–7.76 (m, 3 H), 7.68–7.66 (t, *J* = 5.5 Hz, 1 H, 3-ClC_6H_4), 7.62–7.59 (t, *J* = 6.5 Hz, 1 H, 3-ClC_6H_4), 7.47–7.44 (t, *J* = 7.9 Hz, 1 H, 3-ClC_6H_4).

MS (EI): *m*/*z* (%) = 286 (15) [M$^+$, ^{37}Cl], 284 (45) [M$^+$, ^{35}Cl], 250 (60), 218 (^{37}Cl, 20), 216 (^{35}Cl, 60), 181 (20), 173 (100), 139 (52), 105 (80), 77 (50), 51 (5).

2-Chlorobenzophenone (1s)[37]
Yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.75–7.73 (t, *J* = 4.2 Hz, 2 H, H_{phenyl}), 7.54–7.51 (t, *J* = 7.4 Hz, 1 H, 2-ClC_6H_4), 7.41–7.37 (m, 4 H), 7.30–7.29 (t, *J* = 4.3 Hz, 2 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 218 (15) [M$^+$, ^{37}Cl], 216 (50) [M$^+$, ^{35}Cl], 181 (20), 139 (50), 111 (50), 105 (80), 77 (100), 51 (70).

(2-Chlorophenyl)(4-methoxyphenyl)methanone (1t)[38]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.72–7.71 (d, *J* = 9.0 Hz, 2 H, 2-ClC_6H_4), 7.37–7.34 (d, *J* = 8.1 Hz, 2 H, 4-$MeOC_6H_4$), 7.28–7.27 (m, 2 H, 2-ClC_6H_4), 6.87–6.85 (d, *J* = 3.5 Hz, 2 H, 4-$MeOC_6H_4$), 3.80 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 248 (10) [M$^+$, ^{37}Cl], 246 (35) [M$^+$, ^{35}Cl], 218 (^{37}Cl, 15), 216 (^{35}Cl, 50), 212 (60) 181 (20), 139 (100), 135 (80), 111 (60), 105 (80), 77 (70), 51 (10).

(4-Methoxyphenyl)(4-tolyl)methanone (1u)[39]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (d, *J* = 8.8 Hz, 2 H, 4-MeC_6H_4), 7.69–7.68 (d, *J* = 8.1 Hz, 2 H, 4-$MeOC_6H_4$), 7.29–7.27 (d, *J* = 5.0 Hz, 2 H, 4-MeC_6H_4), 6.98–6.96 (d, *J* = 8.8 Hz, 2 H, 4-$MeOC_6H_4$), 3.90 (s, 3 H, OCH_3), 2.45 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 226 (40) [M$^+$], 212 (70), 196 (40), 181 (3), 165 (5), 152 (5), 135 (80), 119 (100), 105 (10), 91 (50), 77 (28), 51 (6).

[4-(Trifluoromethyl)phenyl)](4-tolyl)methanone (1v)[40]
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89–7.87 (d, *J* = 8.0 Hz, 2 H, 4-$F_3CC_6H_4$), 7.76–7.74 (d, *J* = 8.0 Hz, 2 H, 4-$F_3CC_6H_4$), 7.73–7.72 (d, *J* = 7.9 Hz, 2 H, 4-MeC_6H_4), 7.32–7.31 (d, *J* = 6.4 Hz, 2 H, 4-MeC_6H_4), 2.47 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 264 (100) [M$^+$], 250 (60), 196 (40), 181 (10), 173 (60), 152 (5), 119 (60), 105 (30), 91 (50), 77 (40).

(2-Naphthyl)(4-tolyl)methanone (1w)[39]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.26 (s, 1 H, $H_{naphthyl}$), 7.94–7.91 (m, 4 H, $H_{naphthyl}$), 7.80 (d, *J* = 7.6 Hz, 2 H, 4-MeC_6H_4), 7.78 (s, 1 H, $H_{naphthyl}$), 7.33 (s, 1 H, $H_{naphthyl}$), 7.31 (d, *J* = 8.0 Hz, 2 H, 4-MeC_6H_4), 2.49 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 246 (100) [M$^+$], 231 (40), 155 (80), 127 (70), 119 (75), 91 (40), 77 (10), 51 (5).

2-Methoxybenzophenone (1x)[5b]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83–7.80 (d, *J* = 7.2 Hz, 2 H, H_{phenyl}), 7.57–7.54 (t, *J* = 7.2 Hz, 1 H, H_{phenyl}), 7.49–7.4 (t, *J* = 1.6 Hz, 1 H, 2-$MeOC_6H_4$), 7.45–7.43 (d, *J* = 8.0 Hz, 2 H, H_{phenyl}), 7.37–7.35 (d, *J* = 6.4 Hz, 1 H, 2-$MeOC_6H_4$), 7.06–7.02 (t, *J* = 8.0 Hz, 1 H, 2-$MeOC_6H_4$), 7.00–6.98 (d, *J* = 8.0 Hz, 1 H, 2-$MeOC_6H_4$), 3.73 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 212 (60) [M$^+$], 195 (30), 181 (10), 135 (100), 121 (20), 105 (30), 92 (18), 77 (70), 51 (25).

2-Benzoylfuran (1y)[38]
Yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89–7.88 (d, *J* = 4.2 Hz, 2 H, H_{phenyl}), 7.63–7.62 (d, *J* = 0.8 Hz, 1 H, H_{furyl}), 7.53–7.49 (m, 1 H, H_{phenyl}), 7.43–7.40 (d, *J* = 7.7 Hz, 2 H, H_{phenyl}), 7.16–7.15 (d, *J* = 3.5 Hz, 1 H, H_{furyl}), 6.52–6.51 (m, 1 H, H_{furyl}).

MS (EI): *m*/*z* (%) = 172 (80) [M$^+$], 105 (100), 95 (80), 77 (60), 67 (30), 51 (10).

2-(4-Methoxybenzoyl)furan (1z)[41]
Yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.02–8.00 (d, *J* = 8.8 Hz, 2 H, 4-$MeOC_6H_4$), 7.66–7.65 (d, *J* = 0.9 Hz, 1 H, H_{furyl}), 7.21–7.20 (d, *J* = 3.5 Hz, 1 H, H_{furyl}), 6.97–6.95 (d, *J* = 8.8 Hz, 2 H, 4-$MeOC_6H_4$), 6.57–6.56 (t, *J* = 1.7 Hz, 1 H, H_{furyl}), 3.86 (s, 3 H, OCH_3).

MS (EI): *m*/*z* (%) = 202 (80) [M$^+$], 172 (20), 135 (100), 95 (80), 77 (60), 67 (30), 51 (10).

2-[4-(Trifluoromethyl)benzoyl]furan (1aa)
White solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.09–8.08 (d, *J* = 8.1 Hz, 2 H, 4-$F_3CC_6H_4$), 7.78–7.76 (d, *J* = 8.2 Hz, 2 H, 4-$F_3CC_6H_4$), 7.74–7.73 (d, *J* = 0.9 Hz, 1 H, H_{furyl}), 7.29–7.28 (d, *J* = 3.5 Hz, 1 H, H_{furyl}), 6.64–6.63 (t, *J* = 1.7 Hz, 1 H, H_{furyl}).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 181.5, 152.3, 147.9, 140.4, 134.1 (q, *J* = 32.9 Hz), 129.9, 125.7 (q, *J* = 4.3 Hz), 122.8, 121.3 (q, *J* = 271.3 Hz), 112.8.

MS (EI): *m*/*z* (%) = 240 (100) [M$^+$], 173 (80), 172 (40), 95 (80), 77 (60), 67 (40), 51 (5).

HRMS (EI): *m*/*z* [M$^+$] calcd for $C_{12}H_7F_3O_2$: 240.0393; found: 240.0393.

(2-Furyl)(2-naphthyl)methanone (1ab)[42]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54 (s, 1 H, $H_{naphthyl}$), 8.04–7.96 (m, 1 H, $H_{naphthyl}$), 7.95–7.91 (t, *J* = 7.6 Hz, 2 H, $H_{naphthyl}$), 7.79– 7.77 (d, *J* = 8.0 Hz, 1 H, $H_{naphthyl}$), 7.64–7.62 (d, *J* = 6.4 Hz, 1 H, H_{furyl}), 7.61–7.56 (m, 2 H, $H_{naphthyl}$), 7.37–7.31 (d, *J* = 2.8 Hz, 1 H, H_{furyl}), 6.65–6.64 (t, *J* = 1.8 Hz, 1 H, H_{furyl}).

MS (EI): *m*/*z* (%) = 222 (100) [M$^+$], 194 (15), 165 (20), 155 (55), 127 (55), 95 (15), 77 (10), 51 (5).

(2-Furyl)(2-thienyl)methanone (1ac)[43]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19–8.18 (d, *J* = 1.2 Hz, 1 H, H_{furyl}), 7.72–7.70 (d, *J* = 1.0 Hz, 1 H, $H_{thienyl}$), 7.69–7.68 (d, *J* = 0.8 Hz, 1 H, $H_{thienyl}$), 7.41–7.40 (d, *J* = 3.2 Hz, 1 H, H_{furyl}), 7.21–7.19 (t, *J* = 4.0 Hz, 1 H, $H_{thienyl}$), 6.62–6.61 (q, *J* = 2.0 Hz, 1 H, H_{furyl}).

MS (EI): *m*/*z* (%) = 178 (95) [M^+], 111 (100), 95 (30), 39 (25).

1-Phenylhexan-1-one (1ad)[29]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97–7.94 (m, 2 H, H_{phenyl}), 7.54–7.52 (t, *J* = 3.8 Hz, 1 H, H_{phenyl}), 7.47–7.43 (d, *J* = 5.1 Hz, 2 H, H_{phenyl}), 2.97–2.93 (t, *J* = 7.4 Hz, 2 H, C_5H_{11}), 1.81–1.72 (m, 2 H, C_5H_{11}), 1.38–1.34 (m, 4 H, C_5H_{11}), 0.93–0.89 (m, 3 H, C_5H_{11}).

MS (EI): *m*/*z* (%) = 176 (20) [M^+], 133 (10), 120 (70), 105 (100), 77 (50), 51 (10).

1-(4-Methoxyphenyl)hexan-1-one (1ae)[29]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83–7.82 (d, *J* = 4.8 Hz, 2 H, H_{phenyl}), 6.88–6.85 (d, *J* = 9.2 Hz, 2 H, H_{phenyl}), 3.80 (s, 3 H, OCH_3), 2.86–2.83 (t, *J* = 5.4 Hz, 2 H, C_5H_{11}), 1.67–1.64 (t, *J* = 7.4 Hz, 2 H, C_5H_{11}), 1.30–1.19 (m, 4 H, C_5H_{11}), 0.86–0.82 (t, *J* = 4.5 Hz, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 206 (10) [M^+], 177 (4), 163 (10), 150 (60), 135 (100), 107 (10), 77 (20).

1,2-Diphenylethanone (1af)[44]
White solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03–8.00 (d, *J* = 3.2 Hz, 2 H), 7.56–7.54 (d, *J* = 6.8 Hz, 1 H), 7.48–7.46 (q, *J* = 2.8 Hz, 2 H), 7.44–7.43 (d, *J* = 0.8 Hz, 2 H), 7.35–7.27 (m, 3 H), 4.29 (s, 2 H, CH_2).

MS (EI): *m*/*z* (%) = 196 (50) [M^+], 181 (20), 165 (5), 152 (5), 119 (100), 105 (58), 91 (40), 77 (40), 65 (20), 51 (10).

(*E*)-Chalcone (2a)[27]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06–8.04 (d, *J* = 6.8 Hz, 2 H, H_{phenyl}), 7.86–7.82 (d, *J* = 16.0 Hz, 1 H, CH=CH), 7.68–7.66 (q, *J* = 3.2 Hz, 2 H, H_{phenyl}), 7.61–7.59 (d, *J* = 3.2 Hz, 1 H, CH=CH), 7.58–7.51 (m, 3 H, H_{phenyl}), 7.45–7.43 (m, 3 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 208 (80) [M^+], 207 (100), 131 (60), 105 (50), 103 (50), 77 (60), 51 (20).

(*E*)-3-Phenyl-1-(4-tolyl)prop-2-en-1-one (2b)[27]
Yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97–7.95 (d, *J* = 8.4 Hz, 2 H, H_{phenyl}), 7.85–7.81 (d, *J* = 16.0 Hz, 1 H, CH=CH), 7.68–7.66 (q, *J* = 2.3 Hz, 2 H, H_{phenyl}), 7.58–7.54 (d, *J* = 16.0 Hz, 1 H, CH=CH), 7.45–7.43 (m, 3 H, H_{phenyl}), 7.34–7.32 (d, *J* = 8.0 Hz, 2 H, H_{phenyl}), 2.46 (s, 3 H, CH_3).

MS (EI): *m*/*z* (%) = 222 (100) [M^+], 207 (20), 179 (15), 131 (20), 119 (50), 103 (25), 91 (40), 77 (30), 51 (10).

(*E*)-4-(3-Phenylacryloyl)benzonitrile (2c)[27]
Yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.10–8.08 (d, *J* = 8.0 Hz, 2 H, H_{phenyl}), 7.86–7.83 (m, 1 H, CH=CH), 7.82–7.80 (m, 2 H, H_{phenyl}), 7.66–7.65 (d, *J* = 15.8 Hz, 1 H, CH=CH), 7.49–7.44 (m, 4 H, H_{phenyl}), 7.28 (m, 1 H, H_{phenyl}).

MS (EI): *m*/*z* (%) = 233 (80) [M^+], 207 (100), 179 (15), 131 (20), 119 (50), 103 (20), 91 (40), 77 (45), 51 (20).

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