Molybdenum Hexacarbonyl Mediated Alkoxycarbonylation of Aryl Halides

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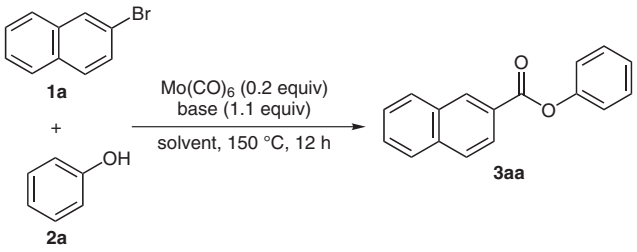
Abstract: $Mo(CO)_6$-mediates the alkoxycarbonylation of aryl halides in their reaction with alcohols to afford arenecarboxylic acid esters. The molybdenum carbonyl complexes act as the catalyst and the source with carbon monoxide. The alkoxycarbonylation proceeds with a small excess of carbon monoxide in the form of $Mo(CO)_6$ and the procedure is simple compared to the conventional method, which uses palladium catalyst under gaseous carbon monoxide. Using this procedure, a variety of carboxylic acid esters were prepared.

Key words: molybdenum, carbonyl complexes, carbonylation, esterification, acylation

Palladium-catalyzed carbonylation reactions of aryl and vinyl halides were first reported by R. F. Heck in the mid-1970s.[1] Since then, great progress has been made because it is a powerful synthetic method for the preparation of carboxylic acids and their derivatives from simple building blocks such as organic halides.[2,3] Traditionally, these reactions require a large excess of carbon monoxide because the reaction proceeds under gaseous carbon monoxide. To avoid using gaseous carbon monoxide, various carbon monoxide sources, such as aldehydes, formamide, acetic formic anhydride, and acid chlorides have been tested.[4] Larhed and co-workers reported that the group 6 metal carbonyl complexes [$M(CO)_6$, M = Cr, Mo, and W] could be used as a condensed source of carbon monoxide in the palladium-catalyzed carbonylation reactions of aromatic halides.[5] Recently, our group reported that molybdenum carbonyl complexes could be used not only as the carbon monoxide source but also as the catalyst for the carbamoylation of aryl halides, eliminating the need to use the palladium catalyst. The reaction proceeds efficiently with 0.2 equivalents of molybdenum hexacarbonyl [$Mo(CO)_6$] which constitutes 20 mol% of molybdenum catalyst and 1.2 equivalents of carbon monoxide.[6] Herein, we report a simple and efficient alkoxycarbonylation of aryl halides with a low loading of $Mo(CO)_6$.[7]

Firstly, we examined the alkoxycarbonylation of 2-bromonaphthalene (**1a**) with phenol (**2a**) in the presence of base and 0.20 equivalents of $Mo(CO)_6$ (Table 1). When 2-bromonaphthalene was treated with $Mo(CO)_6$ (0.20 equiv), phenol (1.5 equiv), tributylamine (1.1 equiv), and tetraethylammonium chloride (0.2 equiv) in diglyme at 150 °C, phenyl naphthalene-2-carboxylate (**3aa**) was obtained in 82% yield (entry 1). The yield was not affected when tetraethylammonium chloride was omitted (entry 2).[8] Polar solvents such as *N*,*N*-dimethylformamide afforded a lower yield (entry 3) and inorganic bases such as potassium carbonate gave trace amounts of product (entry 4).

Table 1 $Mo(CO)_6$-Mediated Alkoxycarbonylation of 2-Bromonaphthalene[a]



Entry	Solvent	Base	Yield (%)
1	diglyme	Bu_3N	82[b]
2	diglyme	Bu_3N	82
3	DMF	Bu_3N	68
4	diglyme	K_2CO_3	3

[a] Reaction conditions: **1a** (0.50 mmol), **2a** (0.75 mmol), $Mo(CO)_6$ (0.10 mmol), base (0.55 mmol), solvent (5 mL), 150 °C, 12 h.
[b] Et_4NCl (0.10 mmol) was added.

Next, the optimized reaction conditions were employed for the alkoxycarbonylation of 2-bromonaphthalene (**1a**) with different alcohols **2a**–**n** (Table 2). The alkoxycarbonylation reactions proceeded in good yields with monosubstituted phenols **2b**–**e** regardless of the position and electronic properties of the substituents on the phenol ring (entries 2–5). While 3,5-dimethylphenol (**2f**) gave the corresponding ester **3af** in 75% yield, 2,6-dimethylphenol (**2g**) only afforded the desired product **3ag** in 32% yield due to steric hindrance (entries 6 and 7). Naphthalen-1-ol reacted well with 2-bromonaphthalene to give ester **3ah** in 83% yield (entry 8). Although benzyl alcohol could be utilized to afford benzyl naphthalene-2-carboxylate (**3ai**), the yield decreased to 59% (entry 9). Lower yields were also observed when we used alkyl alcohols **2j**–**m** (entries 10–13). Primary and secondary alkyl alcohols could be converted into the corresponding alkyl naphthalenecarboxylates **3aj**–**am** in 48–56% yields. We also tested the

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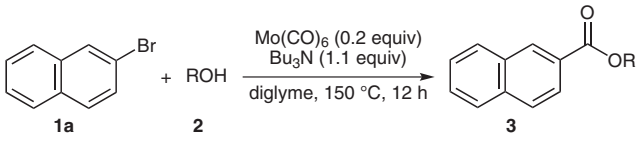
reaction with tertiary alcohols such as *tert*-butyl alcohol, however, the desired esters were not obtained. It is noteworthy that this molybdenum-mediated reaction is water-tolerant and naphthalene-2-carboxylic acid (**3an**) could be obtained in 45% yield in the reaction with water (entry 14). Throughout these examples, the aryl halide was recovered when the yield of the ester was low.

Table 2 Scope of Alcohols for $Mo(CO)_6$-Mediated Alkoxycarbonylation of 2-Bromonaphthalene[a]



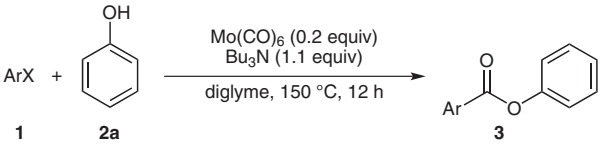
1a **2** **3**

Entry	Substrate	R	Product	Yield (%)
1	**2a**	Ph	**3aa**	82
2	**2b**	2-$MeOC_6H_4$	**3ab**	73
3	**2c**	3-$MeOC_6H_4$	**2ac**	82
4	**2d**	4-$MeOC_6H_4$	**3ad**	78
5	**2e**	4-$MeO_2CC_6H_4$	**3ae**	70
6	**2f**	3,5-$Me_2C_6H_3$	**3af**	75
7	**2g**	2,6-$Me_2C_6H_3$	**3ag**	32
8	**2h**	1-naphthyl	**3ah**	83
9	**2l**	Bn	**3ai**	59
10	**2j**	4-$MeOC_6H_4(CH_2)_2$	**3aj**	48
11	**2k**	*i*-Bu	**3ak**	51[b]
12	**2l**	$(CH_2)_4Me$	**3al**	56[b]
13	**2m**	cyclopentyl	**3am**	52[b]
14	**2n**	H	**3an**	45[c]

[a] Reaction conditions: **1a** (0.50 mmol), **2** (0.75 mmol), $Mo(CO)_6$ (0.10 mmol), Bu_3N (0.55 mmol), diglyme (5 mL), 150 °C, 12 h, unless otherwise stated.
[b] Alcohol **2k** or **2l** (2.50 mmol) was used.
[c] H_2O (3.0 mmol) was used.

Further investigation of the scope of aryl halides is summarized in Table 3. Both iodobenzene and 1-iodonaphthalene can undergo alkoxycarbonylation with phenol to give esters **3ba** and **3ca** in 81 and 89% yields, respectively (entries 1 and 2). 2-, 3- and 4-Iodoanisole were converted into the corresponding esters **3da–fa** in good yields regardless of the positions of the substitution on the benzene ring (entries 3–5). Aryl, heteroaryl, and vinyl bromides could be transformed to the corresponding arenecarboxylate **3ga**, heteroarenecarboxylate **3ha**, and α,β-unsaturated ester **3ia** in 62–89% yields (entry 6–8). In the reactions of 1-bromo-4-iodobenzene (**1j**), diphenyl terephthalate (**3ja′**) was obtained as the major product when using excess phenol (3 equiv) (entry 10). On the other hand, the reaction underwent chemoselective alkoxycarbonylation when we lowered the amount of phenol (1.1 equiv), to afford phenyl 4-bromobenzoate (**3ja**) as the main product in 61% yield (entry 9). To check the steric effect of aryl halide, we also tested the reaction with 2,6-dimethylphenyl iodide, however, most of the starting material was recovered and only a trace amount of the desired ester was obtained.

Table 3 Scope of Aryl Halides for $Mo(CO)_6$-Mediated Alkoxycarbonylation with Phenol[a]



1 **2a** **3**

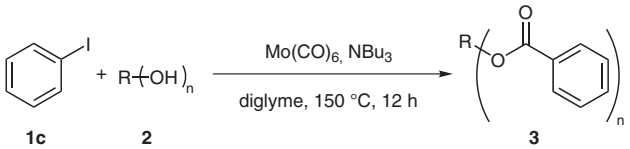
Entry	Substrate	Ar	X	Product	Yield (%)
1	**1b**	1-naphthyl	I	**3ba**	81
2	**1c**	Ph	I	**3ca**	89
3	**1d**	2-$MeOC_6H_4$	I	**3da**	75
4	**1e**	3-$MeOC_6H_4$	I	**3ea**	75
5	**1f**	4-$MeOC_6H_4$	I	**3fa**	87
6	**1g**	4-$EtO_2CC_6H_4$	Br	**3ga**	62
7	**1h**	3-thienyl	Br	**3ha**	89
8	**1i**	(*E*)-CH=CHPh	Br	**3ia**	77
9[b]	**1j**	4-BrC_6H_4	I	**3ja**	61 (7)[c]
10[d]	**1j**	4-BrC_6H_4	I	**3ja′**[e]	55 (5)[f]

[a] Reaction conditions: **1** (0.50 mmol), **2a** (0.75 mmol), $Mo(CO)_6$ (0.10 mmol), Bu_3N (0.55 mmol), diglyme (5 mL), 150 °C, 12 h unless otherwise stated.
[b] Using a 1.1:1.0 ratio of **1j** (0.55 mmol) and **2a** (0.5 mmol).
[c] Yield of diphenyl terephthalate (**3ja′**).
[d] The molar ratio of **1j**/**2a**/$Mo(CO)_6$/Bu_3N 1.0:3.0:0.4:2.2.
[e] Product was diphenyl terephthalate.
[f] Yield of **3ja**.

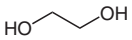
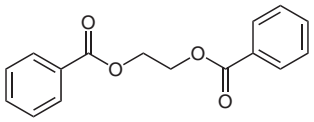
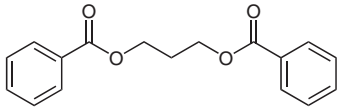
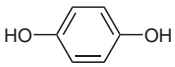
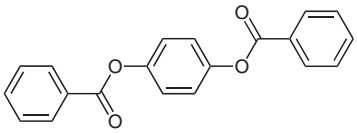
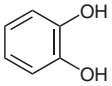
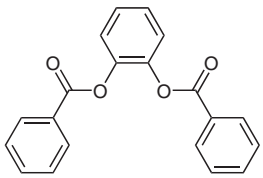
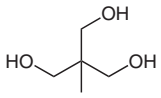
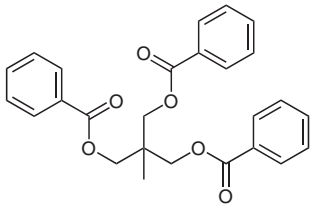
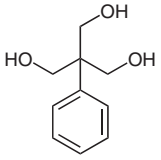
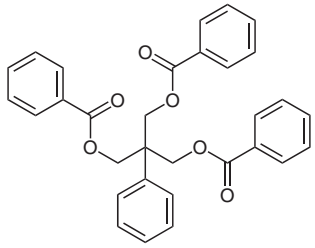
We attempted to apply the reaction to the multi-acylation of di- and triols (Table 4). When ethylene glycol (**2o**) and propylene glycol (**2p**) were subjected to the reaction with iodobenzene (**1c**) and $Mo(CO)_6$ in the molar ratio of **2o**,**p**/**1c**/$Mo(CO)_6$ 0.5:1.5:0.4, diacylation proceeded to give diesters **3co** and **3cp** in 75 and 72% yields, respectively (entries 1 and 2), Diacylation of hydroquinone and catechol also proceeded efficiently to afford phenylene dibenzoates **3cq** and **3cr** in 81 and 70% yields, respectively (entries 3 and 4). Additionally, triacylation was performed by using triols **2s** and **2t** and triesters **3cs** and **3ct** were obtained in good yields (entries 5 and 6).

Finally, we investigated the intramolecular alkoxycarbonylation reaction (Scheme 1). When 2-iodophenyl alcohols **2u** and **2v** were treated with 0.2 equivalents of $Mo(CO)_6$, five- and six-membered ring lactones **3u** and **3v** were obtained in 70 and 66%, respectively.

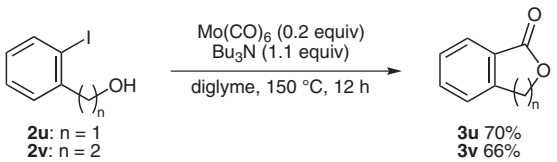
Table 4 Mo(CO)$_6$-Mediated Multi-Acylation of Di- or Triols[a]



1c **2** **3**

Entry	R(OH)$_n$	Product	Yield (%)
1[b]	 **2o**	 **3co**	75
2[b]	 **2p**	 **3cp**	72
3	 **2q**	 **3cq**	81
4	 **2r**	 **3cr**	70
5[c]	 **2s**	 **3cs**	72
6[c]	 **2t**	 **3ct**	75

[a] Reaction conditions: **1c** (1.50 mmol), **2** (0.50 mmol), Mo(CO)$_6$ (0.20 mmol), Bu$_3$N (1.10 mmol), diglyme, 150 °C, 12 h.
[b] Using a molar ratio of **1c**/**2**/Mo(CO)$_6$/Bu$_3$N 3.0:1.0:0.8:2.2.
[c] Using a molar ratio of **1c**/**2**/Mo(CO)$_6$/Bu$_3$N 4.5:1.0:1.2:3.3.



2u: n = 1
2v: n = 2

3u 70%
3v 66%

Scheme 1

In summary, we have developed an efficient molybdenum-mediated alkoxycarbonylation of aryl halides with alcohols. The procedure is simple and requires only a small excess of carbon monoxide in the form of Mo(CO)$_6$. The reaction is applicable for the multi-acylation of polyols and provides a methodology for the synthesis of a variety of carboxylic acid derivatives.

^{1}H and ^{13}C NMR spectra were recorded on two different spectrometers (^{1}H, 300 MHz; ^{13}C, 75 MHz) and (^{1}H, 400 MHz; ^{13}C, 100 MHz). Spectra were calibrated using the residual ^{1}H chemical shift in $CDCl_3$ (δ = 7.26, internal reference standards) for ^{1}H NMR, and $CDCl_3$ (δ = 77.0) for ^{13}C NMR spectra. Melting points are uncorrected. Wakogel® B-5F (Wako Pure Chemical Industries) was used for preparative TLC. Diethylene glycol dimethyl ether (diglyme) was distilled from CaH_2 and stored over MS 4Å.

$Mo(CO)_6$-Mediated Alkoxycarbonylation of Aryl Halides; General Procedure

A mixture of alcohol (0.75 mmol), aryl halide (0.50 mmol), $Mo(CO)_6$ (0.0264 g, 0.10 mmol), and Bu_3N (0.102 g, 0.55 mmol) in diglyme (5 mL) was heated at 150 °C for 12 h under a N_2 atmosphere. When the reaction was complete, the solvent was removed under reduced pressure and the residue was purified by flash column chromatography (silica gel, hexane–EtOAc, 19:1) to afford pure esters.

Phenyl Naphthalene-2-carboxylate (3aa)[9]

Yield: 102 mg (82%); white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.30–7.35 (m, 3 H), 7.46–7.52 (m, 2 H), 7.57–7.68 (m, 2 H), 7.92–8.04 (m, 3 H), 8.24 (dd, *J* = 1.5, 8.4 Hz, 1 H), 8.83 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 121.7, 125.4, 125.9, 126.7, 126.8, 127.8, 128.3, 128.6, 129.4, 129.5, 131.9, 132.4, 135.8, 151.0, 165.3.

2-Methoxyphenyl Naphthalene-2-carboxylate (3ab)

Yield: 101 mg (73%); white solid; mp 89–91 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3053, 1734, 1500, 1284, 1265, 1192, 736, 704 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.85 (s, 3 H), 7.02–7.08 (m, 2 H), 7.24–7.33 (m, 2 H), 7.56–7.67 (m, 2 H), 7.92–8.03 (m, 3 H), 8.27 (dd, *J* = 1.5, 8.5 Hz, 1 H), 8.87 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.8, 112.5, 120.8, 122.9, 125.6, 126.6, 126.7, 126.9, 127.7, 128.2, 128.4, 129.4, 131.9, 132.4, 135.7, 140.0, 151.3, 164.8.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{15}O_3$: 279.1021; found: 279.1025.

3-Methoxyphenyl Naphthalene-2-carboxylate (3ac)

Yield: 114 mg (82%); white solid; mp 72–74 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3059, 1730, 1629, 1608, 1489, 1265, 1220, 1190, 1138, 1062, 954, 775 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.84 (s, 3 H), 6.86–6.93 (m, 3 H), 7.35–7.40 (m, 1 H), 7.56–7.67 (m, 2 H), 7.91–8.02 (m, 3 H), 8.22 (dd, *J* = 1.8, 8.7 Hz, 1 H), 8.81 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.3, 107.7, 111.8, 113.9, 125.4, 126.7, 126.8, 127.8, 128.3, 128.5, 129.4, 129.8, 131.8, 132.4, 135.7, 152.0, 160.5, 165.2.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{15}O_3$: 279.1021; found: 279.1026.

4-Methoxyphenyl Naphthalene-2-carboxylate (3ad)

Yield: 109 mg (78%); white solid; mp 122–124 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3057, 1730, 1629, 1506, 1265, 1193, 1128, 1078, 1066, 736 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.86 (s, 3 H), 6.98–7.02 (d, *J* = 9.0 Hz, 2 H), 7.20–7.25 (m, 2 H), 7.58–7.69 (m, 2 H), 7.93–8.04 (m, 3 H), 8.23 (dd, *J* = 1.8, 8.6 Hz, 1 H), 8.82 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.6, 114.5, 122.5, 125.4, 126.75, 126.80, 127.8, 128.3, 128.5, 129.4, 131.8, 132.5, 135.7, 144.5, 157.3, 165.7.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{18}H_{15}O_3$: 279.1021; found: 279.1022.

4-(Methoxycarbonyl)phenyl Naphthalene-2-carboxylate (3ae)

Yield: 107 mg (70%); white solid; mp 149–151 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3055, 1745, 1720, 1265, 1190, 893, 736, 704 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.94 (s, 3 H), 7.36 (d, *J* = 8.7 Hz, 2 H), 7.55–7.67 (m, 2 H), 7.90–8.00 (m, 3 H), 8.14–8.20 (m, 3 H), 8.79 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.1, 121.7, 125.3, 126.2, 126.9, 127.7, 127.8, 128.4, 128.7, 129.4, 131.2, 132.0, 132.4, 135.8, 154.6, 164.7, 166.3.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{19}H_{15}O_4$: 307.0970; found: 307.0972.

3,5-Dimethylphenyl Naphthalene-2-carboxylate (3af)

Yield: 104 mg (75%); white solid; mp 68–70 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3055, 1730, 1618, 1591, 1265, 1220, 1192, 1138, 1072, 738, 704 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.39 (s, 6 H), 6.93–6.95 (m, 3 H), 7.57–7.67 (m, 2 H), 7.92–8.03 (m, 3 H), 8.22 (d, *J* = 8.4 Hz, 1 H), 8.81 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.2, 119.3, 125.4, 126.7, 126.9, 127.6, 127.8, 128.3, 128.5, 129.4, 131.8, 132.5, 135.7, 139.3, 150.9, 165.5.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{19}H_{17}O_2$: 277.1229; found: 277.1232.

2,6-Dimethylphenyl Naphthalene-2-carboxylate (3ag)

Yield: 44 mg (32%); white solid; mp 102–104 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3057, 1728, 1629, 1469, 1354, 1265, 1224, 1192, 1168, 1128, 1091, 1064, 950, 866, 827, 775, 736 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.25 (s, 6 H), 7.12–7.17 (m, 3 H), 7.58–7.68 (m, 2 H), 7.94–8.04 (m, 3 H), 8.26 (dd, *J* = 1.6, 8.4 Hz, 1 H), 8.86 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.4, 125.5, 125.9, 126.5, 126.8, 127.8, 128.4, 128.6, 128.62, 129.5, 130.4, 131.9, 132.5, 135.8, 148.4, 164.5.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{19}H_{17}O_2$: 277.1229; found: 277.1237.

Naphthalen-1-yl Naphthalene-2-carboxylate (3ah)

Yield: 124 mg (83%); light-yellow solid; mp 117–119 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3053, 1735, 1265, 1220, 1190, 1089, 740, 704 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45 (d, *J* = 7.2 Hz, 1 H), 7.50–7.70 (m, 5 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.93–8.07 (m, 5 H), 8.34 (dd, *J* = 1.6, 8.4 Hz, 1 H), 8.96 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 118.3, 121.3, 125.48, 125.51, 126.1, 126.5, 126.9, 127.0, 127.9, 128.1, 128.5, 128.7, 129.5, 132.1, 132.5, 134.7, 135.9, 146.9, 165.4.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{21}H_{15}O_2$: 299.1072; found: 299.1078.

Benzyl Naphthalene-2-carboxylate (3ai)[10]

Yield: 77 mg (59%); white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.47 (s, 2 H), 7.37–7.62 (m, 7 H), 7.89 (d, *J* = 8.8 Hz, 2 H), 7.96 (d, *J* = 8.0 Hz, 1 H), 8.13 (d, *J* = 8.4 Hz, 1 H), 8.68 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 66.8, 125.2, 126.6, 127.3, 127.7, 128.1, 128.2, 128.22, 128.6, 129.3, 131.2, 132.4, 135.5, 136.1, 166.5.

2-(4-Methoxyphenyl)ethyl Naphthalene-2-carboxylate (3aj)
Yield: 74 mg (48%); white solid; mp 95–97 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3055, 1714, 1631, 1514, 1265, 1228, 1195, 1130, 1095, 736, 704 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.06 (t, *J* = 7.2 Hz, 2 H), 3.77 (s, 3 H), 4.54 (t, *J* = 7.2 Hz, 2 H), 6.86 (d, *J* = 8.4 Hz, 2 H), 7.22 (d, *J* = 8.4 Hz, 2 H), 7.50–7.58 (m, 2 H), 7.85 (d, *J* = 8.4 Hz, 2 H), 7.92 (d, *J* = 8.0 Hz, 1 H), 8.03 (d, *J* = 8.4 Hz, 1 H), 8.57 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 34.4, 55.2, 65.8, 113.9, 125.2, 126.6, 127.5, 127.7, 128.1, 128.2, 129.3, 129.85, 129.91, 131.0, 132.4, 135.5, 158.3, 166.6.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{20}H_{19}O_3$: 307.1334; found: 307.1337.

2-Methylpropyl Naphthalene-2-carboxylate (3ak)
Yield: 58 mg (51%); colorless oil.

IR (NaCl, CH_2Cl_2): 3059, 2962, 1712, 1631, 1469, 1375, 1276, 1226, 1195, 1130, 1095, 989, 866, 825, 779, 761 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.09 (d, *J* = 6.6 Hz, 6 H), 2.17 (hept, *J* = 6.6 Hz, 1 H), 4.20 (d, *J* = 6.6 Hz, 2 H), 7.54–7.64 (m, 2 H), 7.91 (d, *J* = 8.4 Hz, 2 H), 7.99 (d, *J* = 7.8 Hz, 1 H), 8.10 (dd, *J* = 1.5, 8.6 Hz, 1 H), 8.64 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.2, 28.0, 71.1, 125.2, 126.6, 127.7, 127.8, 128.08, 128.13, 129.3, 130.9, 132.5, 135.5, 166.8.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{15}H_{17}O_2$: 229.1229; found: 229.1235.

Pentyl Naphthalene-2-carboxylate (3al)[11]
Yield: 68 mg (56%); colorless oil.

IR (NaCl, CH_2Cl_2): 3059, 2956, 2870, 1712, 1631, 1467, 1354, 1278, 1228, 1195, 1130, 1095, 970, 866, 779, 738, 704 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.97 (t, *J* = 7.2 Hz, 3 H), 1.40–1.51 (m, 4 H), 1.77–1.88 (m, 2 H), 4.40 (t, *J* = 6.6 Hz, 2 H), 7.51–7.61 (m, 2 H), 7.87 (d, *J* = 8.7 Hz, 2 H), 7.96 (d, *J* = 8.1 Hz, 1 H), 8.10 (dd, *J* = 1.5, 8.6 Hz, 1 H), 8.63 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.9, 22.3, 28.2, 28.4, 65.2, 125.2, 126.5, 127.67, 127.73, 128.0, 128.1, 129.3, 130.9, 132.5, 135.4, 166.8.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{16}H_{19}O_2$: 243.1385; found: 243.1387.

Cyclopentyl Naphthalene-2-carboxylate (3am)
Yield: 63 mg (52%); colorless oil.

IR (NaCl, CH_2Cl_2): 3059, 2964, 2827, 1708, 1631, 1465, 1352, 1284, 1228, 1197, 1130, 1097, 962, 866, 779, 761, 736, 704 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.64–2.06 (m, 8 H), 5.46–5.49 (m, 1 H), 7.52–7.60 (m, 2 H), 7.87 (d, *J* = 8.4 Hz, 2 H), 7.96 (d, *J* = 8.0 Hz, 1 H), 8.05 (d, *J* = 8.8 Hz, 1 H), 8.57 (s, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 23.9, 32.8, 77.8, 125.3, 126.5, 127.7, 128.0, 128.07, 128.13, 129.3, 130.8, 132.5, 135.4, 166.5.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{16}H_{17}O_2$: 241.1229; found: 241.1229.

Naphthalene-2-carboxylic Acid (3an)[12]
Yield: 39 mg (45%); white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56–7.65 (m, 2 H), 7.92 (m, 2 H), 8.00 (d, *J* = 8.4 Hz, 1 H), 8.14 (dd, *J* = 1.6, 8.4 Hz, 1 H), 8.74 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 125.4, 126.5, 126.8, 127.8, 128.3, 128.7, 129.5, 132.2, 132.4, 135.9, 172.2.

Phenyl Naphthalene-1-carboxylate (3ba)[13]
Yield: 101 mg (81%); white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.35–7.73 (m, 8 H), 7.96 (d, *J* = 8.4 Hz, 1 H), 8.13 (d, *J* = 8.1 Hz, 1 H), 8.53 (dd, *J* = 1.2, 7.5 Hz, 1 H), 9.13 (d, *J* = 8.7 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 121.8, 124.4, 125.7, 125.8, 125.9, 126.3, 128.1, 128.6, 129.5, 131.1, 131.6, 133.8, 134.2, 150.9, 165.7.

Phenyl Benzoate (3ca)[3g]
Yield: 88 mg (89%); white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.20–7.27 (m, 3 H), 7.42 (t, *J* = 8.0 Hz, 2 H), 7.49 (t, *J* = 8.0 Hz, 2 H), 7.61 (t, *J* = 7.6 Hz, 1 H), 8.20 (d, *J* = 7.6 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 121.7, 125.8, 128.5, 129.4, 129.5, 130.1, 133.5, 150.9, 165.1.

Phenyl 2-Methoxybenzoate (3da)[14]
Yield: 86 mg (75%); pale-yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.91 (s, 3 H), 7.00–7.05 (m, 2 H), 7.20–7.25 (m, 3 H), 7.38–7.54 (m, 3 H), 8.01 (d, *J* = 7.6 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.9, 112.1, 119.0 120.1, 121.8, 125.6, 129.3, 132.1, 134.2, 150.9, 159.8, 164.3.

Phenyl 3-Methoxybenzoate (3ea)[15]
Yield: 86 mg (75%); white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.85 (s, 3 H), 7.15–7.28 (m, 4 H), 7.41 (q, *J* = 8.0 Hz, 3 H), 7.70 (m, 1 H), 7.80 (d, *J* = 8.0 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 114.4, 120.1, 121.6, 122.5, 125.8, 129.4, 129.5, 130.8, 150.9, 159.6, 165.0.

Phenyl 4-Methoxybenzoate (3fa)[16]
Yield: 99 mg (87%); white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.86 (s, 3 H), 6.96 (d, *J* = 9.2 Hz, 2 H), 7.19–7.26 (m, 3 H), 7.41 (t, *J* = 8.0 Hz, 2 H), 8.14 (d, *J* = 9.2 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 113.8, 121.7, 121.8, 125.6, 129.4, 132.2, 151.0, 163.8, 164.8.

Ethyl Phenyl Terephthalate (3ga)
Yield: 84 mg (62%); white solid; mp 99–101 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3055, 1737, 1716, 1635, 1490, 1408, 1265, 1195, 1107, 1076, 1018, 736, 705 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.45 (t, *J* = 7.2 Hz, 3 H), 4.45 (q, *J* = 7.2 Hz, 2 H), 7.24–7.33 (m, 3 H), 7.46 (t, *J* = 7.8 Hz, 2 H), 8.19 (d, *J* = 8.4 Hz, 2 H), 8.28 (d, *J* = 8.4 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 61.5, 121.5, 126.0, 129.5, 129.6, 130.0, 133.2, 134.8, 150.7, 164.3, 165.6.

HRMS (ESI): *m*/*z* [M + H]$^+$ calcd for $C_{16}H_{15}O_4$: 271.0970; found: 271.0974.

Phenyl Thiophene-3-carboxylate (3ha)[16]
Yield: 91 mg (89%); white solid; mp 59–61 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3113, 1728, 1643, 1595, 1521, 1492, 1417, 1400, 1246, 1195, 1163, 1082, 1068, 931, 873, 842, 734, 688 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.19–7.25 (m, 3 H), 7.34–7.43 (m, 3 H), 7.65 (dd, *J* = 1.2, 5.2 Hz, 1 H), 8.29 (dd, *J* = 1.2, 3.2 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 121.6, 125.8, 126.3, 128.2, 129.4, 132.8, 134.0, 150.6, 161.0.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{11}H_9O_2S$: 205.0323; found: 205.0324.

Phenyl Cinnamate (3ia)[17]
Yield: 86 mg (77%); pale-yellow solid.

^1H NMR (400 MHz, $CDCl_3$): δ = 6.03 (d, *J* = 16.0 Hz, 1 H), 7.16–7.25 (m, 3 H), 7.31–7.47 (m, 5 H), 7.54–7.58 (m, 2 H), 7.87 (d, *J* = 16.0 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 117.2, 121.6, 125.7, 128.2, 128.9, 129.4, 130.6, 134.1, 146.5, 150.7, 165.3.

Phenyl 4-Bromobenzoate (3ja)[18]
Yield: 85 mg (61%); white solid.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.19–7.29 (m, 3 H), 7.40–7.44 (m, 2 H), 7.64 (d, *J* = 8.4 Hz, 2 H), 8.05 (d, *J* = 8.4 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 121.6, 126.0, 128.5, 128.8, 129.5, 131.6, 131.9, 150.7, 164.4.

Diphenyl Terephthalate (3ja′)[19]
Yield: 88 mg (55%); white solid; mp 192–194 °C (EtOAc).

IR (NaCl, CH_2Cl_2): 3057, 1732, 1454, 1265, 1192, 1078, 916, 871, 848, 740, 719 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.24–7.33 (m, 6 H), 7.46 (t, *J* = 8.0 Hz, 4 H), 8.34 (s, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 121.6, 126.2, 129.6, 130.3, 133.9, 150.8, 164.4.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{20}H_{15}O_4$: 319.0970; found: 319.0976.

Ethylene Dibenzoate (3co)[20]
Yield: 101 mg (75%); white solid.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.67 (s, 4 H), 7.44 (t, *J* = 8.0 Hz, 4 H), 7.56 (t, *J* = 8.0 Hz, 2 H), 8.07 (d, *J* = 8.0 Hz, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 62.7, 128.4, 129.7, 129.8, 133.1, 166.3.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{16}H_{15}O_4$: 271.0970; found: 271.0969.

1,3-Propylene Dibenzoate (3cp)[21]
Yield: 102 mg (72%); colorless oil.

IR (NaCl, CH_2Cl_2): 1728, 1452, 1288, 1109, 1070, 715 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 2.27 (quint, *J* = 8.0 Hz, 2 H), 4.51 (t, *J* = 8.0 Hz, 4 H), 7.42 (t, *J* = 8.0 Hz, 4 H), 7.55 (t, *J* = 8.0 Hz, 2 H), 8.04 (d, *J* = 8.0 Hz, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 28.2, 61.7, 128.3, 129.5, 130.0, 133.0, 166.5.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{17}H_{17}O_4$: 285.1127; found: 285.1125.

***p*-Phenylene Dibenzoate (3cq)[22]**
Yield: 129 mg (81%); white crystals.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.30 (s, 4 H), 7.53 (t, *J* = 8.0 Hz, 4 H), 7.66 (t, *J* = 8.0 Hz, 2 H), 8.21 (d, *J* = 8.0 Hz, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 122.6, 128.6, 129.4, 130.2, 133.7, 148.4, 165.0.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{20}H_{15}O_4$: 319.0970; found: 319.0971.

***o*-Phenylene Dibenzoate (3cr)[22]**
Yield: 111 mg (70%); colorless oil.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.32–7.40 (m, 8 H), 7.52 (t, *J* = 8.0 Hz, 2 H), 8.06 (d, *J* = 8.0 Hz, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 123.5, 126.7, 128.4, 128.7, 130.1, 133.6, 142.5, 164.2.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{20}H_{15}O_4$: 319.0970; found: 319.0967.

3-(Benzoyloxy)-2-[(benzoyloxy)methyl]-2-methylpropyl Benzoate (3cs)[23]
Yield: 156 mg (72%); colorless oil.

IR (NaCl, CH_2Cl_2): 1714, 1600, 1450, 1381, 1265, 1176, 1109, 1026, 707 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.32 (s, 3 H), 4.50 (s, 6 H), 7.42 (t, *J* = 8.0 Hz, 6 H), 7.56 (t, *J* = 8.0 Hz, 3 H), 8.03 (d, *J* = 8.0 Hz, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.5, 39.2, 66.6 128.4, 128.56, 128.65, 133.1, 166.2.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{26}H_{25}O_6$: 433.1651; found: 433.1656.

3-(Benzoyloxy)-2-[(benzoyloxy)methyl]-2-phenylpropyl Benzoate (3ct)[24]
Yield: 185 mg (75%); colorless oil;

IR (NaCl, CH_2Cl_2): 1718, 1600, 1450, 1265, 1176, 1111, 1026, 709 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.95 (s, 6 H), 7.31–7.59 (m, 14 H), 7.94 (d, *J* = 8.0 Hz, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 46.2, 65.8 126.5, 127.6, 128.4, 128.9, 129.48, 129.53, 133.1, 138.2, 166.1.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_{31}H_{27}O_6$: 495.1808; found: 495.1805.

Isobenzofuran-1(3*H*)-one (3u)[25]
Yield: 47 mg (70%); white solid.

^1H NMR (400 MHz, $CDCl_3$): δ = 5.31 (s, 2 H), 7.48–7.53 (m, 2 H), 7.67 (t, *J* = 7.6 Hz, 1 H), 7.89 (d, *J* = 7.6 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 69.6, 122.1, 125.6, 128.9, 134.0, 146.5, 171.1.

Isochroman-1-one (3v)[25]
Yield: 49 mg (66%); colorless oil.

^1H NMR (400 MHz, $CDCl_3$): δ = 3.07 (t, *J* = 6.0 Hz, 2 H), 4.54 (t, *J* = 6.0 Hz, 2 H), 7.27 (d, *J* = 7.2 Hz, 1 H), 7.40 (t, *J* = 7.6 Hz, 1 H), 7.54 (dt, *J* = 1.2, 7.6 Hz, 1 H), 8.10 (d, *J* = 8.0 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 27.8, 67.3, 125.2, 127.2, 127.6, 130.3, 133.6, 139.5, 165.1.

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