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Bu_4NI-Catalyzed C–C Bond Cleavage and Oxidative Esterification of Allyl Alcohols with Toluene Derivatives

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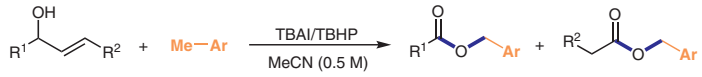

- Unusual C–C Bond Cleavage of Allyl Alcohol
- Readily Available Toluene Derivative as Precursor

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Abstract A novel oxidative esterification of 1-arylprop-2-en-1-ols with toluene derivatives catalyzed by tetrabutylammonium iodide (TBAI) is reported. The optimization of the reaction conditions illustrates that each of experiment parameters including the catalyst, solvent, and oxidant is significant for present oxidative functionalization. This metal-free protocol has a broad substrate scope including the halogen groups for further functionalization and enriches the reactivity profile of allyl alcohol and toluene derivatives. In addition, this protocol represents a new transformation of allyl alcohol involving C–C bond cleavage and C–O bond forming.

Key words esterification, allyl alcohol, toluene derivative, oxidative coupling, C–H activation

Among the cross-coupling reactions, esterification is an essential conversion in synthetic field.[1] The fascinating profile of esterification that differs from other transformation is its versatile application in academia and industry. In most cases, esterification derives largely from the alcohols and available carboxylic acids.[2] However, the usual protocols of esterification with aldehydes/alcohols as acyl sources require carboxylation in advance. On the other hand, direct esterification from aldehydes is attractive with the assistance of transition-metal reagents.[3] Except for the acyl sources, the other partner of esterification always involves the aryl/alkyl halogen or alcohol, which is usually prepared from hydrocarbon in advance or requires transition metal reagents as crucial catalyst. Furthermore, these multistep processes are inefficient with respect to the large amount of resultant waste. With the increasing demand of environmentally friendly organic processes, therefore, allyl alcohol skeleton is extensively used as synthetic intermediate in modern synthesis and academic research because of its versatility.[4] As shown in Scheme 1, allyl alcohol or its masked versions could construct versatile compounds via allylic substitution (Scheme 1, path a and c).[5] For instance, Zhang and co-authors disclosed a series of allylic alkylation catalyzed by Pd[5a,b] or dual Ir/Cu[5c] affording the valuable asymmetric compounds. Other related transformations were also intensively investigated by Loh, Li, Trost, and Hartwig group.[6] Moreover, regioselective reduction of alcohol unit of allyl alcohol skeleton could afford the valuable alkenes (Scheme 1, path b and d).[7] In 2006, Das group reported I_2-mediated reduction of allyl alcohol using $NaBH_4$ as reducing agent.[7a] Of late, Lipshutz group also obtained trisubstituted alkene via copper hydride reductions of allyl alcohol.[7c] In addition, multiple oxidation could occur due to the hydroxyl group (Scheme 1, path e).[8–10] Recently, Coelho,[8a] Lawrence,[8b] and Rao group[8c] achieved selective carbonylation of hydroxyl of allyl alcohol using λ^5-iodane reagents as oxidant, respectively. Interestingly, Zhao group reported an organoselenium-catalyzed synthesis of α,β-unsaturated aldehydes from allyl alcohol involving the migration of double C=C bond.[9] Despite these advances, further exploration of allyl alcohol skeleton is still highly desired. In particular, due to diverse products, new oxidative process affording valuable compound is considerably attractive.

With simple and abundant profile, toluene and its derivatives have found versatile application in modern synthesis.[11–13] Among these valuable transformations, functionalization of benzylic Csp^3–H of toluene derivatives was particularly important because of its ability to efficiently afford the direct construction of the carbon–carbon/heteroatom bond.[12] In our previous work, we have disclosed the coupling reaction of toluene derivatives with carbonyl com-

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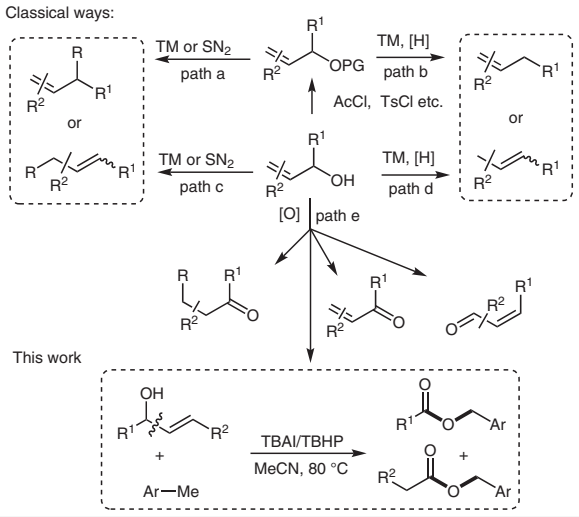


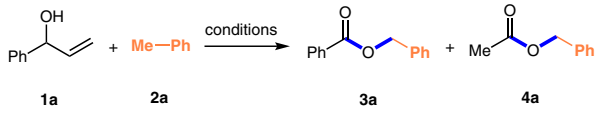
Scheme 1 Diverse conversion of allyl alcohol and its derivatives

pounds, isocyanides, and olefins, respectively.[14] And a careful literature survey illustrated that the cross-coupling between allyl alcohols and toluene derivatives via Csp^3–H functionalization was still underexploited. As a continuation of our previous investigation,[14,15] herein we report the reaction between allyl alcohol and toluene derivative towards a new esterification process.

Initially, we used 1-phenylprop-2-en-1-ol (**1a**) and toluene (**2a**) as the model substrates in MeCN to optimize the reaction conditions. As described in Table 1, poor performance was observed when using copper salt as the catalyst and TBHP as the oxidant (Table 1, entries 1, 2). To our delight, iron salt as catalyst increased the yields up to 35% for **3a** and 19% for **4a** (entries 3–5). No desired product was achieved when other metal-free catalysts, such as TBAB, NIS, or I_2 were used (entries 6–8). To our delight, 71% of **3a** and 75% of **4a** were obtained when tetrabutylammonium iodide (TBAI) was used as the catalyst (entry 10). Then, other solvents including EtOAc, PhCl, and DCE were screened, which did not lead to any further improvement (entries 11–14). After that, oxidants such as H_2O_2, $(t\text{-BuO})_2$ and $K_2S_2O_8$ were also examined (entries 15–17).

With the optimal conditions in hand, we next investigated the substrates scope of this present transformation. First, different substituents on the arene ring of 1-arylprop-2-en-1-ol were evaluated. As shown in Scheme 2, electron-donating groups on both *ortho*- and *para*-positions of the aromatic ring of 1-arylprop-2-en-1-ol were well tolerated (Scheme 2, → **3b–d** and **3h**,**i**). Furthermore, halogen atoms (Scheme 2, → **3e–g** and **3j**) for further functionalization were well tolerated affording moderate to good yields. It was also worthy to note that the simultaneous esterification of toluene was achieved in moderate yields (→ **4a**). Additionally, the aryl group in allyl alcohol seemed to be necessary since no desired products were detected when the

Table 1 Optimization of the Reaction Conditions[a]



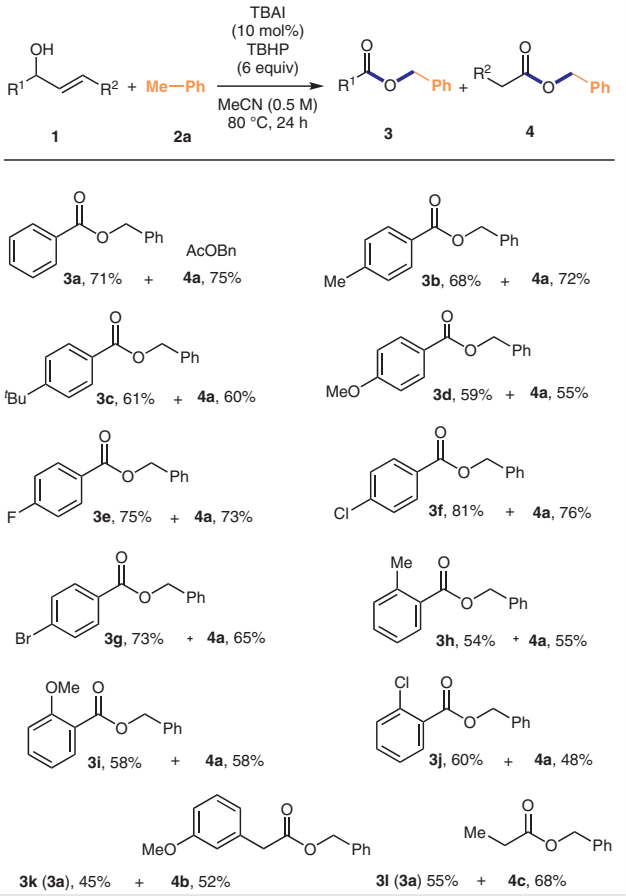
1a **2a** **3a** **4a**

Entry	Catalyst	Oxidant	Solvent	Yield (%)[b]
1	CuI	TBHP	MeCN	0
2	CuBr	TBHP	MeCN	<5 (**3a**) + <5 (**4a**)
3	$FeCl_3$	TBHP	MeCN	0
4	$FeBr_3$	TBHP	MeCN	35 (**3a**) + 19 (**4a**)
5	$FeBr_2$	TBHP	MeCN	22 (**3a**) + 5 (**4a**)
6	TBAB	TBHP	MeCN	0
7	NIS	TBHP	MeCN	0
8	I_2	TBHP	MeCN	0
9	KI	TBHP	MeCN	trace
10	TBAI	TBHP	MeCN	71 (**3a**) + 75 (**4a**)
11	TBAI	TBHP	EtOAc	61 (**3a**) + 54 (**4a**)
12	TBAI	TBHP	PhCl	trace
13	TBAI	TBHP	DCE	<10
14	TBAI	TBHP	THF	trace
15	TBAI	H_2O_2	MeCN	0
16	TBAI	$(t\text{-BuO})_2$	MeCN	trace
17	TBAI	$K_2S_2O_8$	MeCN	0
18	–	TBHP	MeCN	0

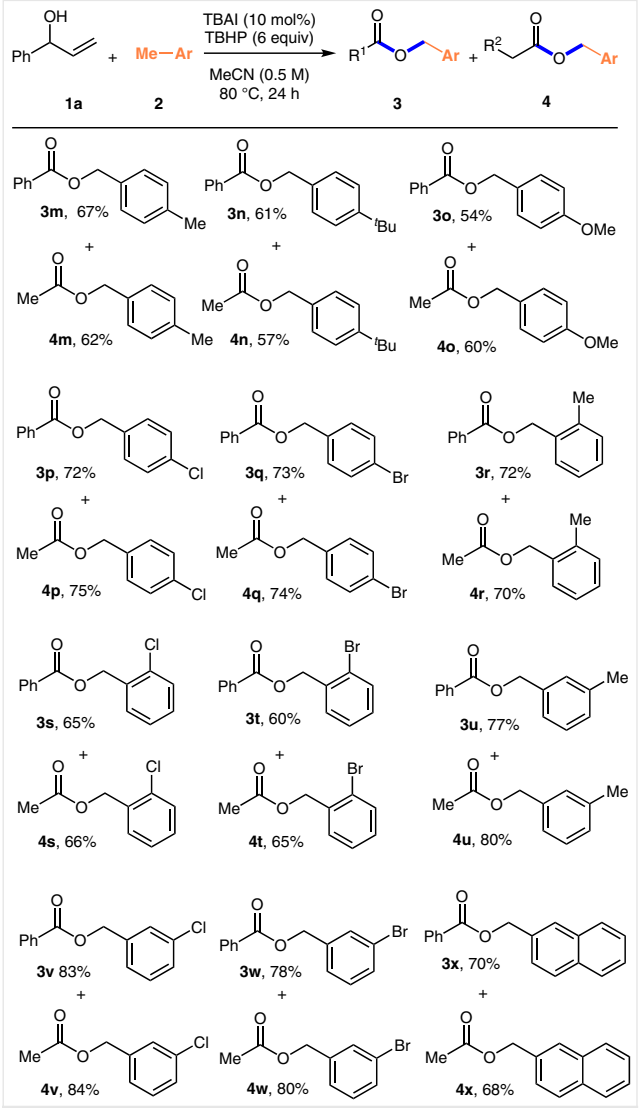
[a] Reaction conditions: 1-phenylprop-2-en-1-ol (**1a**; 1.0 mmol), toluene (**2a**; 4.0 mmol), catalyst (0.1 equiv), oxidant (6.0 equiv), solvent (2.0 mL), 80 °C, 24 h, in a sealed tube.
[b] Yields of product after silica gel chromatography.

aryl group was replaced by aliphatic groups such as ethyl and isopropyl ones. Moreover, **1k** (R^1 = Ph, R^2 = 3-MeOPh) and **1l** (R^1 = Ph, R^2 = Me), as different substrates to terminal olefins, were tested for the universality of allyl alcohol part.

Next, we examined many toluene derivatives for the present oxidative esterification. As described in Scheme 3, employment of individual substrate **2** bearing chlorine or bromine atom at *ortho*-, *meta*-, or *para*- position could achieve the product in good yield for further functionalization (Scheme 3, → **3p**,**q**, **3s**,**t** and **3v**,**w**). Moreover, electron-donating groups, such as alkyl (→ **3m**,**n**, **3r**, and **3u**) and methoxy (Scheme 3, → **3o**), were also suitable substituents for the current transformation. In addition, 2-methylnaphthalene was examined for the extended investigation. Simultaneously, benzyl acetates **4m–x** bearing diverse substituents on the arene ring were successfully synthesized in good yields, which afforded an opportunity for functionalized benzyl alcohols from toluene derivatives via direct C–H activation. Unfortunately, no reaction occurred when alkylarenes such as ethylbenzene and ibuprofen were used as components.



Scheme 2 Scope of the reaction with respect to the substrate **1**. *Reagents and conditions*: **1** (1.0 mmol), **2a** (4.0 equiv), TBAI (0.1 equiv), TBHP (6.0 equiv), MeCN (2.0 mL), 80 °C, 24 h. Isolated yields after silica gel chromatography are shown.

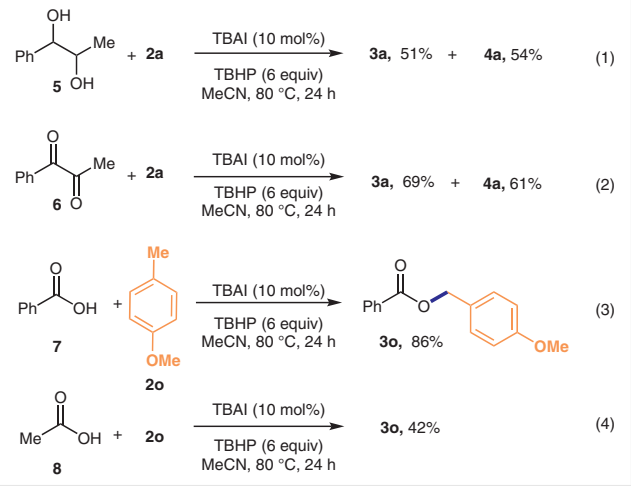


Scheme 3 Scope of the reaction with respect to the substrate **2**. *Reagents and conditions*: **1a** (1.0 mmol), **2** (4.0 equiv), TBAI (0.1 equiv), TBHP (6.0 equiv), MeCN (2.0 mL), 80 °C, 24 h. Isolated yields after silica gel chromatography are shown.

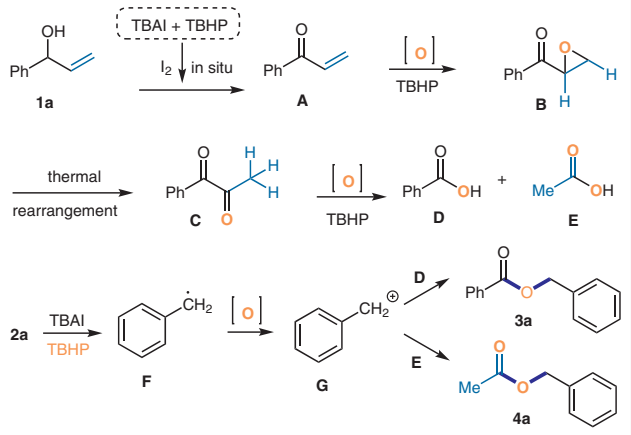
Preliminary mechanistic experiments were subsequently investigated to have a deeper insight into the present oxidative esterification. As shown in Scheme 4, 1-phenylpropane-1,2-diol (**5**) and 1-phenylpropane-1,2-dione (**6**) reacted with toluene to access the desired ester (Scheme 4, eq 1 and eq 2) in moderate yields, which suggested that these species are possible intermediates. Then, benzoic acid (**7**) and acetic acid (**8**) were treated with 1-methoxy-4-methylbenzene (**2o**) under the standard conditions, respectively. As a result, esterification could also proceed well (Scheme 4, eq 3 and eq 4). These results imply that carboxylic acids, derived from allyl alcohol via stepwise oxidation, might also be involved in this present conversion.

According to the above-mentioned experiments and previous literature,[16–22] a plausible mechanistic process is proposed to explain the oxidative conversion of allyl alcohols. As described in Scheme 5, allyl alcohol **1a** is first oxidized to the α,β-unsaturated ketone **A**,[16] which is converted to **B** via epoxidation process.[17] Then, the 1,2-dione compound **C** is formed through the thermal rearrangement.[18] As reported previously, 1,2-dione compound could be converted to carboxylic acids **D** and **E** with the assistance of oxidant via C–C bond cleavage.[19] On the other hand, toluene is oxidized by TBHP to benzyl cation **G** through the radical intermediate **F**.[20–22] Finally, the nucleophilic electrophilic reaction between **D** and **G** provides the product **3a**, meanwhile, coupling between **E** and **G** yields the product **4a**.

In summary, we have developed an efficient protocol for the oxidative esterification of allyl alcohol with toluene derivatives. This protocol represents a new reactivity profile of allyl alcohol under oxidative conditions. It is proposed to proceed through C–C bond cleavage of 1-arylprop-2-en-1-ol and simultaneous benzylic Csp^3–H activation of toluene



Scheme 4 Preliminary mechanistic investigation



Scheme 5 Possible mechanism for esterification of allyl alcohols with toluene derivatives

derivatives. This current transformation demonstrates a wide substrate scope and tolerates carbon–halogen bonds for further functionalization.

NMR spectra were recorded on Bruker AC-500 spectrometer (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR) with $CDCl_3$ as the solvent and TMS as internal reference. ^{1}H NMR spectral data were reported as follows: chemical shift (δ, ppm), multiplicity, integration, and coupling constant (Hz). ^{13}C NMR spectral data were reported in terms of the chemical shift. Standard abbreviations were used to indicate multiplicities. Low-resolution mass spectra were obtained on a Shimadzu LCMS-2010EV spectrometer in ESI mode. High-resolution mass spectra (HRMS) were recorded on a Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS instrument. Melting points were obtained on an X-4 digital melting point apparatus without correction. Chemical yields are referred to pure isolated product. Purification of products was accomplished by column chromatography packed with silica gel. Unless otherwise stated, all reagents were commercially purchased and used without further purification.

Esterification of Allyl Alcohols 1 with Toluene Derivatives 2; General Procedure

Under air atmosphere, a sealable reaction tube equipped with a magnetic stir bar and covered with a rubber septum was charged with alcohol compound **1** (1.0 mmol), toluene derivative **2** (4.0 mmol), and Bu_4NI (36.9 mg, 10 mol%) in MeCN (2.0 mL). To this mixture was added TBHP (70% wt/v in H_2O, 6.0 equiv) at r.t. The rubber septum was then replaced by a Teflon-coated screw cap, and the reaction vessel was placed in an oil bath at 80 °C for 24 h. After the completion of the reaction (monitored by TLC), the mixture was cooled to r.t. The resulting solution was poured into a mixture of sat. aq $Na_2S_2O_3$ (5 mL) and sat. aq $NaHCO_3$ (5 mL), and extracted with EtOAc (2 ×). The combined organic layers were dried (anhyd Na_2SO_4) and the solvents were removed in vacuo. The residue was purified by flash chromatography on silica gel (eluent: PE/EtOAc) to give the desired product.

Benzyl Benzoate (3a)[12a]

Colorless oil; yield: 150 mg (71%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.18 (d, *J* = 7.5 Hz, 2 H), 7.61 (t, *J* = 7.5 Hz, 1 H), 7.54 (d, *J* = 8.0 Hz, 2 H), 7.48 (dd, *J* = 12.5, 7.5 Hz, 4 H), 7.41 (d, *J* = 7.0 Hz, 1 H), 5.45 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.46, 136.19, 133.12, 130.24, 12.80, 128.70, 128.48, 128.34, 128.27, 66.76.

Benzyl 4-Methylbenzoate (3b)[23]

Colorless oil; yield: 154 mg (68%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.03 (d, *J* = 8.1 Hz, 2 H), 7.49 (d, *J* = 7.3 Hz, 2 H), 7.43 (t, *J* = 7.4 Hz, 2 H), 7.40–7.35 (m, 1 H), 7.29–7.24 (m, 2 H), 5.40 (s, 2 H), 2.43 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.51, 143.75, 136.28, 129.79, 129.15, 128.62, 128.22, 128.16, 127.46, 66.52, 21.68.

Benzyl 4-(*tert*-Butyl)benzoate (3c)[24]

Colorless oil; yield: 163.5 mg (61%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.11 (d, *J* = 8.0 Hz, 2 H), 7.50 (d, *J* = 8.5 Hz, 4 H), 7.43 (t, *J* = 7.0 Hz, 2 H), 7.38 (t, *J* = 7.5 Hz, 1 H), 5.43 (s, 2 H), 1.39 (s, 9 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.46, 156.72, 136.39, 129.73, 128.65, 128.23, 128.17, 127.50, 125.45, 66.51, 35.12, 31.20.

Benzyl 4-Methoxybenzoate (3d)[12a]

Colorless oil; yield: 143 mg (59%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.10 (d, *J* = 9.0 Hz, 2 H), 7.50 (d, *J* = 7.5 Hz, 2 H), 7.43 (t, *J* = 7.5 Hz, 2 H), 6.95 (d, *J* = 8.5 Hz, 1 H), 5.39 (s, 2 H), 3.86 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.20, 163.50, 136.39, 131.79, 128.62, 128.20, 128.15, 122.58, 113.69, 66.42, 55.42.

Benzyl 4-Fluorobenzoate (3e)[24]

Colorless oil; yield: 173 mg (75%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.14 (dd, *J* = 9.0, 5.5 Hz, 2 H), 7.51 (d, *J* = 7.5 Hz, 2 H), 7.45 (t, *J* = 7.5 Hz, 2 H), 7.42–7.38 (m, 1 H), 7.12 (t, *J* = 8.5 Hz, 2 H), 5.42 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.85, 165.84 (*J* = 251.25 Hz), 136.08, 132. 32 (*J* = 10.0 Hz), 128.70, 128.39, 128.30, 126.50 (*J* = 10.0 Hz), 115.55 (*J* = 22.5 Hz), 66.86.

Benzyl 4-Chlorobenzoate (3f)[24]

Colorless oil; yield: 200 mg (81%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.05 (d, *J* = 7.5 Hz, 2 H), 7.50 (d, *J* = 7.5 Hz, 2 H), 7.48–7.37 (m, 5 H), 5.41 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.57, 139.52, 135.88, 131.15, 128.77, 128.70, 128.64, 128.43, 128.31, 66.98.

Benzyl 4-Bromobenzoate (3g)[23]

Colorless oil; yield: 212 mg (73%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.97 (d, *J* = 8.5 Hz, 2 H), 7.59 (d, *J* = 8.5 Hz, 2 H), 7.49 (d, *J* = 7.5 Hz, 2 H), 7.46–7.38 (m, 3 H), 5.41 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.69, 135.85, 131.78, 131.29, 129.09, 128.71, 128.44, 128.31, 128.23, 67.01.

Benzyl 2-Methylbenzoate (3h)[24]

Colorless oil; yield: 122 mg (54%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.05 (d, *J* = 8.0 Hz, 1 H), 7.52 (d, *J* = 7.5 Hz, 2 H), 7.45 (dd, *J* = 7.0, 2.0 Hz, 3 H), 7.40 (t, *J* = 7.0 Hz, 1 H), 7.29 (t, *J* = 7.0 Hz, 2 H), 5.42 (s, 2 H), 2.70 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.37, 140.44, 136.25, 132.14, 131.79, 130.78, 129.53, 128.67, 128.28, 128.26, 125.80, 66.56, 21.91.

Benzyl 2-Methoxybenzoate (3i)[12a]

Colorless oil; yield: 140 mg (58%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.88 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.50–7.46 (m, 3 H), 7.41 (t, *J* = 7.5 Hz, 2 H), 7.36 (t, *J* = 7.0 Hz, 1 H), 6.99 (t, *J* = 7.5 Hz, 2 H), 5.39 (s, 2 H), 3.90 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.93, 159.40, 136.32, 133.72, 131.79, 128.57, 128.11, 120.15, 119.93, 112.11, 66.50, 55.95.

Benzyl 2-Chlorobenzoate (3j)[12a]

Colorless oil; yield: 148 mg (60%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.90 (dd, *J* = 7.5, 1.5 Hz, 1 H), 7.51 (d, *J* = 7.5 Hz, 2 H), 7.49–7.37 (m, 5 H), 7.31 (t, *J* = 7.5 Hz, 1 H), 5.43 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.47, 135.66, 133.89, 132.72, 131.59, 131.16, 130.00, 128.69, 128.44, 128.43, 126.66, 67.32.

4-Methylbenzyl Benzoate (3m)[24]

Colorless oil; yield: 151 mg (67%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.17 (d, *J* = 7.5 Hz, 2 H), 7.58 (t, *J* = 7.5 Hz, 1 H), 7.47 (t, *J* = 7.5 Hz, 2 H), 7.43 (d, *J* = 7.5 Hz, 2 H), 7.26 (d, *J* = 7.5 Hz, 2 H), 5.41 (s, 2 H), 2.43 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.47, 138.09, 133.21, 133.05, 130.35, 129.78, 129.37, 128.47, 128.44, 66.73, 21.28.

4-(*tert*-Butyl)benzyl Benzoate (3n)[25]

Colorless oil; yield: 163 mg (61%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.11 (d, *J* = 7.5 Hz, 2 H), 7.57 (t, *J* = 7.5 Hz, 1 H), 7.46–7.42 (m, 6 H), 5.37 (s, 2 H), 1.36 (s, 9 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.54, 151.33, 133.11, 133.01, 130.28, 129.76, 128.39, 128.16, 125.57, 66.61, 34.65, 31.37.

4-Methoxybenzyl Benzoate (3o)[26]

Colorless oil; yield: 131 mg (54%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.11 (d, *J* = 8.0 Hz, 2 H), 7.57 (d, *J* = 7.5 Hz, 1 H), 7.47–7.43 (m, 4 H), 6.95 (d, *J* = 8.5 Hz, 2 H), 5.34 (s, 2 H), 3.83 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.53, 159.71, 133.00, 130.31, 130.13, 129.71, 128.39, 128.21, 114.02, 66.59, 55.28.

4-Chlorobenzyl Benzoate (3p)[26]

Colorless oil; yield: 177 mg (72%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.07–8.05 (m, 2 H), 7.58–7.53 (m, 1 H), 7.45–7.40 (m, 2 H), 7.40–7.33 (m, 4 H), 5.32 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.16, 134.66, 134.02, 133.15, 129.92, 129.65, 129.56, 128.75, 128.74, 128.43, 65.78, 65.77.

4-Bromobenzyl Benzoate (3q)[24]

Colorless oil; yield: 212 mg (73%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.10 (dd, *J* = 8.1, 0.9 Hz, 2 H), 7.55 (t, *J* = 7.4 Hz, 1 H), 7.50 (d, *J* = 8.3 Hz, 2 H), 7.43 (t, *J* = 7.8 Hz, 2 H), 7.32 (d, *J* = 8.3 Hz, 2 H), 5.31 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.21, 135.19, 133.22, 131.77, 129.95, 129.90, 129.74, 128.50, 122.30, 65.88.

2-Methylbenzyl Benzoate (3r)[24]

Colorless oil; yield: 163 mg (72%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.19–8.13 (m, 2 H), 7.59 (t, *J* = 7.4 Hz, 1 H), 7.49 (dt, *J* = 15.7, 7.6 Hz, 3 H), 7.36–7.26 (m, 3 H), 5.45 (s, 2 H), 2.48 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.41, 137.10, 134.10, 133.10, 130.50, 130.24, 129.75, 129.34, 128.65, 128.48, 126.15, 65.27, 19.07.

2-Chlorobenzyl Benzoate (3s)[24]

Colorless oil; yield: 160 mg (65%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.14 (dd, *J* = 8.2, 1.0 Hz, 2 H), 7.60–7.51 (m, 2 H), 7.50–7.39 (m, 3 H), 7.29 (dt, *J* = 5.7, 2.1 Hz, 2 H), 5.50 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.19, 133.81, 133.73, 133.19, 129.98, 129.83, 129.79, 129.64, 129.58, 128.48, 126.99, 64.07.

2-Bromobenzyl Benzoate (3t)[24]

Colorless oil; yield: 175 mg (60%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.17–8.15 (m, 2 H), 7.61 (dd, *J* = 8.0, 0.8 Hz, 1 H), 7.60–7.55 (m, 1 H), 7.53 (d, *J* = 7.5 Hz, 1 H), 7.46 (t, *J* = 7.7 Hz, 2 H), 7.34 (td, *J* = 7.6, 0.9 Hz, 1 H), 7.20 (td, *J* = 7.9, 1.5 Hz, 1 H), 5.48 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.12, 135.48, 133.22, 132.94, 129.98, 129.90, 129.83, 129.81, 128.51, 127.62, 123.52, 66.24.

3-Methylbenzyl Benzoate (3u)[27]

Colorless oil; yield: 150 mg (77%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.12–8.05 (m, 2 H), 7.57–7.47 (m, 1 H), 7.45–7.35 (m, 2 H), 7.33–7.22 (m, 3 H), 7.13 (d, *J* = 6.3 Hz, 1 H), 5.32 (s, 2 H), 2.35 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.35, 138.21, 136.01, 133.02, 130.19, 129.68, 129.01, 128.92, 128.52, 128.39, 125.28, 66.70, 21.36.

3-Chlorobenzyl Benzoate (3v)[27]

Colorless oil; yield: 174 mg (83%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.12 (dd, *J* = 8.1, 0.8 Hz, 2 H), 7.61–7.53 (m, 1 H), 7.50–7.41 (m, 3 H), 7.33 (ddt, *J* = 12.3, 8.4, 4.3 Hz, 3 H), 5.34 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.18, 138.20, 134.49, 133.23, 129.96, 129.91, 129.76, 128.49, 128.40, 128.15, 126.19, 65.75.

3-Bromobenzyl Benzoate (3w)[25]

Colorless oil; yield: 227 mg (78%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.19–8.07 (m, 2 H), 7.64 (s, 1 H), 7.62–7.56 (m, 1 H), 7.48 (dd, *J* = 15.6, 7.7 Hz, 3 H), 7.40 (d, *J* = 7.6 Hz, 1 H), 7.28 (t, *J* = 7.8 Hz, 1 H), 5.35 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.25, 138.38, 133.26, 131.37, 131.09, 130.24, 129.86, 129.78, 128.50, 126.70, 122.68, 65.73.

Naphthalen-2-ylmethyl Benzoate (3x)[24]

Colorless oil; yield: 183 mg (70%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.20 (d, *J* = 7.9 Hz, 2 H), 7.97 (s, 1 H), 7.96–7.88 (m, 3 H), 7.62 (t, *J* = 8.5 Hz, 2 H), 7.56 (pent, *J* = 4.6 Hz, 2 H), 7.50 (t, *J* = 7.7 Hz, 2 H), 5.60 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.54, 133.56, 133.31, 133.23, 133.15, 130.23, 129.82, 128.52, 128.49, 128.10, 127.82, 127.42, 126.41, 126.37, 125.97, 66.94.

Benzyl Acetate (4a)[13a]

Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.39 (dd, *J* = 9.3, 3.6 Hz, 4 H), 7.35 (ddd, *J* = 8.8, 7.1, 4.2 Hz, 1 H), 5.15 (s, 2 H), 2.11 (d, *J* = 1.4 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.75, 136.08, 128.59, 128.29, 128.25, 66.26, 20.93.

Benzyl 2-(3-Methoxyphenyl)acetate (4b)[28]

Colorless oil; yield: 133 mg (52%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.48–7.37 (m, 5 H), 7.33 (t, *J* = 7.9 Hz, 1 H), 6.97 (dd, *J* = 8.0, 4.9 Hz, 2 H), 6.92 (dd, *J* = 8.2, 2.5 Hz, 1 H), 5.24 (s, 2 H), 3.84 (s, 3 H), 3.74 (s, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.11, 158.64, 134.80, 134.24, 128.44, 127.42, 127.10, 127.05, 120.54, 113.73, 116.69, 65.46, 53.96, 40.21.

Benzyl Propionate (4c)[26]

Colorless oil; yield: 111 mg (68%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.38–7.316 (m, 5 H), 5.14 (s, 2 H), 2.40 (d, *J* = 7.5 Hz, 2 H), 1.83 (t, *J* = 7.5 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 174.27, 136.19, 128.57, 128.19, 66.14, 27.61, 9.13.

4-Methylbenzyl Acetate (4m)[29]

Colorless oil; yield: 105 mg (62%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32 (d, *J* = 7.9 Hz, 4 H), 7.23 (d, *J* = 7.8 Hz, 4 H), 5.13 (s, 4 H), 2.41 (s, 7 H), 2.13 (s, 6 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.88, 138.05, 133.08, 129.28, 128.51, 66.26, 21.20, 20.98.

4-(*tert*-Butyl)benzyl Acetate (4n)[30]

Colorless oil; yield: 117 mg (57%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.48 (d, *J* = 8.2 Hz, 2 H), 7.39 (d, *J* = 8.1 Hz, 2 H), 5.17 (s, 2 H), 2.14 (s, 3 H), 1.42 (s, 9 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.77, 151.24, 133.12, 128.33, 125.53, 66.17, 34.62, 31.40, 20.98.

4-Methoxybenzyl Acetate (4o)[30]

Colorless oil; yield: 108 mg (60%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28 (d, *J* = 7.9 Hz, 2 H), 7.23 (d, *J* = 7.8 Hz, 2 H), 5.13 (s, 2 H), 2.41 (s, 3 H), 2.13 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.87, 159.65, 130.12, 128.10, 113.91, 66.05, 55.15, 20.94.

4-Chlorobenzyl Acetate (4p)[29]

Colorless oil; yield: 138 mg (75%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28 (q, *J* = 8.5 Hz, 4 H), 5.04 (s, 2 H), 2.07 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.68, 134.52, 129.61, 128.69, 128.17, 65.38, 20.85.

4-Bromobenzyl Acetate (4q)[31]

Colorless oil; yield: 170 mg (74%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46 (d, *J* = 8.3 Hz, 2 H), 7.21 (d, *J* = 8.2 Hz, 2 H), 5.03 (s, 2 H), 2.08 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.69, 135.00, 131.68, 129.91, 122.23, 65.44, 20.93.

2-Methylbenzyl Acetate (4r)[13a]

Colorless oil; yield: 115 mg (70%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.37 (d, *J* = 7.2 Hz, 1 H), 7.31–7.26 (m, 1 H), 7.23 (dd, *J* = 10.3, 7.4 Hz, 2 H), 5.16 (s, 2 H), 2.39 (s, 3 H), 2.10 (d, *J* = 1.2 Hz, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.62, 136.90, 134.09, 130.37, 129.26, 128.52, 126.06, 64.59, 20.71, 18.78.

2-Chlorobenzyl Acetate (4s)[29]

Colorless oil; yield: 121 mg (66%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.41–7.36 (m, 1 H), 7.36–7.32 (m, 1 H), 7.25–7.20 (m, 2 H), 5.19 (s, 2 H), 2.09 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.51, 135.64, 133.57, 129.79, 129.50, 129.47, 126.88, 63.52, 20.73.

2-Bromobenzyl Acetate (4t)[29]

Colorless oil; yield: 149 mg (65%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.57 (dd, *J* = 8.0, 1.1 Hz, 1 H), 7.40 (dd, *J* = 7.6, 1.6 Hz, 1 H), 7.31 (td, *J* = 7.5, 1.2 Hz, 1 H), 7.19 (td, *J* = 7.8, 1.7 Hz, 1 H), 5.19 (s, 2 H), 2.14 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.57, 135.25, 132.82, 129.84, 129.72, 127.52, 123.40, 65.79, 20.85.

3-Methylbenzyl Acetate (4u)[13a]

Colorless oil; yield: 131 mg (80%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.31 (t, *J* = 7.5 Hz, 1 H), 7.25–7.17 (m, 3 H), 5.13 (s, 2 H), 2.42 (s, 3 H), 2.14 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.86, 138.24, 135.95, 129.06, 129.04, 128.53, 125.42, 66.37, 21.35, 20.98.

3-Chlorobenzyl Acetate (4v)[29]

Colorless oil; yield: 155 mg (84%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.14–8.10 (m, 3 H), 7.34 (s, 1 H), 7.29–7.25 (m, 2 H), 7.24–7.19 (m, 1 H), 5.05 (s, 2 H), 2.09 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.63, 138.02, 134.37, 129.84, 128.29, 128.10, 126.15, 65.28, 20.83.

3-Bromobenzyl Acetate (4w)[32]

Colorless oil; yield: 183 mg (80%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.48 (s, 1 H), 7.41 (d, *J* = 7.9 Hz, 1 H), 7.24 (d, *J* = 7.7 Hz, 1 H), 7.18 (t, *J* = 7.8 Hz, 1 H), 5.03 (s, 2 H), 2.07 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.61, 138.27, 131.23, 131.02, 130.13, 126.65, 122.54, 65.22, 20.88.

Naphthalen-2-ylmethyl Acetate (4x)[29]

Colorless oil; yield: 136 mg (68%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.92–7.82 (m, 4 H), 7.58–7.48 (m, 3 H), 5.32 (s, 2 H), 2.18 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.01, 133.38, 133.24, 133.16, 128.43, 128.02, 127.76, 127.42, 126.36, 126.32, 125.95, 66.50, 21.09.

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Supporting Information

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