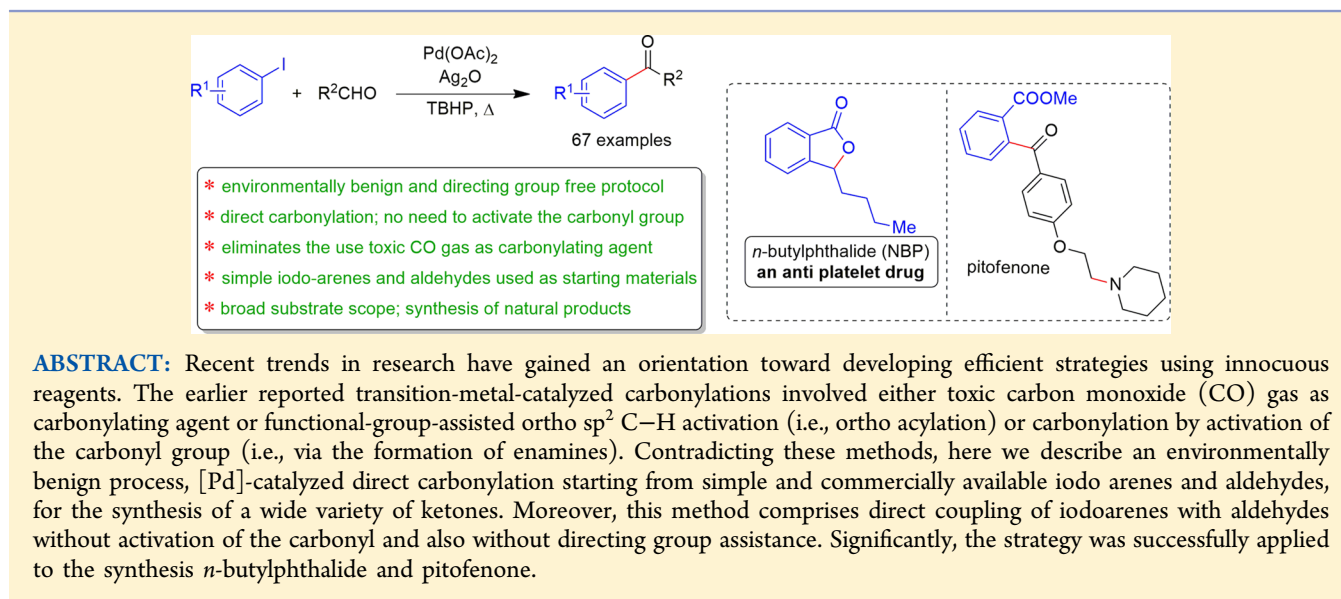


Palladium-Catalyzed Environmentally Benign Acylation

Basuli Suchand and Gedu Satyanarayana*

Department of Chemistry, Indian Institute of Technology Hyderabad, Kandi 502 285, Sangareddy, Telangana, India

S Supporting Information



INTRODUCTION

Aryl ketones are ubiquitous structural units that can be found in natural products, pharmaceuticals, agrochemicals, fragrances, dyes, and electronic materials.¹ Also, ketones have been recognized as crucial synthons in the realm of organic chemistry, as their electrophilic nature permits a broad range of bond-forming transformations. Classical methods for their synthesis include the following: (a) Friedel–Crafts acylation,² (b) Grignard/Barbier reaction with aldehydes and followed by oxidation³ or Grignard reaction with nitriles,^{3f} and (c) organometallic 1,2-additions to Weinreb amides. Among these, the Friedel–Crafts acylation has drawbacks, as it usually leads to isomeric mixtures and fails with electron-deficient arenes; the latter one provides clean products but involves a two-step process. While Weinreb amides afford pure products, the reaction works under strong nucleophilic conditions and, therefore, might not be suitable to aromatic systems with sensitive functionalities (e.g., esters, etc.). On the other hand, recently, transition-metal catalysis⁴ proved to be effective to furnish ketones such as (a) nickel-catalyzed coupling between iodoarenes and benzaldehydes,^{4a} (b) Stille coupling of acyl chlorides with organostannanes,⁵ (c) metal-mediated carbonylation between halo arenes and transmetalation reagents using toxic carbon monoxide (CO) gas as the carbonylating agent [(e.g., organostannanes, boronic acids etc.), Figure 1],⁶ (d) metal-catalyzed direct acylations through activation of the carbonyl group of aldehydes via the formation of enamine

(Figure 1),⁷ (e) directing group assisting metal-catalyzed ortho acylations (Figure 1),⁸ and (f) alkene hydroacylations.⁹

To the best of our knowledge, there are no reports on direct acylations of aldehydes without the directing group assistance or without activating the carbonyl group of the aldehydes with secondary amines, such as pyrrolidine (in situ enamine formation), under [Pd] catalysis. With this background, we intended to design and develop sustainable catalytic methods. In particular, the development of methods based on acylations becomes indispensable when the reaction can be executed: (i) under environmentally benign conditions; (ii) without the use of toxic carbon monoxide (CO) gas; (iii) using direct carbonylation without activation of the carbonyl group of aldehydes; (iv) without the assistance of functional groups (enabling the accomplishment of structurally diverse carbonyl compounds); (v) using simple, nontoxic, inexpensive, and benchtop chemicals.

In a continuation of our research interests on transition-metal-mediated efficient transformations,¹⁰ herein we disclose a novel and environmentally benign new one-pot method based on [Pd]-catalyzed direct carbonylation reaction of iodo arenes with aldehydes (alkyl/aryl), leading to alkyl–aryl and aryl–aryl ketones. This straightforward catalytic protocol does not require any directing group assistance or in situ enamine formation. Interestingly, this reaction made use of water present

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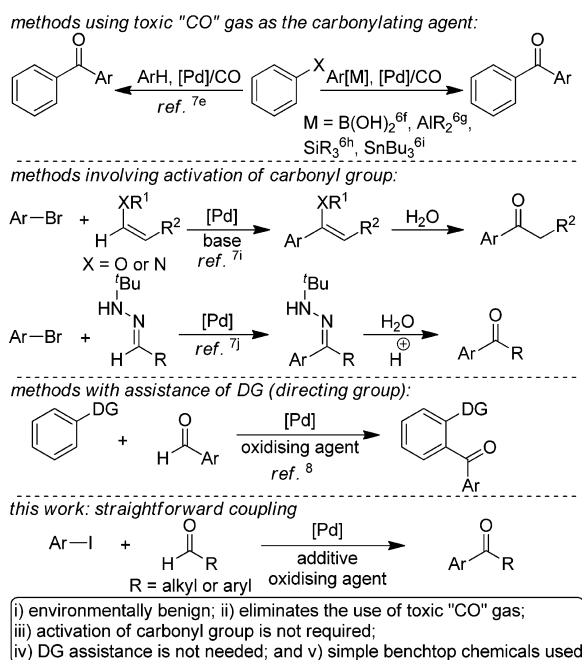


Figure 1. Reported approaches vs the present method for the carbonylation compounds.

in the oxidizing agent (aqueous TBHP) as the sole reaction medium. Notably, this process was successful by using simple and commercially available benchtop iodo arenes and aldehydes as coupling partners. This is unlike other earlier reports on [Pd] catalysis, which were applied to a limited number of examples or either limited to synthesis of benzophenone derivatives or restricted to the accomplishment of acetophenone derivatives. This protocol was successfully applied for the synthesis of both benzophenones as well as acetophenone derivatives. Thus, there was broad substrate scope and we accomplished 67 examples. In addition, significantly, the present strategy was extended to the synthesis of biologically important benzofuranes and also to the synthesis of the antiplatelet drug *n*-butylphthalide (NBP) and further applied to the synthesis of pitofenone, an antispasmodic which is used in Spasmalgon as combined drug.

RESULTS AND DISCUSSION

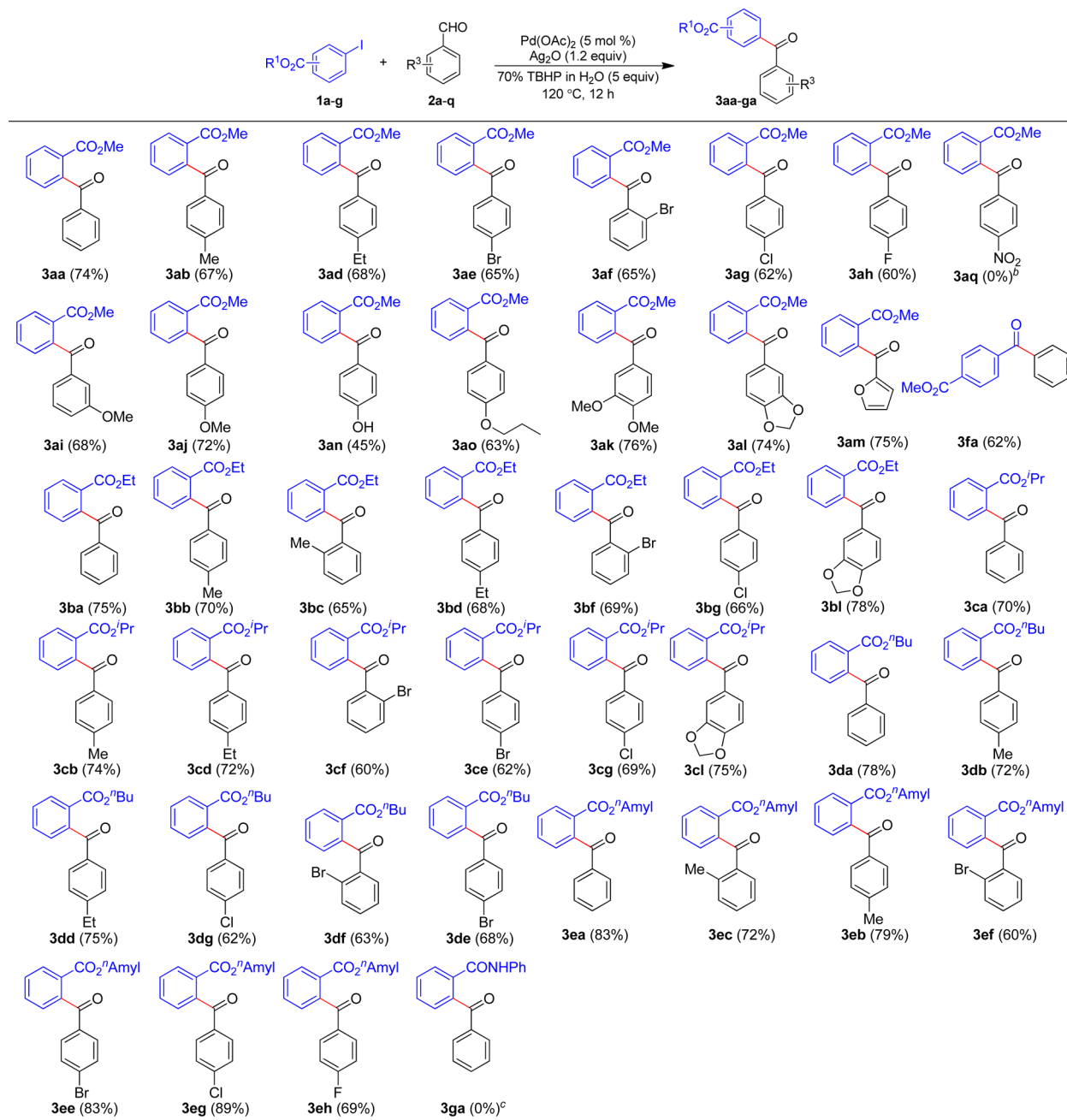
Initially, we explored the reaction between 2-iodomethylbenzoate **1a** and benzaldehyde **2a** in the presence of Pd(OAc)₂ (3 mol %)/Ag₂O (0.5 equiv) and aqueous TBHP in DCE at 80 °C for 18 h. As anticipated, the diaryl ketone **3aa** was obtained, however in poor yield (Table 1, entry 1). An increase of benzaldehyde **2a** and Pd(OAc)₂/Ag₂O improved the product **3aa** yield (Table 1, entries 2–4). Rather than using more [Pd] catalyst, a further increase of aldehyde **2a** and aqueous TBHP proved to be effective (Table 1, entries 5–7). To our delight, the reaction at elevated temperature (120 °C) without solvent DCE furnished the product **3aa** in good yield (Table 1, entry 8). Similarly, other solvents also proved to be inefficient (Table 1, entries 9 and 10). This may be due to a further increase of dilution with solvents (Table 1, entries 1–5, 9, 10, and 21). With PdCl₂ (5 mol %), the product **3aa** was obtained in moderate yield (Table 1, entry 11), while with AgOAc and Ag₂CO₃ the phenone **3aa** was formed in good and fair yields, respectively (Table 1, entries 12 and 13). Also, with TBHP in decane, the reaction furnished **3aa** in good yield (Table 1, entry

Table 1. Optimization Studies for the Carbonylation Product **3aa**^a

entry	additives (equiv)	oxidants (equiv)	temp (°C)	time (h)	yield (%) ^b
1 ^{c,d,e}	Ag ₂ O (0.5)	TBHP in H ₂ O (3.0)	80	18	34
2 ^{d,f}	Ag ₂ O (0.5)	TBHP in H ₂ O (3.0)	80	18	47
3 ^{d,f,g}	Ag ₂ O (1.2)	TBHP in H ₂ O (3.0)	80	18	52
4 ^{d,f,h}	Ag ₂ O (1.2)	TBHP in H ₂ O (3.0)	80	18	50
5 ^d	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	80	18	67
6	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	100	16	70
7 ⁱ	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	100	16	69
8	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	120	12	74
9 ^j	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	80	18	40
10 ^k	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	120	12	37
11 ^l	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	120	13	54
12	AgOAc (1.2)	TBHP in H ₂ O (5.0)	120	12	71
13	Ag ₂ CO ₃ (1.2)	TBHP in H ₂ O (5.0)	120	12	59
14	Ag ₂ O (1.2)	TBHP in Decane (5.0)	120	12	71
15	Ag ₂ O (1.2)	DTBP (5.0)	120	12	16
16	Ag ₂ O (1.2)	H ₂ O ₂ (3.0)	120	14	26
17 ^d	Ag ₂ O (1.2)	<i>m</i> -CPBA (5.0)	80	16	10
18	Ag ₂ O (1.2) + TBAI (30 mol %)	TBHP in H ₂ O (5.0)	120	11	62
19 ^m	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	120	12	–
20		TBHP in H ₂ O (5.0)	120	12	15
21 ^d	Ag ₂ O (1.2)		80	17	24
22	K ₂ CO ₃ (3.0)	TBHP in H ₂ O (5.0)	120	15	50
23	Cs ₂ CO ₃ (3.0)	TBHP in H ₂ O (5.0)	120	15	30
24 ⁿ	Ag ₂ O (1.2)	TBHP in H ₂ O (5.0)	120	14	69
25 ⁿ		TBHP in H ₂ O (5.0)	120	15	34

^aUnless otherwise mentioned, all reactions were carried out using 104.8 mg (0.40 mmol) of aryl iodide **1a**, 5 mol % of Pd(OAc)₂, 169.8 mg (1.6 mmol) of aldehyde **2a**, and TBHP in H₂O (5.0 equiv).

^bIsolated yields of chromatographically pure products. ^c1.5 equiv of aldehyde **2a** was used. ^d1.5 mL of DCE was used as solvent. ^e3 mol % of Pd(OAc)₂ was used. ^f2.0 equiv of aldehyde **2a** was used. ^g10 mol % of Pd(OAc)₂ was used. ^h20 mol % of Pd(OAc)₂ was used. ⁱ6.0 equiv of aldehyde **2a** was used. ^jReaction was carried out in 1.5 mL of MeCN. ^kReaction was carried out in 1.5 mL of DMSO. ^l5 mol % of PdCl₂ was used. ^mReaction was performed without palladium catalyst. ⁿ5 mol % of Pd(PPh₃)₄ was used.

Table 2. Scope and Generality of Formation of Aryl–Aryl Ketone 3aa–ga^a

14). On the other hand, other oxidants were found to be inferior (Table 1, entries 15–17). Even TBAI (30 mol %) as additive does not show improvement (Table 1, entry 18). The role of [Pd] catalyst was found to be andvital, as no product 3aa was formed without the catalyst (Table 1, entry 19). Also, the reaction without silver salt or the oxidant gave a very poor yield of the product 3aa (Table 1, entries 20 and 21). On the other hand, the reaction with other bases, such as K₂CO₃ and Cs₂CO₃, gave 3aa in low yields (Table 1, entries 22 and 23). The reaction in the presence of Pd(0) catalyst with and without Ag₂O furnished the product in fair and poor yields, respectively (Table 1, entries 24 and 25). Thus, the optimization studies

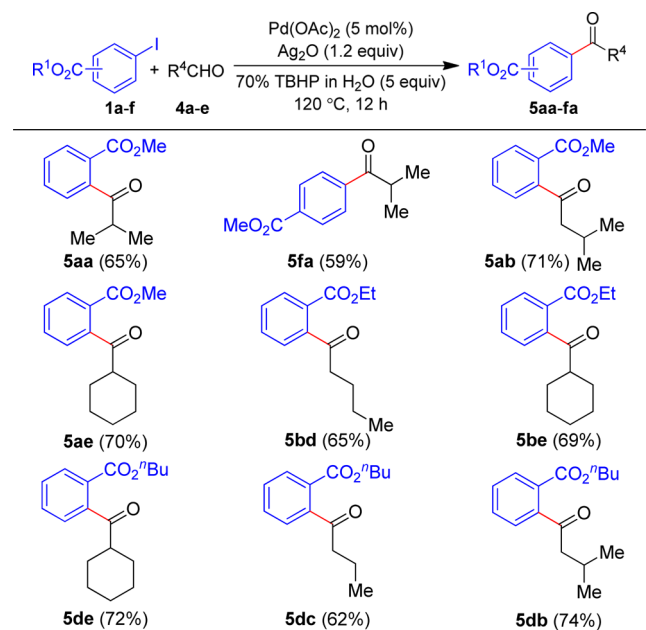
revealed that the [Pd] catalyst, silver salt, and the oxidant TBHP are very much crucial to drive the reaction.

Therefore, the best optimized conditions were identified as 1a (1.0 equiv), 2a (4.0 equiv), Pd(OAc)₂ (5 mol %)/Ag₂O (1.2 equiv), and aqueous TBHP (5.0 equiv) at 120 °C for 12 h to give 3aa (Table 1, entry 8). With these optimized conditions in hand, to check the scope and generality of the method, we explored the reaction between electron-deficient iodo arenes 1a–g and benzaldehydes 2a–q. Gratifyingly, the reaction was found amenable with various benzaldehydes 2a–q; it showed broad substrate scope and afforded the corresponding aryl–aryl ketones 3aa–ga in moderate to very good yields (Table 2). Interestingly, the reaction was successful with benzaldehydes 2

having F, Cl, and Br substituents. Notably, the reaction was also amenable with 2-furaldehyde **2m** (Table 2, **3am**). However, the reaction was unsuccessful with benzaldehyde having a strong electron-withdrawing nitro group (Table 2, **3aq**); even after many trials with varying amounts of the aldehydes and at different temperatures neither the starting materials nor the products were isolated. In fact, these observations were in good agreement with that of reactions under such oxidative conditions. This may be probably due to the radical stabilizing effect of the electron-withdrawing substituent.¹¹ Also, the reaction was unsuccessful with heterocyclic (pyridine and thiophene) aldehydes. It was also observed that the reaction with bromo arene did not afford the ketone, which implies that the more reactive iodo arenes are essential for the success of the reaction. On the other hand, the reaction with the amide **1g** was unsuccessful to give **3ga** and furnished only the reductive deiodinated byproduct in 41% yield (Table 2).

To further check the scope and applicability of the strategy, the reaction was carried out between electron-deficient iodo arenes **1a–f** and aliphatic aldehydes **4a–e** under standard conditions. To our delight, the reaction was amenable with the aliphatic aldehydes **4a–e** as well, showed very good substrate scope, furnished the corresponding alkyl–aryl ketones **5aa–fa** in moderate to good yields, and thus reveals the importance of the present protocol (Table 3).

Table 3. Scope and Generality of Formation of Alkyl–Aryl Ketone 5aa–fa^a



^aIsolated yields of chromatographically pure products **5aa–fa**.

In addition, to the spectroscopic data, the structure of aryl–aryl ketones **3** was further confirmed by single-crystal X-ray diffraction analysis of **3ah** (Figure 2); for details see the Supporting Information.

Furthermore, to show the applicability and significance of the present protocol, the reaction was also performed between simple to electron-rich iodo arenes **6a–j** and aldehydes **2a–q** or **4a–e** under established conditions. Gratifyingly, the reaction was quite successful and gave the corresponding diaryl ketones **7aa–ja** in moderate to good yields (Table 4). Notably, the

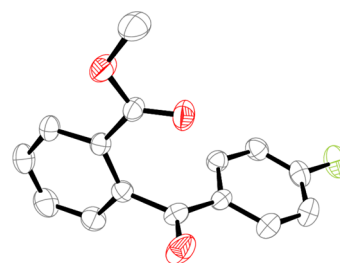
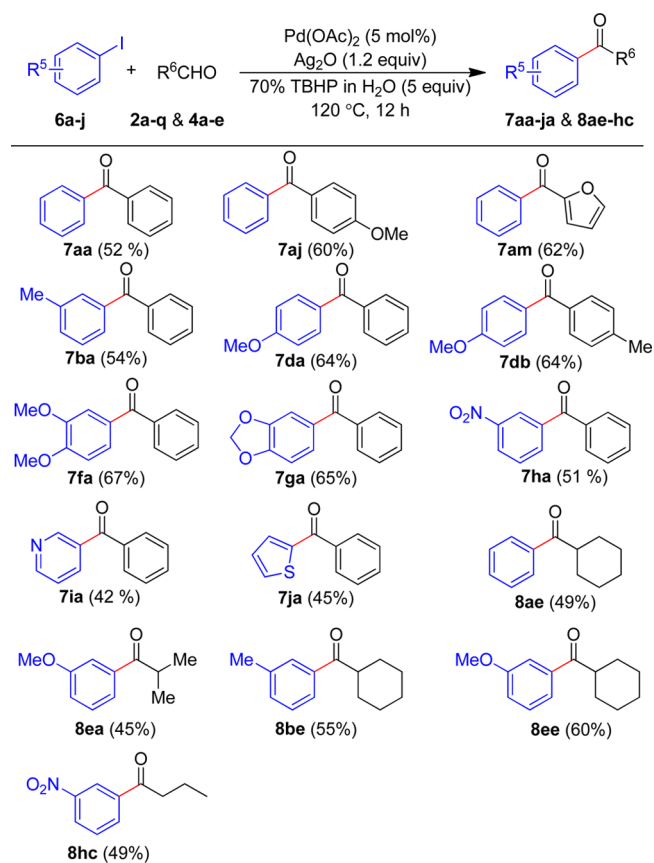


Figure 2. X-ray crystal structure of compound **3ah**. Ellipsoids are drawn at the 50% probability level.

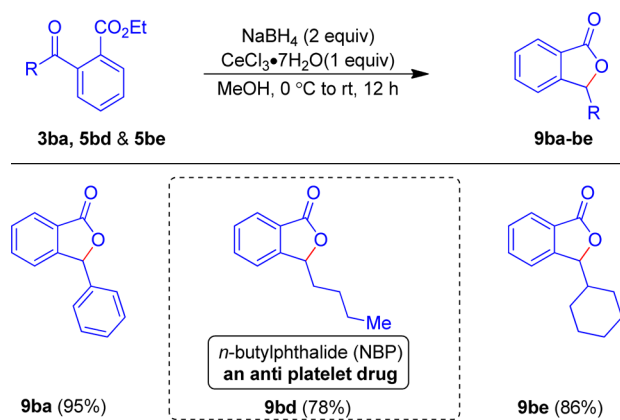
Table 4. Scope and Generality of Formation of Aryl–Aryl and Aryl–Alkyl Ketone 7aa–ja and 8ae–hc^a



^aIsolated yields of chromatographically pure products **7aa–ja** and **8ae–hc**.

reaction was compatible with strong electron-withdrawing *m*-nitroiodobenzene **6h** (Table 4, **7ha** and **8hc**). Also, it was suitable with heteroaryl iodides (Table 4, **7ia** and **7ja**). Significantly, the reaction was also amenable with the aliphatic aldehydes (**4c** and **4e**) and afforded the products **8ea–hc** in fair yields (Table 4).

To further demonstrate the synthetic utility of the present strategy, it was extended to the synthesis of benzofuranones, biologically important scaffolds. To our delight, as anticipated, selective reduction of the keto group of **3ba**, **5bd**, and **3be** with NaBH₄ and in situ intramolecular nucleophilic attack of the secondary hydroxyl group to an ester moiety afforded the benzofuranones **9ba–be** (Table 5). Significantly, this strategy was also successfully applied to the synthesis of *n*-butylph-

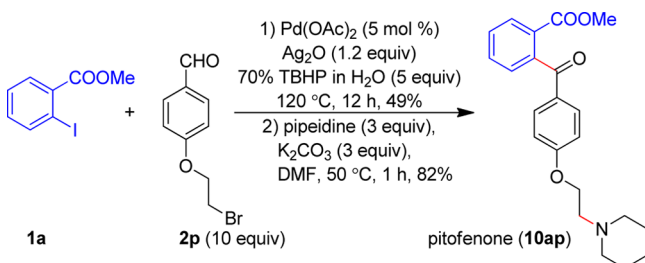
Table 5. Synthesis of Benzofuranones 9ba–be^a

^aIsolated yields of chromatographically pure products 9ba–be.

thalide (NBP)¹² 9bd. The NBP is currently serving as an antiplatelet drug in the market for ischemia-cerebral apoplexy.

Most importantly, the strategy was further applied to the synthesis of the antispasmodic pitofenone, a combined drug in Spasmalgon. This drug helps in releasing pain and spasms of smooth muscles. Notably, the present protocol proved to be efficient as it enables the synthesis of pitofenone 10ap in just two steps (Scheme 1).¹³

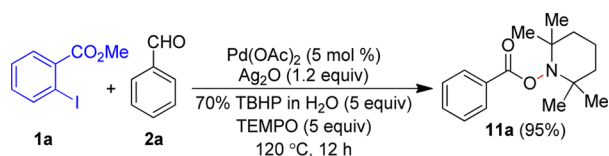
Scheme 1. Synthesis of Pitofenone 10ap



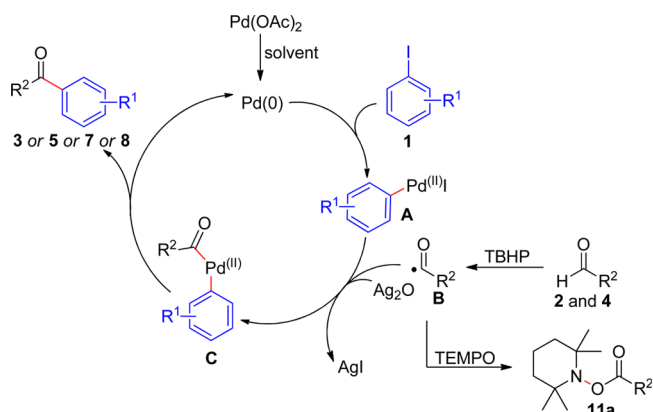
To further investigate the mechanism of the reaction, we assumed that the reaction may proceed via the formation of an acyl radical from the aldehydes (2 and 4) by TBHP. To prove the formation of radical, the reaction was performed in the presence of (2,2,6,6-tetramethylpiperidin-1-yl)oxy (TEMPO), a free radical scavenger, under standard conditions. Gratifyingly, as predicted, the aldehyde radical-trapped ester 11a was formed in 95% yield with TEMPO (Scheme 2).¹⁴ On the other hand, the iodo ester 1a was transformed into the simple methyl benzoate in 15% yield via reductive deiodination (Scheme 2).

Though the exact reaction mechanism is not clearly known, a plausible mechanism is depicted in Scheme 3. In this context, we presumed that the reaction might proceed via Pd(0)/Pd(II) intermediates^{15–17} and Ag₂O could act as a base [as reaction is working in the presence of K₂CO₃/Cs₂CO₃ (Table

Scheme 2. Synthesis of Tempoester 11a



Scheme 3. Plausible Reaction Mechanism for the Synthesis of Ketones 3/5/7/8 from Aldehydes 2 and 4



1, entries 22 and 23)]. Pd(OAc)₂ could be reduced to Pd(0) by the solvent.^{15c} Thus, oxidative addition of Pd(0) onto the Ar–I bond leads to the formation of Pd(II)-intermediate A. In an independent path, the acyl radical B formed from aldehyde by the reaction of TBHP. Now, combination of A and B would furnish the Pd(II)-intermediate C, which undergoes reductive elimination and affords the ketones 3/5/7/8 along with the Pd(0) catalyst and thus completes the catalytic cycle. However, because of the halophilic nature of Ag⁺ ions, it might be reasonable to form AgI while combining B with A. On the other hand, acceleration of the reaction in the presence of Ag₂O might also be due to the salt effect on [Pd] catalysis.¹⁸

In summary, we disclosed a simple [Pd]-catalyzed direct acylation of iodo arenes with alkyl/aryl aldehydes for the synthesis of wide variety of aryl–aryl and alkyl–aryl ketones with good substrate scope. Unlike some of the transition-metal-catalyzed reactions that made use of toxic carbon monoxide (CO) gas as carbonylating agent or activation of the carbonyl group or directing group assistance, the present protocol was successful under environmentally benign conditions, without the need to activate the carbonyl group and also without directing group assistance. Significantly, the strategy was extended to the synthesis of antiplatelet drug *n*-butylphthalide (NBP) and the antispasmodic drug pitofenone. Currently, studies for the extension of this protocol to identify other substrates as aldehyde are in progress.

EXPERIMENTAL: GENERAL CONSIDERATIONS

IR spectra were recorded on a FTIR spectrophotometer. ¹H NMR spectra were recorded on 400 MHz spectrometer at 295 K in CDCl₃; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (δ_H = 0.00 ppm) or CHCl₃ (δ_H = 7.25 ppm). ¹³C NMR spectra were recorded on a 100 MHz spectrometer at RT in CDCl₃; chemical shifts (δ ppm) are reported relative to CHCl₃ [δ_C = 77.00 ppm (central line of triplet)]. In ¹³C NMR, the nature of carbons (C, CH, CH₂, and CH₃) was determined by recording the DEPT-135 spectra. In ¹H NMR, the following abbreviations are used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sept = septet, dd = doublet of doublet, m = multiplet and br s = broad singlet. High-resolution mass spectra (HR-MS) were recorded in Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. Melting points were determined on an electrothermal melting point apparatus and are uncorrected. All small-scale reactions were carried out using a Schlenk tube. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Reactions were

generally run under argon or a nitrogen atmosphere. Solvents were distilled prior to use; petroleum ether with a boiling range of 60–80 °C was used. Acme's silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per gram of crude material).

Compounds **1a–f**, **6a–j**, **2a–q**, and **4a–e** which have been used are commercially available.

GP-1 [General Procedure for Preparation of Benzophenones and Acetophenones (3, 5, 7 and 8)]. To an oven-dried Schlenk tube were added aryl iodide **1/6** (81.3–127.3 mg, 0.40 mmol), aldehyde **2/4** (169.8–366.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). The resulting reaction mixture was stirred at 120 °C for 12 h. The progress of the product **3/5/7/8** formation was monitored by TLC until the reaction was completed. The reaction mixture was allowed to cool to room temperature, diluted with aqueous NaHCO₃ solution, and then extracted with ethyl acetate (3 × 15 mL). The organic layers were washed with saturated NaCl solution, dried (Na₂SO₄), and filtered. Evaporation of the solvent under reduced pressure and purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate) furnished the products **3/5/7/8** (32.0–117.8 mg, 42–89%) as viscous liquid/solid.

GP-2 [General Procedure for Preparation of Benzofurones (9)]. To a cold (0 °C), magnetically stirred solution of keto ester **3ba/Sbd/Sbe** (117.1–130.2 mg, 0.50 mmol) in MeOH (1 mL) were added sequentially CeCl₃·7H₂O (186.3 mg, 0.50 mmol) and NaBH₄ (38 mg, 1.0 mmol). The reaction mixture was stirred at 0 °C to room temperature for 12–24 h. The progress of the product **9ba/9bd/9be** formation was monitored by TLC. The reaction mixture was quenched with H₂O and then extracted with ethyl acetate (3 × 10 mL). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate) furnished the product **9ba/9bd/9be** (112.5–146.0 mg, 78–95%) as a liquid/solid.

Ethyl-2-benzoyl Benzoate (3aa).^{19a} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3aa** (71.1 mg, 74%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.80, R_f(**3aa**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2930, 17190, 1686, 1599, 1450, 1287, 1089, 934, 718 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.03 (d, 1H, J = 7.3), 7.73 (dd, 2H, J = 8.57 and 1.2 Hz), 7.65–7.61 (m, 1H), 7.58–7.51 (m, 2H), 7.44–7.39 (m, 1H), 3.59 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 51.1 (CH₃), 127.8 (CH), 128.5 (2 × CH), 129.2 (2 × CH), 129.6 (CH), 130.0 (CH), 131.4 (C_q), 132.4 (CH), 133.0 (CH), 137.2 (C_q), 141.6 (C_q), 166.4 (C_q), 197.0 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₁₃O₃]⁺ = [M + H]⁺ 241.0859; found 241.0868.

Methyl-2-(4-methylbenzoyl) Benzoate (3ab). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3ab** (68.1 mg, 67%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.90, R_f(**3ab**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2980, 1722, 1678, 1591, 1451, 1278, 1084, 953, 715 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.02 (dd, 1H, J = 7.8 and 0.9 Hz), 7.65–7.60 (m, 3H), 7.54 (td, 1H, J = 7.7 and 1.2 Hz), 7.38 (dd, 1H, J = 7.5 and 1.2 Hz), 7.21 (d, 2H, J = 7.8 Hz), 3.60 (s, 3H), 2.39 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.7 (CH₃), 52.1 (CH₃), 127.5 (CH), 129.2 (2 × CH), 129.4 (2 × CH), 129.5 (CH), 130.1 (CH), 130.2 (C_q), 132.3 (CH), 134.7 (C_q), 141.9 (C_q), 143.9 (C_q), 166.5 (C_q), 196.7 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₆H₁₅O₃]⁺ = [M + H]⁺ 255.1016; found 255.1016.

Methyl-2-(4-ethylbenzoyl) Benzoate (3ad).^{19b} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2d** (214.7 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl

acetate, 97:03–95:05) furnished the product **3ad** (73.0 mg, 68%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.90, R_f(**3ad**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2992, 1724, 1685, 1588, 1451, 1279, 1088, 9334, 712 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.03 (d, 1H, J = 7.8 Hz), 7.66 (d, 2H, J = 7.3 Hz), 7.61 (td, 1H, J = 7.5 and 1.4 Hz), 7.54 (td, 1H, J = 7.5 and 1.4 Hz), 7.38 (dd, 1H, J = 7.3 and 0.9 Hz), 7.24 (d, 2H, J = 7.8 Hz), 3.61 (s, 3H), 2.68 (q, 2H, J = 7.6 Hz), 1.23 (t, 3H, J = 7.6 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 15.1 (CH₃), 28.9 (t, ArCH₂CH₃), 52.2 (CH₃), 127.7 (CH), 128.0 (2 × CH), 129.1 (C_q), 129.4 (CH), 129.5 (2 × CH), 130.0 (CH), 132.3 (CH), 134.8 (C_q), 141.9 (C_q), 150.1 (C_q), 166.4 (C_q), 196.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₇H₁₇O₃]⁺ = [M + H]⁺ 269.1172; found 269.1178.

Methyl-2-(4-bromobenzoyl) Benzoate (3ae).^{19c} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2e** (296.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3ae** (83.0 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.90, R_f(**3ae**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2946, 1725, 1685, 1598, 1490, 1287, 1023, 999, 722 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04 (d, 1H, J = 7.8), 7.64 (ddd, 1H, J = 7.8, J = 5.8 and 1.4 Hz), 7.61–7.58 (m, 2H), 7.58–7.55 (m, 3H), 7.37 (dd, 1H, J = 7.5 and 1.2 Hz), 3.64 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 52.3 (CH₃), 127.6 (CH), 128.3 (C_q), 129.0 (C_q), 129.8 (CH), 130.2 (CH), 130.6 (CH), 131.8 (2 × CH), 132.6 (CH), 136.0 (C_q), 141.3 (C_q), 166.2 (C_q), 196.0 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₁₂BrO₃]⁺ = [M + H]⁺ 318.9964; found 318.9951.

Methyl-2-(2-bromobenzoyl) Benzoate (3af). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2f** (296.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3af** (76.6 mg, 60%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.90, R_f(**3af**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2960, 1722, 1679, 1591, 1458, 1287, 1093, 943, 722 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.85 (dd, 1H, J = 5.8 and 2.9, Ar–H), 7.69–7.66 (m, 1H), 7.57 (dd, 2H, J = 5.8 and 3.4 Hz), 7.47 (dd, 1H, J = 5.8 and 2.9 Hz), 7.37–7.34 (m, 1H), 7.29–7.32 (m, 2H), 3.67 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 52.5 (CH₃), 121.3 (C_q), 127.0 (CH), 129.4 (CH), 129.8 (CH), 131.0 (CH), 131.1 (C_q), 131.6 (CH), 132.5 (CH), 134.5 (CH), 138.2 (C_q), 139.9 (C_q), 167.4 (C_q), 195.3 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₁₂BrO₃]⁺ = [M + H]⁺ 318.9964; found 318.9957.

Methyl-2-(4-chlorobenzoyl) Benzoate (3ag).^{19d} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2g** (224.9 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3ag** (71.4 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.80, R_f(**3ag**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2960, 1721, 1695, 1588, 1450, 1277, 1088, 943, 723 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04 (d, 1H, J = 7.3 Hz), 7.69–7.62 (m, 3H), 7.57 (ddd, 1H, J = 7.3, 6.5, and 1.4 Hz), 7.41–7.36 (m, 3H), 3.64 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 52.3 (CH₃), 127.6 (CH), 128.9 (2 × CH), 129.0 (C_q), 129.8 (CH), 130.2 (CH), 132.5 (2 × CH), 135.6 (C_q), 139.5 (C_q), 141.3 (C_q), 166.2 (C_q), 195.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₁₂ClO₃]⁺ = [M + H]⁺ 275.0469; found 275.0476.

Methyl-2-(4-fluorobenzoyl) Benzoate (3ah).^{19e} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2h** (198.6 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3ah** (64.0 mg, 62%) as a

white solid (mp 80–83 °C). [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(1a) = 0.90$, $R_f(3ah) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2960, 1730, 1695, 1592, 1451, 1271, 1081, 932, 710 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.04$ (d, 1H, $J = 7.8$ Hz), 7.76 (dd, 2H, $J = 8.8$ and 5.3 Hz), 7.63 (ddd, 1H, $J = 7.3$, $J = 5.3$ and 1.4 Hz), 7.58–7.54 (m, 1H), 7.37 (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.08 (t, 2H, $J = 8.8$ Hz), 3.64 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.3$ (CH_3), 115.7 ($2 \times \text{CH}$), 127.6 (CH), 129.1 (C_q), 129.7 (CH), 130.2 (CH), 131.9 ($2 \times \text{CH}$), 132.5 (CH), 133.7 (C_q), 141.5 (C_q), 166.3 (C_q), 166.9 (d, $^1\text{J}_{\text{C-F}} = 255.3$ Hz), 195.5 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{15}\text{H}_{12}\text{FO}_3]^+ = [\text{M} + \text{H}]^+$ 259.0765; found 259.0774.

Methyl-2-(3-methoxybenzoyl) Benzoate (3ai). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2i** (217.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 93:07–88:12) furnished the product **3ai** (73.5 mg, 68%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), $R_f(1a) = 0.90$, $R_f(3ai) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2966, 1721, 1670, 1599, 1450, 1278, 1083, 933, 716 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.03$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.62 (td, 1H, $J = 7.4$ and 1.2 Hz), 7.55 (td, 1H, $J = 7.7$ and 1.2 Hz), 7.41–7.39 (m, 2H), 7.29 (t, 1H, $J = 7.8$ Hz), 7.18–7.16 (m, 1H), 7.08 (ddd, 1H, $J = 8.0$, $J = 2.6$ and 0.9 Hz), 3.83 (s, 3H), 3.62 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.2$ (CH_3), 55.4 (CH_3), 112.8 (CH), 119.8 (CH), 122.5 (CH), 127.8 (CH), 129.2 (C_q), 129.5 ($2 \times \text{CH}$), 129.6 (CH), 132.4 (CH), 138.5 (C_q), 141.7 (C_q), 159.8 (C_q), 166.4 (C_q), 196.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{15}\text{O}_4]^+ = [\text{M} + \text{H}]^+$ 271.0965; found 271.0968.

Methyl-2-(4-methoxybenzoyl) Benzoate (3aj). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2j** (217.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 93:07–88:12) furnished the product **3aj** (77.8 mg, 72%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), $R_f(1a) = 0.90$, $R_f(3aj) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2950, 1721, 1675, 1578, 1451, 1282, 1084, 934, 719 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.02$ (dd, 1H, $J = 7.8$ and 0.9 Hz), 7.71 (d, 2H, $J = 8.8$ Hz), 7.61 (td, 1H, $J = 7.5$ and 1.4 Hz), 7.53 (td, 1H, $J = 7.5$ and 1.4 Hz), 7.37 (dd, 1H, $J = 7.3$ and 0.9 Hz), 6.89 (d, 1H, $J = 8.8$ Hz), 3.84 (s, 3H), 3.62 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.2$ (CH_3), 55.4 (CH_3), 113.7 ($2 \times \text{CH}$), 127.7 (CH), 129.1 (C_q), 129.3 (CH), 130.1 (CH), 130.2 (C_q), 131.6 ($2 \times \text{CH}$), 132.3 (CH), 142.0 (C_q), 163.5 (C_q), 166.5 (C_q), 195.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{15}\text{O}_4]^+ = [\text{M} + \text{H}]^+$ 271.0965; found 271.0967.

Methyl-2-(3,4-dimethoxybenzoyl) Benzoate (3ak).^{20b} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2k** (265.9 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 88:12–85:15) furnished the product **3ak** (91.3 mg, 76%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 85:15), $R_f(1a) = 0.95$, $R_f(3ak) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2966, 1723, 1645, 1598, 1430, 1272, 1083, 936, 719 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.02$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.63–7.52 (m, 3H), 7.38 (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.06 (dd, 1H, $J = 8.3$ and 1.9 Hz), 6.77 (d, 1H, $J = 8.8$ Hz), 3.93 (s, 3H), 3.90 (s, 3H), 3.64 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.2$ (CH_3), 55.9 (CH_3), 56.0 (CH_3), 109.8 (CH), 110.2 (CH), 125.1 (CH), 127.7 (CH), 129.1 (C_q), 129.4 (CH), 130.0 (CH), 130.4 (C_q), 132.2 (CH), 141.8 (C_q), 149.1 (C_q), 153.3 (C_q), 166.4 (C_q), 195.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{17}\text{H}_{17}\text{O}_5]^+ = [\text{M} + \text{H}]^+$ 301.1071; found 301.1071.

Methyl-2-(benzo[d][1,3]dioxole-5-carbonyl) Benzoate (3al). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2l** (240.2 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the

crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–92:08) furnished the product **3al** (84.1 mg, 74%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), $R_f(1a) = 0.95$, $R_f(3al) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2970, 1721, 1672, 1592, 1451, 1277, 1082, 933, 723 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.02$ (d, 1H, $J = 7.3$ Hz), 7.60 (td, 1H, $J = 7.5$ and 1.2 Hz), 7.53 (td, 1H, $J = 7.5$ and 1.4 Hz), 7.37–7.34 (m, 2H), 7.18–7.15 (m, 1H), 6.76 (d, 1H, $J = 8.3$ Hz), 6.02 (s, 2H), 3.67 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.2$ (CH_3), 101.9 (CH_2), 107.8 (CH), 108.5 (CH), 126.4 (CH), 127.6 (CH), 128.9 (C_q), 129.4 (CH), 130.1 (CH), 132.1 (C_q), 132.3 (CH), 141.9 (C_q), 148.2 (C_q), 151.9 (C_q), 166.3 (C_q), 195.3 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{13}\text{O}_5]^+ = [\text{M} + \text{H}]^+$ 285.0757; found 285.0766.

Methyl-2-(furan-2-carbonyl) Benzoate (3am).^{19e} GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2m** (153.7 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 92:08–88:12) furnished the product **3am** (69.5 mg, 75%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), $R_f(1a) = 0.90$, $R_f(3am) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2929, 1721, 1689, 1598, 1450, 1277, 1084, 938, 719 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.00$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.64–7.57 (m, 3H), 7.49 (dd, 1H, $J = 7.3$ and 1.4 Hz), 6.98 (d, 1H, $J = 3.4$ Hz), 6.51 (dd, 1H, $J = 3.4$ and 1.4 Hz), 3.69 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.4$ (CH_3), 112.3 (CH), 119.0 (CH), 128.1 (CH), 129.7 (C_q), 130.0 (CH), 130.2 (CH), 132.2 (CH), 140.0 (C_q), 147.1 (CH), 152.7 (C_q), 166.6 (C_q), 184.2 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{13}\text{H}_{11}\text{O}_4]^+ = [\text{M} + \text{H}]^+$ 231.0652; found 231.0653.

Methyl-2-(4-hydroxybenzoyl) Benzoate (3an). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2n** (195.4 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 93:07–90:10) furnished the product **3an** (46.0 mg, 45%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), $R_f(1a) = 0.90$, $R_f(3an) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 3421, 2951, 1722, 1670, 1592, 1451, 1272, 1088, 933, 712 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.00$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.82 (br s, 1H), 7.63–7.59 (m, 3H), 7.53 (td, 1H, $J = 7.5$ and 1.2 Hz), 7.36 (dd, 1H, $J = 7.5$ and 1.2 Hz), 6.78–6.76 (m, 2H), 3.63 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.4$ (CH_3), 115.5 ($2 \times \text{CH}$), 127.7 (CH), 128.9 (C_q), 129.3 (C_q), 129.5 (CH), 130.1 (CH), 132.1 ($2 \times \text{CH}$), 132.5 (CH), 141.7 (C_q), 161.4 (C_q), 166.9 (C_q), 196.9 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{15}\text{O}_4]^+ = [\text{M} + \text{H}]^+$ 271.0965; found 271.0969.

Methyl-2-(4-propoxybenzoyl) Benzoate (3ao). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2o** (262.7 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3ao** (75.2 mg, 63%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(1a) = 0.90$, $R_f(3ao) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2932, 1730, 1690, 1593, 1451, 1278, 1088, 934, 719 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.02$ (dd, 1H, $J = 7.8$ and 0.9 Hz), 7.71–7.62 (m, 2H), 7.60 (td, 1H, $J = 7.4$ and 1.2 Hz), 7.53 (td, 1H, $J = 7.4$ and 1.2 Hz), 7.36 (dd, 1H, $J = 7.8$ and 0.9 Hz), 6.89–6.87 (m, 2H), 3.95 (t, 2H, $J = 6.6$ Hz), 3.63 (s, 3H), 1.85–1.76 (m, 2H), 1.02 (t, 3H, $J = 7.5$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 10.4$ (CH_3), 22.4 (CH_2), 52.4 (CH_3), 69.7 (CH_2), 114.1 ($2 \times \text{CH}$), 127.6 (CH), 129.0 (C_q), 129.3 (CH), 129.9 (C_q), 130.0 (CH), 131.6 ($2 \times \text{CH}$), 132.2 (CH), 142.0 (C_q), 163.1 (C_q), 166.4 (C_q), 195.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{18}\text{NaO}_4]^+ = [\text{M} + \text{Na}]^+$ 321.1097; found 321.1097.

Methyl-2-(4-(2-bromoethoxy)benzoyl) Benzoate (3ap). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **2p** (916.6 mg, 4.0 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg,

0.48 mmol), and TBHP (514.2 mg, 4.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3ap** (72.0 mg, 49%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1a}) = 0.90$, $R_f(\mathbf{3ap}) = 0.50$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2966, 1722, 1685, 1598, 1450, 1277, 1083, 933, 715 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz $\delta = 8.03$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.74–7.71 (m, 2H), 7.62 (td, 1H, $J = 7.3$ and 1.4 Hz), 7.54 (td, 1H, $J = 7.3$ and 1.4 Hz), 7.37 (dd, 1H, $J = 7.5$ and 1.2 Hz), 6.93–6.89 (m, 2H), 4.36 (t, 2H, $J = 6.3$ Hz) 3.64 (s, 3H), 3.64 (t, 2H, $J = 6.3$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 28.5$ (CH_2), 52.2 (CH_3), 67.8 (CH_2), 114.3 ($2 \times \text{CH}$), 127.6 (CH), 129.1 (C_q), 129.4 (CH), 130.1 (CH), 130.9 (C_q), 131.7 ($2 \times \text{CH}$), 132.3 (CH), 141.9 (C_q), 161.9 (C_q), 166.4 (C_q), 195.7 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{17}\text{H}_{16}\text{BrO}_4]^+ = [\text{M} + \text{H}]^+ 365.0208$; found 365.0206.

Methyl-4-benzoyl Benzoate (3fa)^{19f}. GP-1 was carried out with aryl iodide **1f** (104.8 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3fa** (59.6 mg, 62%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1f}) = 0.90$, $R_f(\mathbf{3fa}) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 3000, 1780, 1685, 1620, 1490, 1310, 1110, 988, 763 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.14$ (d, 2H, $J = 8.8$ Hz), 7.80 (dd, 4H, $J = 8.8$ and 7.5 Hz), 7.61 (t, 1H, $J = 7.5$ Hz), 7.49 (t, 2H, $J = 7.5$ Hz), 3.95 (s, 3H) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 52.4$ (CH_3), 128.4 ($2 \times \text{CH}$), 129.5 ($2 \times \text{CH}$), 129.7 ($2 \times \text{CH}$), 130.1 ($2 \times \text{CH}$), 132.9 (CH), 133.2 (C_q), 136.9 (C_q), 141.3 (C_q), 166.3 (C_q), 196.0 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{15}\text{H}_{13}\text{O}_3]^+ = [\text{M} + \text{H}]^+ 241.0859$; found 241.0869.

Ethyl-2-benzoyl Benzoate (3ba). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3ba** (76.3 mg, 75%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3ba}) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2919, 2850, 1715, 1670, 1596, 1446, 1366, 1276, 1130, 1040, 931, 714 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.05$ (d, 1H, $J = 7.3$), 7.74 (d, 2H, $J = 6.5$ Hz), 7.64–7.52 (m, 3H), 7.40 (dd, 3H, $J = 7.3$ and 6.5 Hz), 4.05 (q, 2H, $J = 7.3$ Hz), 1.04 (t, 2H, $J = 7.3$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.7$ (CH_3), 61.4 (CH_2), 127.6 (CH), 128.4 ($2 \times \text{CH}$), 129.3 ($2 \times \text{CH}$), 129.5 (CH), 130.1 (CH), 132.3 (CH), 133.0 (CH), 137.1 (C_q), 141.5 (C_q), 165.9 (C_q), 196.9 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{15}\text{O}_3]^+ = [\text{M} + \text{H}]^+ 255.1016$; found 255.1017.

Ethyl-2-(4-methylbenzoyl) Benzoate (3bb). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3bb** (75.3 mg, 70%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3bb}) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2921, 2853, 1719, 1670, 1605, 1446, 1366, 1277, 1130, 1050, 981, 714 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.04$ (d, 1H, $J = 7.3$ Hz), 7.6 (d, 2H, $J = 8.3$ Hz), 7.59 (dd, 1H, $J = 7.3$ and 1.4 Hz), 7.54 (ddd, 1H, $J = 8.3$, $J = 7.3$ and 1.4 Hz), 7.36 (d, 1H, $J = 7.8$ Hz), 7.20 (d, 2H, $J = 7.8$ Hz), 4.06 (q, 2H, $J = 7.1$ Hz), 2.38 (s, 3H), 1.05 (t, 2H, $J = 7.1$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 21.6 (CH_3), 61.4 (CH_2), 129.1 ($3 \times \text{CH}$), 129.3 (CH), 129.5 ($2 \times \text{CH}$), 130.1 (C_q), 132.2 (CH), 134.7 (C_q), 141.8 (C_q), 143.9 (C_q), 166.0 (C_q), 196.6 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{17}\text{H}_{17}\text{O}_3]^+ = [\text{M} + \text{H}]^+ 269.1172$; found 269.1172.

Ethyl-2-(2-methylbenzoyl) Benzoate (3bc). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2c** (192.2 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48

mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–94:06) furnished the product **3bc** (69.7 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3bc}) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2956, 1720, 1675, 1598, 1450, 1277, 1083, 933, 713 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 7.93$ (d, 1H, $J = 8.8$ Hz), 7.60–7.51 (m, 2H), 7.42–7.34 (m, 2H), 7.28 (d, 1H, $J = 7.8$ Hz), 7.23 (d, 1H, $J = 7.8$ Hz), 7.11 (t, 1H, $J = 7.8$ Hz), 4.08 (q, 2H, $J = 7.3$ Hz), 2.65 (s, 3H), 1.09 (t, 2H, $J = 7.3$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.7$ (CH_3), 21.6 (CH_3), 61.4 (CH_2), 125.3 (CH), 128.3 (CH), 129.8 (CH), 130.3 (C_q), 131.6 (CH), 131.8 (CH), 131.9 (CH), 136.5 (C_q), 140.0 (C_q), 142.5 (C_q), 166.5 (C_q), 198.6 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{17}\text{H}_{17}\text{O}_3]^+ = [\text{M} + \text{H}]^+ 269.1172$; found 269.1179.

Ethyl-2-(4-ethylbenzoyl) Benzoate (3bd). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2d** (214.7 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3bd** (76.8 mg, 68%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3bd}) = 0.30$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2921, 2853, 1719, 1671, 1607, 1453, 1363, 1277, 1130, 1050, 981, 814, 810, 713 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.07$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.69 (d, 2H, $J = 8.3$ Hz), 7.66–7.62 (m, 1H), 7.59–7.55 (m, 1H), 7.39 (dd, 1H, $J = 7.3$ and 0.9 Hz), 7.27 (d, 2H, $J = 1.4$ Hz), 4.09 (q, 2H, $J = 7.3$ Hz), 2.71 (q, 2H, $J = 7.8$ Hz), 1.25 (t, 2H, $J = 7.8$ Hz), 1.07 (t, 2H, $J = 7.8$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 15.1 (CH_3), 28.9 (CH_2), 61.4 (CH_2), 127.6 (CH), 128.0 ($2 \times \text{CH}$), 129.4 (CH), 129.4 (C_q), 129.6 ($2 \times \text{CH}$), 130.1 (CH), 132.2 (CH), 135.0 (C_q), 141.9 (C_q), 150.1 (C_q), 166.0 (C_q), 196.7 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{19}\text{O}_3]^+ = [\text{M} + \text{H}]^+ 283.1329$; found 283.1332.

Ethyl-2-(2-bromobenzoyl) Benzoate (3bf). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2f** (296.0 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3bf** (87.9 mg, 66%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3bf}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2959, 2921, 2851, 1720, 1671, 1603, 1462, 1279, 1129, 1084, 1015, 933, 847, 751, 714 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 7.87$ – 6.85 (m, 1H), 7.68–7.65 (m, 1H), 7.58–7.53 (m, 2H), 7.44–7.40 (m, 2H), 7.33–7.30 (m, 2H), 4.16 (q, 2H, $J = 7.0$ Hz), OCH_2CH_3 1.18 (t, 2H, $J = 7.0$ Hz, CH_3) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.9$ (CH_3), 61.6 (CH_2), 121.4 (C_q), 127.0 (CH), 129.2 (CH), 129.7 (CH), 130.9 (CH), 131.4 (CH), 131.5 (C_q), 131.8 (CH), 132.5 (CH), 134.5 (CH), 138.2 (C_q), 140.0 (C_q), 167.0 (C_q), 195.4 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{14}\text{BrO}_3]^+ = [\text{M} + \text{H}]^+ 333.0121$; found 333.0125.

Ethyl-2-(4-chlorobenzoyl) Benzoate (3bg). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2g** (224.9 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–96:04) furnished the product **3bg** (79.7 mg, 69%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1b}) = 0.90$, $R_f(\mathbf{3bg}) = 0.35$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2959, 2921, 2851, 1720, 1671, 1603, 1463, 1279, 1139, 1084, 1015, 935, 899, 857, 751, 710 \text{ cm}^{-1}$. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): $\delta = 8.06$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.70–7.67 (m, 2H), 7.65–7.54 (m, 2H), 7.41–7.35 (m, 3H), 4.10 (q, 2H, $J = 7.3$ Hz), 1.09 (t, 2H, $J = 7.3$ Hz) ppm. $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): $\delta = 13.7$ (CH_3), 61.6 (CH_2), 127.5 (CH), 128.8 ($2 \times \text{CH}$), 129.3 (C_q), 129.7 (CH), 130.2 (CH), 130.7 ($2 \times \text{CH}$), 132.4 (CH), 135.6 (C_q), 139.5 (C_q), 141.2 (C_q), 165.7 (C_q), 195.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{16}\text{H}_{14}\text{ClO}_3]^+ = [\text{M} + \text{H}]^+ 289.0626$; found 289.0633.

Ethyl-2-(benzo[d][1,3]dioxole-5-carbonyl) Benzoate (3bl). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **2l** (240.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–92:08) furnished the product **3bl** (93.0 mg, 78%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**1b**) = 0.90, R_f(**3bl**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2925, 2857, 1718, 1668, 1598, 1456, 1364, 1277, 1230, 1050, 983, 713 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.03 (d, 1H, J = 7.3 Hz), 7.62–7.50 (m, 2H), 7.36–7.32 (m, 2H), 7.16 (dd, 1H, J = 8.0 and 1.7 Hz), 6.75 (d, 1H, J = 7.8 Hz), 6.01 (s, 2H), 4.12 (q, 2H, J = 6.8 Hz), 1.11 (t, 2H, J = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.7 (CH₃), 61.4 (CH₂), 101.8 (CH₂), 107.8 (CH), 108.5 (CH), 126.5 (CH), 127.5 (CH), 129.1 (C_q), 129.3 (CH), 130.1 (CH), 132.1 (C_q), 132.2 (CH), 141.8 (C_q), 148.1 (C_q), 151.8 (C_q), 165.8 (C_q), 195.3 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₇H₁₅O₃]⁺ = [M + H]⁺ 299.0914; found 299.0919.

Isopropyl-2-benzoyl Benzoate (3ca). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3ca** (75.1 mg, 70%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3ca**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2980, 2924, 2853, 1716, 1673, 1594, 1451, 1347, 1279, 1081, 932, 709, 639 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.06 (dd, 1H, J = 7.8 and 0.9 Hz), 7.75 (dd, 2H, J = 8.3 and 1.4 Hz), 7.63–7.51 (m, 3H), 7.43–7.35 (m, 3H), 4.97 (sep, 1H, J = 6.3 Hz), 1.00 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.1 (2 × CH₃), 69.4 (CH), 128.4 (2 × CH), 129.4 (CH), 129.5 (2 × CH), 129.5 (C_q), 130.1 (CH), 132.2 (CH), 133.1 (CH), 137.2 (C_q), 141.5 (C_q), 165.3 (C_q), 196.9 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₇H₁₇O₃]⁺ = [M + H]⁺ 269.1172; found 269.1165.

Isopropyl-2-(4-methylbenzoyl) Benzoate (3cb). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3cb** (83.5 mg, 74%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3cb**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2981, 2851, 1715, 1669, 1604, 1351, 1311, 1278, 1106, 1079, 932, 714 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04 (dd, 1H, J = 7.8 and 0.9 Hz), 7.65 (d, 2H, J = 8.3 Hz), 7.58 (td, 1H, J = 7.3 and 1.4 Hz), 7.57–7.51 (m, 1H), 7.33 (dd, 1H, J = 7.5 and 1.2 Hz), 7.20 (d, 2H, J = 8.3 Hz), 4.97 (sep, 1H, J = 6.3 Hz), 2.38 (s, 3H), 1.01 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.2 (2 × CH₃), 21.6 (CH₃), 69.3 (CH), 127.5 (CH), 129.1 (2 × CH), 129.2 (CH), 129.6 (C_q), 129.6 (2 × CH), 130.1 (CH), 132.1 (CH), 134.8 (C_q), 141.7 (C_q), 143.9 (C_q), 165.3 (C_q), 196.5 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₈H₁₉O₃]⁺ = [M + H]⁺ 283.1329; found: 283.1330.

Isopropyl-2-(4-ethylbenzoyl) Benzoate (3cd). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2d** (214.7 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **3cd** (85.3 mg, 72%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3cd**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2971, 2931, 2874, 1715, 1671, 1605, 1415, 1351, 1278, 1106, 1079, 933, 714, 579 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04 (dd, 1H, J = 7.8 and 0.9 Hz), 7.67 (d, 2H, J = 8.3 Hz), 7.59 (td, 1H, J = 7.3 and 1.4 Hz), 7.55–7.51 (m, 1H), 7.34 (dd, 1H, J = 7.3 and 1.4 Hz), 7.22 (d, 2H, J = 8.3 Hz), 4.97 (sep, 1H, J = 6.3 Hz), 2.67 (q, 2H, J = 7.3 Hz), 7.67 (t, 3H, J = 7.3 Hz), 1.01 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 15.2 (CH₃), 21.1 (2 × CH₃), 28.9 (CH₂), 69.3 (CH), 127.5 (CH), 127.9 (2 × CH), 129.2

(CH), 129.6 (C_q), 129.7 (2 × CH), 130.1 (CH), 132.1 (CH), 135.0 (C_q), 141.7 (C_q), 150.1 (C_q), 165.4 (C_q), 196.6 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₉H₂₁O₃]⁺ = [M + H]⁺ 297.1485; found 297.1496.

Isopropyl-2-(2-bromobenzoyl) Benzoate (3cf). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2f** (296.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 98:02–97:03) furnished the product **3cf** (83.3 mg, 60%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3cf**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2981, 2927, 1715, 1675, 1584, 1373, 1272, 1143, 1104, 1068, 929, 842, 396, 709, 645, 515 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.85 (dd, 1H, J = 6.8 and 1.9 Hz), 7.66 (dd, 1H, J = 5.8 and 3.4 Hz), 7.55–7.53 (m, 2H), 7.46–7.44 (m, 1H), 7.40–7.38 (m, 1H), 7.31 (dd, 1H, J = 5.8 and 3.4 Hz), 5.07 (sep, 1H, J = 6.3 Hz), 1.16 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.4 (2 × CH₃), 69.4 (CH), 121.5 (C_q), 127.0 (CH), 129.0 (CH), 129.6 (CH), 130.7 (CH), 131.3 (CH), 132.0 (C_q), 132.0 (CH), 132.5 (CH), 134.5 (CH), 138.2 (C_q), 140.1 (C_q), 166.5 (C_q), 195.3 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₇H₁₅BrNaO₃]⁺ = [M + Na]⁺ 369.0097; found 369.0106.

Isopropyl-2-(4-bromobenzoyl) Benzoate (3ce). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2e** (396.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 98:02–97:03) furnished the product **3ce** (86.1 mg, 62%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3ce**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2980, 2925, 1712, 1674, 1584, 1467, 1373, 1272, 1133, 1104, 1068, 929, 842, 396, 709, 645, 515. ¹H NMR (CDCl₃, 400 MHz): δ = 8.06 (dd, 1H, J = 7.8 and 0.9 Hz), 7.64–7.60 (m, 3H), 7.58–7.54 (m, 3H), 7.33 (dd, 1H, J = 7.3 and 0.9 Hz), 4.99 (sep, 1H, J = 6.3 Hz), 1.06 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.3 (2 × CH₃), 69.6 (CH), 127.4 (CH), 128.3 (C_q), 129.5 (C_q), 129.6 (CH), 130.3 (CH), 130.9 (2 × CH), 131.8 (2 × CH), 132.4 (CH), 136.0 (C_q), 141.1 (C_q), 165.1 (C_q), 196.0 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₇H₁₆BrO₃]⁺ = [M + H]⁺ 347.0277; found 347.0268.

Isopropyl-2-(4-chlorobenzoyl) Benzoate (3cg). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2g** (224.9 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 98:02–97:03) furnished the product **3cg** (83.5 mg, 69%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.90, R_f(**3cg**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2980, 2922, 2851, 1715, 1674, 1586, 1465, 1373, 1276, 1106, 1089, 931, 845, 746, 711, 515 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.05 (dd, 1H, J = 7.8 and 0.9 Hz), 7.69 (d, 2H, J = 8.3 Hz), 7.63–7.53 (m, 2H), 7.39–7.32 (m, 3H), 4.99 (sep, 1H, J = 6.3 Hz), 1.05 (d, 6H, J = 6.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.3 (2 × CH₃), 69.5 (CH), 127.3 (CH), 128.7 (2 × CH), 129.4 (C_q), 129.6 (CH), 130.2 (CH), 130.7 (2 × CH), 132.3 (CH), 135.6 (C_q), 139.5 (C_q), 141.1 (C_q), 165.1 (C_q), 195.7 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₇H₁₆ClO₃]⁺ = [M + H]⁺ 303.0782; found 303.0789.

Isopropyl-2-(benzo[d][1,3]dioxole-5-carbonyl) Benzoate (3cl). GP-1 was carried out with aryl iodide **1c** (116.0 mg, 0.40 mmol), aldehyde **2l** (240.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 93:07–90:10) furnished the product **3cl** (93.7 mg, 75%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**1c**) = 0.95, R_f(**3cl**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2982, 2851, 1713, 1665, 1605, 1351, 1313, 1278, 1106, 1079, 932, 714 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.02 (d, 1H, J = 7.8 Hz), 7.59–7.48

(m, 2H), 7.35 (d, 1H, $J = 1.4$ Hz), 7.30 (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.16 (dd, 1H, $J = 8.3$ and 1.4 Hz), 6.74 (d, 1H, $J = 8.3$ Hz), 6.00 (s, 2H), 5.01 (sep, 1H, $J = 6.3$ Hz), 1.07 (d, 6H, $J = 6.3$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 21.3$ ($2 \times \text{CH}_3$), 69.3 (CH), 101.8 (CH_2), 107.8 (CH), 108.6 (CH), 126.5 (CH), 127.4 (CH), 129.2 (CH), 129.5 (C_q), 130.1 (CH), 132.0 (CH), 132.2 (C_q), 141.7 (C_q), 148.1 (C_q), 151.8 (C_q), 165.2 (C_q), 195.1 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{17}\text{O}_3]^+ = [\text{M} + \text{H}]^+$ 313.1071; found 313.1077.

Butyl-2-benzoyl Benzoate (3da). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–95:05) furnished the product **3da** (88.1 mg, 78%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3da}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2966$, 1721, 1675, 1590, 1450, 1257, 1083, 933, 714 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.05$ (dd, 1H, $J = 7.8$ and 0.9 Hz), 7.68 (d, 2H, $J = 8.8$ Hz), 7.62 (td, 1H, $J = 7.5$, and 1.4 Hz), 7.55 (td, 2H, $J = 8.3$ and 0.9 Hz), 7.62 (td, 1H, $J = 7.4$ and 1.2 Hz), 7.59–7.52 (m, 2H), 7.44–7.37 (m, 3H), 4.02 (t, 2H, $J = 6.6$ Hz, $\text{OCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.43–1.36 (m, 2H, $\text{OCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 1.24–1.15 (m, 2H), 0.80 (t, 3H, $J = 7.3$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 19.0 (CH_2), 30.2 (CH_2), 65.4 (CH_2), 127.6 (CH), 128.4 ($2 \times \text{CH}$), 129.4 (C_q), 129.4 ($2 \times \text{CH}$), 129.5 (CH), 130.2 (CH), 132.3 (CH), 133.1 (CH), 137.1 (C_q), 141.6 (C_q), 166.0 (C_q), 196.9 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{19}\text{O}_3]^+ = [\text{M} + \text{H}]^+$ 283.1329; found 283.1336.

Butyl-2-(4-methylbenzoyl) Benzoate (3db). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3db** (85.4 mg, 72%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3db}) = 0.35$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2959$, 1725, 1665, 1520, 1453, 1267, 1072, 936, 720 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.04$ (dd, 1H, $J = 7.8$ and 1.4 Hz), 7.64 (d, 2H, $J = 8.3$ Hz), 7.60 (td, 1H, $J = 7.8$, and 1.4 Hz), 7.53 (td, 1H, $J = 7.5$, and 1.2 Hz), 7.35 (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.20 (d, 2H, $J = 8.3$ Hz), 4.02 (t, 2H, $J = 6.6$ Hz), 2.38 (s, 3H), 1.44–1.37 (m, 2H), 1.24–1.15 (m, 2H), 0.80 (t, 3H, $J = 7.3$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 19.0 (CH_2), 21.6 (CH_3), 30.2 (CH_2), 65.3 (CH_2), 127.5 (CH), 129.1 ($2 \times \text{CH}$), 129.3 (C_q), 129.3 (CH), 129.5 ($2 \times \text{CH}$), 130.1 (CH), 132.2 (CH), 134.6 (C_q), 141.8 (C_q), 143.9 (C_q), 166.0 (C_q), 196.6 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{19}\text{H}_{21}\text{O}_3]^+ = [\text{M} + \text{H}]^+$ 297.1485; found 297.1485.

Butyl-2-(4-ethylbenzoyl) Benzoate (3dd). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2d** (214.7 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3dd** (93.1 mg, 75%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3dd}) = 0.35$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2952$, 1722, 1673, 1596, 1452, 1274, 1086, 938, 719 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.04$ (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.67 (d, 2H, $J = 8.3$ Hz), 7.63–7.52 (m, 2H), 7.35 (dd, 1H, $J = 7.5$ and 1.2 Hz), 7.23 (d, 2H, $J = 8.3$ Hz), 4.02 (t, 2H, $J = 6.6$ Hz), 2.68 (q, 2H, $J = 7.8$ Hz), 1.43–1.36 (m, 2H), 1.25–1.16 (m, 5H), 0.79 (t, 3H, $J = 7.3$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 15.1 (CH_3), 19.0 (CH_2), 28.9 (CH_2), 30.2 (CH_2), 65.4 (CH_2), 127.5 (CH), 128.0 ($2 \times \text{CH}$), 129.3 (C_q), 129.3 (CH), 129.7 ($2 \times \text{CH}$), 130.1 (CH), 132.2 (CH), 134.8 (C_q), 141.8 (C_q), 150.1 (C_q), 166.1 (C_q), 196.7 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{20}\text{H}_{23}\text{O}_3]^+ = [\text{M} + \text{H}]^+$ 311.1642; found 311.1649.

Butyl-2-(4-chlorobenzoyl) Benzoate (3dg). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2g** (224.9 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude

material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3dg** (78.6 mg, 62%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3dg}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2966$, 1721, 1675, 1590, 1450, 1257, 1083, 933, 714 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.05$ (dd, 1H, $J = 7.8$ and 0.9 Hz), 7.68 (d, 2H, $J = 8.8$ Hz), 7.62 (td, 1H, $J = 7.5$, and 1.4 Hz), 7.55 (td, 2H, $J = 8.3$ and 0.9 Hz), 7.39–7.34 (m, 3H), 4.04 (t, 2H, $J = 6.6$ Hz), 1.47–1.40 (m, 2H), 1.27–1.17 (m, 2H), 0.82 (t, 3H, $J = 7.3$ Hz), 1 ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 19.0 (CH_2), 30.2 (CH_2), 65.5 (CH_2), 127.4 (CH), 128.8 ($2 \times \text{CH}$), 129.2 (C_q), 129.6 (CH), 130.2 (CH), 130.7 ($2 \times \text{CH}$), 132.4 (CH), 135.5 (C_q), 139.5 (C_q), 141.1 (C_q), 165.8 (C_q), 195.8 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{18}\text{ClO}_3]^+ = [\text{M} + \text{H}]^+$ 317.0939; found 317.0946.

Butyl-2-(2-bromobenzoyl) Benzoate (3df). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2f** (296.0 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3df** (91.0 mg, 63%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3df}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2960$, 1722, 1674, 1599, 1455, 1280, 1088, 939, 720 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 7.87$ –7.85 (m, 1H), 7.68–7.65 (m, 1H), 7.59–7.51 (m, 2H), 7.44–7.41 (m, 2H), 7.34–7.29 (m, 1H), 4.12 (t, 2H, $J = 6.6$ Hz), 1.58–1.51 (m, 2H), 1.33–1.24 (m, 2H), 0.84 (t, 3H, $J = 7.3$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 19.0 (CH_2), 30.3 (CH_2), 65.5 (CH_2), 121.5 (C_q), 127.0 (CH), 129.1 (CH), 129.7 (CH), 130.8 (CH), 131.4 (CH), 131.6 (C_q), 131.9 (CH), 132.5 (CH), 134.5 (CH), 138.2 (C_q), 140.1 (C_q), 167.1 (C_q), 195.4 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{18}\text{BrO}_3]^+ = [\text{M} + \text{H}]^+$ 361.0434; found 361.0436.

Butyl-2-(4-bromobenzoyl) Benzoate (3de). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **2e** (296.0 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3de** (98.3 mg, 68%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1d}) = 0.90$, $R_f(\mathbf{3de}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2959$, 1720, 1675, 1598, 1450, 1277, 1083, 933, 713 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.05$ (dd, 1H, $J = 7.8$ and 0.9 Hz), 7.65–7.53 (m, 6H), 7.34 (dd, 1H, $J = 7.5$, and 1.2 Hz), 4.04 (t, 2H, $J = 6.6$ Hz), 1.47–1.40 (m, 2H), 1.27–1.18 (m, 2H), 0.82 (t, 3H, $J = 7.3$ Hz), 1 ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.6$ (CH_3), 19.0 (CH_2), 30.2 (CH_2), 65.5 (CH_2), 127.4 (CH), 128.3 (C_q), 129.2 (C_q), 129.7 (CH), 130.2 (CH), 130.8 ($2 \times \text{CH}$), 131.8 ($2 \times \text{CH}$), 132.4 (CH), 135.9 (C_q), 141.1 (C_q), 165.8 (C_q), 196.0 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{18}\text{H}_{18}\text{BrO}_3]^+ = [\text{M} + \text{H}]^+$ 361.0434; found 361.0434.

Pentyl-2-benzoyl Benzoate (3ea). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), $\text{Pd}(\text{OAc})_2$ (5.0 mg, 5 mol %), Ag_2O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3ea** (98.4 mg, 83%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), $R_f(\mathbf{1e}) = 0.90$, $R_f(\mathbf{3ea}) = 0.40$, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): $\nu_{\text{max}} = 2960$, 1722, 1677, 1599, 1456, 1288, 1088, 944, 720 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): $\delta = 8.05$ (dd, 1H, $J = 7.3$ and 1.4 Hz), 7.75 (dd, 2H, $J = 8.5$ and 1.2 Hz), 7.64–7.51 (m, 3H), 7.44–7.36 (m, 3H), 4.00 (t, 2H, $J = 6.6$ Hz), 1.45–1.37 (m, 2H), 1.24–1.11 (m, 4H), 0.81 (t, 3H, $J = 7.0$ Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): $\delta = 13.8$ (CH_3), 22.2 (CH_2), 27.8 (CH_2), 27.9 (CH_2), 65.7 (CH_2), 127.5 (CH), 128.4 ($2 \times \text{CH}$), 129.4 ($2 \times \text{CH}$), 129.5 (CH), 129.6 (C_q), 130.1 (CH), 132.2 (CH), 133.0 (CH), 137.0 (C_q), 141.5 (C_q), 166.0 (C_q), 196.9 (C_q) ppm. HR-MS (ESI^+) m/z calcd for $[\text{C}_{19}\text{H}_{21}\text{O}_3]^+ = [\text{M} + \text{H}]^+$ 297.1485; found 297.1488.

Pentyl-2-(2-methylbenzoyl) Benzoate (3ec). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2c** (192.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3ec** (89.4 mg, 72%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3ec**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2959, 1722, 1685, 1568, 1462, 1255, 1088, 944, 730 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.93 (dd, 1H, J = 7.3 and 1.4 Hz), 7.59–7.51 (m, 2H), 7.41–7.35 (m, 2H), 7.28 (d, 1H, J = 7.3 Hz), 7.25–7.23 (m, 1H), 7.14–7.10 (m, 1H), 4.04 (t, 2H, J = 6.6 Hz), 2.66 (s, 3H), 1.51–1.44 (m, 2H), 1.24–1.15 (m, 4H), 0.81 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.8 (CH₃), 21.6 (CH₃), 22.2 (CH₂), 27.9 (CH₂), 28.0 (CH₂), 65.6 (CH₂), 125.3 (CH), 128.2 (CH), 129.7 (CH), 129.8 (CH), 130.3 (C_q), 131.7 (2 × CH), 131.8 (CH), 131.9 (CH), 136.4 (C_q), 140.1 (C_q), 142.6 (C_q), 166.6 (C_q), 198.5 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₂₀H₂₃O₃]⁺ = [M + H]⁺ 311.1642; found 311.1644.

Pentyl-2-(4-methylbenzoyl) Benzoate (3eb). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3eb** (98.1 mg, 79%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3eb**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2967, 1730, 1680, 1602, 1460, 1279, 1090, 940, 719 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.93 (dd, 1H, J = 7.3 and 1.4 Hz), 7.59–7.51 (m, 2H), 7.41–7.35 (m, 2H), 7.28 (d, 1H, J = 7.3 Hz), 7.25–7.23 (m, 1H), 7.14–7.10 (m, 1H), 4.03 (t, 2H, J = 6.6 Hz), 2.66 (s, 3H), 1.51–1.44 (m, 2H), 1.24–1.15 (m, 4H), 0.81 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.8 (CH₃), 21.6 (CH₃), 22.2 (CH₂), 27.9 (CH₂), 27.9 (CH₂), 65.7 (CH₂), 127.5 (CH), 129.1 (2 × CH), 129.3 (CH), 129.3 (C_q), 129.6 (2 × CH), 130.1 (CH), 132.1 (CH), 134.7 (C_q), 141.8 (C_q), 143.9 (C_q), 166.0 (C_q), 196.6 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₂₀H₂₃O₃]⁺ = [M + H]⁺ 311.1642; found 311.1645.

Pentyl-2-(2-bromobenzoyl) Benzoate (3ef). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2f** (296.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3ef** (86.1 mg, 60%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3ef**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2966, 1730, 1685, 1588, 1460, 1287, 1093, 943, 723 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.88–7.86 (m, 1H), 7.69–7.65 (m, 1H), 7.59–7.52 (m, 2H), 7.45–7.41 (m, 2H), 7.34–7.30 (m, 2H), 4.11 (t, 2H, J = 6.6 Hz), 1.59–1.54 (m, 2H), 1.25–1.20 (m, 4H), 0.82 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.9 (CH₃), 22.2 (CH₂), 28.0 (2 × CH₂), 65.9 (CH₂), 121.5 (C_q), 127.0 (CH), 129.1 (CH), 129.8 (CH), 130.8 (CH), 131.4 (CH), 131.6 (C_q), 132.0 (CH), 132.5 (CH), 134.5 (CH), 138.1 (C_q), 140.1 (C_q), 167.1 (C_q), 195.4 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₉H₂₀BrO₃]⁺ = [M + H]⁺ 375.0590; found 375.0592.

Pentyl-2-(4-bromobenzoyl) Benzoate (3ee). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2e** (296.0 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3ee** (124.6 mg, 83%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3ee**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2957, 1699, 1680, 1600, 1460, 1282, 1085, 940, 720 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.06 (dd, 1H, J = 7.8 and 0.9 Hz), 7.65–7.54 (m, 6H), 7.34 (dd, 1H, J = 7.8 and 0.9 Hz), 4.04 (t, 2H, J = 6.6 Hz), 1.49–1.42 (m, 2H), 1.26–1.14 (m, 4H), 0.83 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.9 (CH₃), 22.2 (CH₂), 27.9 (2 × CH₂), 65.8 (CH₂), 127.4 (CH), 128.3

(C_q), 129.3 (C_q), 129.7 (CH), 130.3 (CH), 130.8 (2 × CH), 131.8 (2 × CH), 132.4 (CH), 135.9 (C_q), 141.1 (C_q), 165.8 (C_q), 195.9 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₉H₂₀BrO₃]⁺ = [M + H]⁺ 375.0590; found 375.0600.

Pentyl-2-(4-chlorobenzoyl) Benzoate (3eg). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2g** (224.9 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3eg** (117.8 mg, 89%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3eg**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2950, 1721, 1676, 1599, 1451, 1278, 1084, 934, 714 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.08–8.06 (m, 1H), 7.68 (d, 2H, J = 8.8 Hz), 7.62 (td, 1H, J = 7.5 and 1.4 Hz), 7.56 (td, 1H, J = 7.5 and 1.4 Hz), 7.41–7.34 (m, 3H), 4.04 (t, 2H, J = 6.6 Hz), 1.49–1.42 (m, 2H), 1.26–1.14 (m, 4H), 0.83 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.8 (CH₃), 22.2 (CH₂), 27.9 (2 × CH₂), 65.8 (CH₂), 127.4 (CH), 128.8 (2 × CH), 129.3 (C_q), 129.7 (CH), 130.3 (CH), 130.7 (2 × CH), 132.4 (CH), 135.5 (C_q), 139.5 (C_q), 141.2 (C_q), 165.8 (C_q), 195.7 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₉H₂₀ClO₃]⁺ = [M + H]⁺ 331.1095; found 331.1091.

Pentyl-2-(4-fluorobenzoyl) Benzoate (3eh). GP-1 was carried out with aryl iodide **1e** (127.3 mg, 0.40 mmol), aldehyde **2h** (198.6 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **3eh** (86.8 mg, 69%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1e**) = 0.90, R_f(**3eh**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2959, 1713, 1678, 1599, 1452, 1288, 1086, 934, 716 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.06 (d, 1H, J = 7.8 Hz), 7.77 (dd, 2H, J = 8.8 and 5.3 Hz), 7.64–7.54 (m, 2H), 7.35 (dd, 1H, J = 8.8 and 5.3 Hz), 7.05 (t, 2H, J = 8.5 Hz), 4.04 (t, 2H, J = 6.8 Hz), 1.49–1.42 (m, 2H), 1.26–1.12 (m, 4H), 0.83 (t, 3H, J = 7.0 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.8 (CH₃), 22.2 (CH₂), 27.9 (2 × CH₂), 65.8 (CH₂), 115.5 (CH), 115.7 (CH), 127.4 (CH), 129.3 (C_q), 129.6 (CH), 130.3 (CH), 132.0 (CH), 132.0 (CH), 132.3 (CH), 133.7 (C_q), 141.4 (C_q), 165.9 (C_q), 166.9 (d, ¹J_{C–F} = 249.4 Hz), 195.4 (C_q) ppm of HR-MS (ESI⁺) *m/z* calcd for [C₁₉H₂₀FO₃]⁺ = [M + H]⁺ 315.1391; found 315.1399.

Methyl-2-isobutryl Benzoate (5aa). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **4a** (115.4 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **5aa** (53.6 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.90, R_f(**5aa**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2933, 1720, 1625, 1488, 1480, 1395, 1313, 1098, 1018, 818, 739 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.91 (dd, 1H, J = 7.8 and 0.9 Hz), 7.54 (td, 1H, J = 7.4 and 1.2 Hz), 7.47 (td, 1H, J = 7.8 and 1.2 Hz), 7.30 (dd, 1H, J = 7.8, and 0.9 Hz), 3.86 (s, 3H), 3.04 (sep, 1H, J = 6.8 Hz), 1.18 (d, H, J = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 18.5 (2 × CH₃), 40.7 (CH), 52.5 (CH₃), 126.8 (CH), 128.3 (C_q), 129.4 (CH), 130.0 (CH), 132.1 (CH), 143.1 (C_q), 166.9 (C_q), 210.1 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₂H₁₅O₃]⁺ = [M + H]⁺ 207.1016; found 207.1018.

Methyl-4-isobutryl Benzoate (5fa).^{20ch} GP-1 was carried out with aryl iodide **1f** (104.8 mg, 0.40 mmol), aldehyde **4a** (115.4 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **5fa** (48.7 mg, 59%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1f**) = 0.90, R_f(**5fa**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2924, 1606, 1488, 1441, 1385, 1303, 1088, 1014, 814, 738 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.11 (d, 2H, J = 7.8 Hz), 7.98 (d, 2H, J = 8.8 Hz), 3.94 (s, 3H), 3.54 (sep, 1H, J = 6.8 Hz), 1.22 (d, H, J = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 19.0

(2 × CH₃), 35.8 (CH), 52.4 (CH₃), 128.2 (2 × CH), 129.8 (2 × CH), 133.6 (C_q), 139.6 (C_q), 166.3 (C_q), 204.0 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₂H₁₅O₃]⁺ = [M + H]⁺ 207.1016; found 207.1013.

Methyl-2-(3-methylbutanoyl) Benzoate (5ab). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **4b** (137.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **5ab** (62.5 mg, 71%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1a**) = 0.80, R_f(**5ab**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2943, 1723, 1683, 1481, 1441, 1386, 1304, 1089, 1018, 815, 739 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.84 (dd, 1H, *J* = 7.8 and 0.9 Hz), 7.53 (td, 1H, *J* = 7.5 and 1.4 Hz, Ar–H), 7.47 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.36 (dd, 1H, *J* = 7.8, and 0.9 Hz, Ar–H), 3.87 (s, 3H, OCH₃), 2.70 (d, 2H, *J* = 6.8 Hz), 2.25 (sep, 1H, *J* = 6.8 Hz), 0.99 (d, 6H, *J* = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 22.6 (2 × CH₃), 24.5 (CH), 51.2 (CH₂), 52.5 (CH₃), 126.5 (CH), 128.9 (C_q), 129.8 (2 × CH), 131.9 (CH), 143.0 (C_q), 167.4 (C_q), 204.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₃H₁₇O₃]⁺ = [M + H]⁺ 221.1172; found 221.1177.

Methyl-2-(cyclohexanecarbonyl) Benzoate (5ae). GP-1 was carried out with aryl iodide **1a** (104.8 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–92:08) furnished the product **5ae** (68.9 mg, 70%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:5), R_f(**1a**) = 0.80, R_f(**5ae**) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2963, 1703, 1695, 1487, 1440, 1385, 1303, 1098, 1014, 818, 737 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.90 (dd, 1H, *J* = 7.8 and 0.9 Hz), 7.53 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.45 (td, 1H, *J* = 7.5 and 0.9 Hz), 7.28 (dd, 1H, *J* = 7.5, and 1.4 Hz), 3.86 (s, 3H), 2.78 (tt, 1H, *J* = 11.7 and 3.4 Hz), 1.91 (d, 2H, *J* = 13.2 Hz), 1.81–1.65 (m, 4H), 1.46–1.37 (m, 2H), 1.27–1.21 (m, 2H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 25.8 (2 × CH₂), 25.9 (CH₂), 28.7 (2 × CH₂), 50.5 (CH), 52.5 (CH₃), 126.7 (CH), 128.3 (C_q), 129.4 (CH), 130.0 (CH), 132.0 (CH), 143.1 (C_q), 167.0 (C_q), 209.3 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₁₉O₃]⁺ = [M + H]⁺ 247.1329; found 247.1336.

Ethyl-2-pentanoyl Benzoate (5bd). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **4d** (137.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 96:04–92:08) furnished the product **5bd** (60.9 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1b**) = 0.85, R_f(**5bd**) = 0.25, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2921, 2853, 1729, 1678, 1615, 1546, 1466, 1301, 1130, 1050, 981, 951, 733 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.88 (dd, 1H, *J* = 7.8 and 1.5 Hz), 7.53 (ddd, 1H, *J* = 7.8, 7.3, and 1.5 Hz), 7.46 (ddd, 1H, *J* = 7.8, 7.3, and 1.5 Hz), 7.33 (dd, 1H, *J* = 7.8 and 1.5 Hz), 4.33 (q, 2H, *J* = 7.3 Hz), 2.79 (t, 2H, *J* = 7.3 Hz), 1.80–1.62 (m, 2H), 1.45–1.30 (m, 2H), 1.34 (t, 3H, *J* = 7.3 Hz), 0.92 (t, 3H, *J* = 7.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.9 (CH₃), 14.0 (CH₃), 22.3 (CH₂), 26.1 (CH₂), 42.6 (CH₂), 61.6 (CH₂), 126.2 (CH), 128.8 (C_q), 129.6 (CH), 129.8 (CH), 131.9 (CH), 143.3 (C_q), 166.7 (C_q), 205.9 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₄H₁₉O₃]⁺ = [M + H]⁺ 235.1329; found 235.1331.

Ethyl-2-(cyclohexanecarbonyl) Benzoate (5be). GP-1 was carried out with aryl iodide **1b** (110.4 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **5be** (71.8 mg, 69%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1b**) = 0.80, R_f(**5be**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2923, 1720, 1685, 1487, 1440, 1385, 1300, 1087, 1014, 894, 739 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.93 (dd, 1H, *J* = 7.8 and 1.0 Hz), 7.54 (ddd, 1H, *J* = 7.8, 7.3, and 1.0 Hz),

7.47 (ddd, 1H, *J* = 7.3, 7.3, and 1.0 Hz), 7.30 (dd, 1H, *J* = 7.3 and 1.0 Hz), 4.35 (q, 2H, *J* = 7.3 Hz), 2.78 (tt, 1H, *J* = 11.7 and 3.4 Hz), 2.00–1.90 (m, 2H), 1.86–1.75 (m, 2H), 1.55–1.40 (m, 2H), 1.36 (t, 3H, *J* = 7.3 Hz), 1.34–1.12 (m, 4H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 14.1 (CH₃), 25.7 (2 × CH₂), 25.8 (CH₂), 28.7 (2 × CH₂), 50.3 (CH), 61.5 (CH₂), 126.7 (CH), 128.7 (C_q), 129.3 (CH), 129.9 (CH), 131.8 (CH), 142.9 (C_q), 166.5 (C_q), 209.2 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₆H₂₁O₃]⁺ = [M + H]⁺ 261.1485; found 261.1486.

Butyl-2-(cyclohexanecarbonyl) Benzoate (5de). GP-1 was carried out with aryl iodide **1c** (121.6 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–94:06) furnished the product **5de** (83.1 mg, 72%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1c**) = 0.85, R_f(**5de**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2953, 1720, 1686, 1487, 1440, 1385, 1303, 1088, 1014, 814, 738 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.91 (dd, 1H, *J* = 7.3 and 0.9 Hz), 7.53 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.45 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.27 (dd, 1H, *J* = 7.3 and 0.9 Hz), 4.28 (t, 2H, *J* = 6.8 Hz), 2.76 (tt, 1H, *J* = 11.6 and 3.3 Hz), 1.93 (d, 2H, *J* = 11.6 Hz), 1.74–1.65 (m, 4H), 1.49–1.38 (m, 4H), 1.29–1.20 (m, 4H), 0.95 (t, 3H, *J* = 7.3 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.7 (CH₃), 19.2 (CH₂), 25.8 (2 × CH₂), 25.9 (CH₂), 28.7 (2 × CH₂), 30.6 (CH₂), 50.4 (CH), 61.5 (CH₂), 126.7 (CH), 128.8 (C_q), 129.3 (CH), 130.0 (CH), 131.9 (CH), 143.1 (C_q), 166.6 (C_q), 209.3 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₈H₂₅O₃]⁺ = [M + H]⁺ 289.1798; found 289.1796.

Butyl-2-butyryl Benzoate (5dc). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **4c** (115.4 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **5dc** (61.6 mg, 62%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1d**) = 0.80, R_f(**5dc**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2963, 1780, 1680, 1482, 1441, 1386, 1304, 1058, 1054, 824, 748 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.87 (dd, 1H, *J* = 7.8 and 0.9 Hz), 7.53 (td, 1H, *J* = 7.4 and 1.2 Hz), 7.46 (td, 1H, *J* = 7.4 and 1.2 Hz), 7.32 (dd, 1H, *J* = 7.8, and 0.9 Hz), 4.27 (t, 2H), 2.75 (t, 2H, *J* = 7.8 Hz), 1.78–1.66 (m, 4H), 1.46–1.37 (m, 2H), 1.00–0.92 (m, 6H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.7 (2 × CH₃), 17.5 (CH₂), 19.1 (CH₂), 30.5 (CH₂), 44.8 (CH₂), 65.5 (CH₂), 126.2 (CH), 128.8 (C_q), 129.5 (CH), 129.8 (CH), 131.9 (CH), 143.3 (C_q), 166.8 (C_q), 205.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₅H₂₁O₃]⁺ = [M + H]⁺ 249.1485; found 249.1485.

Butyl-2-(3-methylbutanoyl) Benzoate (5db). GP-1 was carried out with aryl iodide **1d** (121.6 mg, 0.40 mmol), aldehyde **4b** (137.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **5db** (77.6 mg, 74%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:5), R_f(**1d**) = 0.80, R_f(**5db**) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2935, 1723, 1686, 1482, 1450, 1360, 1304, 1089, 1015, 811, 732 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.83 (dd, 1H, *J* = 7.8 and 0.9 Hz), 7.52 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.45 (td, 1H, *J* = 7.5 and 1.4 Hz), 7.35 (dd, 1H, *J* = 7.8, and 0.9 Hz), 4.28 (t, 2H), 2.70 (d, 2H, *J* = 6.8 Hz), 2.24 (sep, 1H, *J* = 6.8 Hz), 1.73–1.66 (m, 2H), 1.46–1.37 (m, 2H), 0.98 (d, 6H, *J* = 6.8 Hz), 0.94 (t, 3H, *J* = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.7 (CH₃), 19.1 (CH₂), 22.7 (2 × CH₃), 24.6 (CH), 30.5 (CH₂), 51.3 (CH₂), 65.5 (CH₂), 126.5 (CH), 129.3 (C_q), 129.7 (CH), 129.7 (CH), 131.7 (CH), 143.0 (C_q), 167.0 (C_q), 204.9 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₆H₂₃O₃]⁺ = [M + H]⁺ 263.1642; found 263.1640.

Benzophenone (7aa).^{20a} GP-1 was carried out with aryl iodide **6a** (81.6 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05)

furnished the product **7aa** (35.5 mg, 52%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**6a**) = 0.90, R_f (**7aa**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2960, 1733, 1678, 1588, 1460, 1287, 1082, 934, 719 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.81–7.79 (m, 4H), 7.60–7.56 (m, 2H), 7.50–7.46 (m, 4H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 128.3 (3 \times CH), 130.0 (3 \times CH), 132.4 (2 \times CH), 137.6 (2C_q), 196.7 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₃H₁₁O]⁺ = [M + H]⁺ 183.0804; found 183.0808.

(4-Methoxyphenyl)(phenyl)methanone (7aj). GP-1 was carried out with aryl iodide **6a** (81.6 mg, 0.40 mmol), aldehyde **2j** (217.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **7aj** (55.2 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**6a**) = 0.90, R_f (**7aj**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2960, 1730, 1672, 1588, 1452, 1278, 1081, 932, 716 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.82 (d, 2H, J = 8.8 Hz), 7.74 (dd, 2H, J = 7.5 and 1.2 Hz), 7.58–7.53 (m, 1H), 7.46 (t, 2H, J = 7.5 Hz), 6.95 (d, 2H, J = 8.8 Hz), 3.88 (s, 3H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 55.5 (CH₃), 113.5 (2 \times CH), 128.2 (2 \times CH), 129.7 (2 \times CH), 130.1 (C_q), 131.9 (CH), 132.5 (2 \times CH), 138.3 (C_q), 163.2 (C_q), 195.6 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₄H₁₃O₂]⁺ = [M + H]⁺ 213.0910; found 213.0912.

Furan-2-yl(phenyl)methanone (7am).^{20a} GP-1 was carried out with aryl iodide **6a** (81.6 mg, 0.40 mmol), aldehyde **2m** (153.7 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10–88:12) furnished the product **7am** (42.7 mg, 62%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 85:15), R_f (**6a**) = 0.99, R_f (**7am**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2966, 1723, 1676, 1599, 1451, 1278, 1084, 934, 714 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.95 (dd, 2H, J = 8.3 and 1.4 Hz), 7.70 (d, 1H, J = 2.4 Hz), 7.60–7.56 (m, 1H), 7.50–7.46 (m, 2H), 7.22 (d, 1H, J = 2.4 Hz), 6.58 (dd, 1H, J = 3.6 and 1.4 Hz) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 112.2 (CH), 120.5 (CH), 128.4 (2 \times CH), 129.3 (2 \times CH), 132.5 (CH), 137.2 (C_q), 147.1 (CH), 152.3 (C_q), 182.5 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₂₁H₁₄ClNNa]⁺ = [M + Na]⁺ 338.0707; found 338.0708.

Phenyl (m-Tolyl)methanone (7ba). GP-1 was carried out with aryl iodide **6b** (87.2 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **7ba** (43.4 mg, 54%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**6b**) = 0.80, R_f (**7ba**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2999, 1730, 1680, 1600, 1480, 1290, 1063, 944, 744 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.79 (dd, 2H, J = 8.3 and 1.4 Hz), 7.62–7.56 (m, 3H), 7.47 (t, 2H, J = 7.5 Hz), 7.41–7.33 (m, 2H), 2.41 (s, 3H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 21.3 (CH₃), 127.3 (CH), 128.1 (CH), 128.2 (2 \times CH), 130.0 (2 \times CH), 130.4 (CH), 132.3 (CH), 133.2 (CH), 137.6 (C_q), 137.7 (C_q), 138.1 (C_q), 196.9 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₄H₁₂O]⁺ = [M + Na]⁺ 219.0780; found 219.0776.

(4-Methoxyphenyl)(phenyl)methanone (7da).^{20a} GP-1 was carried out with aryl iodide **6d** (93.6 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **7da** (50.9 mg, 60%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**6d**) = 0.80, R_f (**7da**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2988, 1723, 1677, 1599, 1452, 1278, 1083, 935, 715 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.82 (d, 2H, J = 8.8 Hz), 7.74 (dd, 2H, J = 7.5 and 1.2 Hz), 7.58–7.53 (m, 1H), 7.46 (t, 2H, J = 7.5 Hz), 6.95 (d, 2H, J = 8.8 Hz), 3.88 (s, 3H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 55.5 (CH₃), 113.5 (2 \times CH), 128.2 (2 \times

CH), 129.7 (2 \times CH), 130.1 (C_q), 131.9 (CH), 132.5 (2 \times CH), 138.3 (C_q), 163.2 (C_q), 195.6 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₄H₁₃O₂]⁺ = [M + H]⁺ 213.0910; found 213.0912.

(4-Methoxyphenyl)(p-tolyl)methanone (7db). GP-1 was carried out with aryl iodide **6d** (93.6 mg, 0.40 mmol), aldehyde **2b** (192.2 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **7db** (57.9 mg, 64%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f (**6d**) = 0.80, R_f (**7db**) = 0.30, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2960, 1722, 1674, 1599, 1451, 1287, 1087, 934, 720 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.80 (d, 2H, J = 8.8 Hz), 7.67 (d, 2H, J = 7.8 Hz), 7.26 (d, 2H, J = 7.8 Hz), 6.95 (d, 2H, J = 8.8 Hz), 3.88 (s, 3H), 2.43 (s, 3H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 21.6 (CH₃), 55.5 (CH₃), 113.5 (2 \times CH), 128.9 (d, 2 \times CH), 130.0 (2 \times CH), 130.5 (C_q), 132.4 (2 \times CH), 135.5 (C_q), 142.6 (C_q), 163.0 (C_q), 195.4 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₅H₁₅O₂]⁺ = [M + H]⁺ 227.1067; found 227.1058.

(3,4-Dimethoxyphenyl)(phenyl)methanone (7fa). GP-1 was carried out with aryl iodide **6f** (105.6 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10–88:12) furnished the product **7fa** (64.9 mg, 67%) as a white solid (mp 81–83 °C). [TLC control (petroleum ether/ethyl acetate 85:15), R_f (**6f**) = 0.95, R_f (**7fa**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2954, 1730, 1685, 1599, 1451, 1278, 1084, 934, 719 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.73 (d, 2H, J = 6.8 Hz), 7.53 (t, 1H, J = 6.8 Hz), 7.47–7.43 (m, 3H), 7.35 (dd, 1H, J = 8.3 and 1.9 Hz), 6.86 (d, 1H, J = 8.3 Hz), 3.93 (s, 3H), 3.91 (s, 3H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 55.9 (CH₃), 56.0 (CH₃), 109.6 (CH), 112.0 (CH), 125.4 (CH), 128.1 (2 \times CH), 129.6 (2 \times CH), 130.1 (C_q), 131.8 (CH), 138.1 (C_q), 148.9 (C_q), 152.9 (C_q), 195.5 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₅H₁₅O₃]⁺ = [M + H]⁺ 243.1016; found 243.1018.

Benzo[d][1,3]dioxol-5-yl(phenyl)methanone (7ga). GP-1 was carried out with aryl iodide **6g** (99.2 mg, 0.40 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 93:07–90:10) furnished the product **7ga** (58.8 mg, 65%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f (**6g**) = 0.95, R_f (**7ga**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 2957, 1721, 1675, 1598, 1452, 1278, 1084, 934, 716 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.73–7.71 (m, 2H), 7.54 (t, 1H, J = 7.3 Hz), 7.44 (t, 2H, J = 7.3 Hz), 7.33–7.36 (m, 2H), 6.83 (d, 2H, J = 8.3 Hz), 6.03 (s, 2H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 101.8 (CH₂), 107.7 (CH), 109.8 (CH), 126.8 (CH), 128.1 (2 \times CH), 129.6 (2 \times CH), 131.8 (C_q), 131.9 (CH), 138.0 (C_q), 147.8 (C_q), 151.4 (C_q), 195.0 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₄H₁₁O₃]⁺ = [M + H]⁺ 227.0703; found 227.0702.

Cyclohexyl(phenyl)methanone (8ae).^{20a} GP-1 was carried out with aryl iodide **6a** (81.6 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **8ae** (36.9 mg, 49%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**6a**) = 0.90, R_f (**8ae**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max} = 3366, 2993, 1666, 1497, 1450, 1389, 1383, 1028, 1025, 830, 760 cm^{-1} . ^1H NMR (CDCl_3 , 400 MHz): δ = 7.94–7.92 (m, 2H), 7.55–7.51 (m, 1H), 7.45 (t, 2H, J = 7.5 Hz), 3.25 (t, 1H, J = 11.7 and 3.4 Hz), 1.90–1.41 (m, 4H), 1.72 (d, 1H, J = 11.2 Hz), 1.54–1.47 (m, 1H), 1.44–1.36 (m, 2H), 1.34–1.24 (m, 2H) ppm. ^{13}C NMR (CDCl_3 , 100 MHz): δ = 21.4 (CH₃), 25.8 (2 \times CH₂), 25.9 (CH₂), 29.4 (2 \times CH₂), 45.6 (CH), 128.2 (2 \times CH), 128.5 (2 \times CH), 132.7 (CH), 136.3 (C_q), 203.9 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₃H₁₇O]⁺ = [M + H]⁺ 189.1274; found 189.1275.

1-(3-Methoxyphenyl)-2-methylpropan-1-one (8ea).^{20c} GP-1 was carried out with aryl iodide **6e** (93.6 mg, 0.40 mmol), aldehyde **4a** (115.4 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **8ea** (32.1 mg, 45%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**6e**) = 0.90, R_f(**8ea**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2923, 1605, 1487, 1440, 1385, 1303, 1088, 1014, 814, 738 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.52 (d, 1H, J = 7.8 Hz), 7.48–7.47 (m, 1H), 7.35 (t, 1H, J = 8.0 Hz), 7.10–7.01 (m, 1H), 3.84 (s, 3H), 3.52 (sep, 1H, J = 6.8 Hz), 1.20 (d, 6H, J = 6.8 Hz) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 19.2 (2 × CH₃), 35.5 (CH), 55.4 (CH₃), 112.7 (CH), 119.1 (CH), 120.8 (CH), 129.5 (CH), 137.6 (C_q), 159.9 (C_q), 204.3 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₁H₁₅O₂]⁺ = [M + H]⁺ 179.1067; found 179.1064.

Cyclohexyl(m-tolyl)methanone (8be).^{20d} GP-1 was carried out with aryl iodide **6b** (87.2 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 97:03–95:05) furnished the product **8be** (44.5 mg, 55%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**6b**) = 0.90, R_f(**8be**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2924, 1615, 1488, 1441, 1390, 1305, 1078, 1024, 818, 739 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.74–7.72 (m, 2H, Ar–H), 7.34–7.32 (m, 2H), 3.24 (tt, 1H, J = 11.7 and 3.4 Hz), 2.40 (s, 3H), 1.89–1.81 (m, 4H), 1.75–1.71 (m, 1H), 1.53–1.21 (m, 5H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.4 (CH₃), 25.8 (2 × CH₂), 25.9 (CH₂), 29.4 (2 × CH₂), 45.4 (CH), 125.4 (CH), 128.4 (CH), 128.7 (CH), 133.5 (CH), 136.4 (C_q), 138.3 (C_q), 204.2 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₄H₁₉O]⁺ = [M + H]⁺ 203.1430; found 203.1428.

Cyclohexyl(3-methoxyphenyl)methanone (8ee).^{20e} GP-1 was carried out with aryl iodide **6e** (93.6 mg, 0.40 mmol), aldehyde **4e** (179.5 mg, 1.6 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05–93:07) furnished the product **8ee** (52.3 mg, 60%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**6e**) = 0.90, R_f(**8ee**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2933, 1689, 1482, 1443, 1388, 1301, 1080, 1013, 818, 737 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.74–7.72 (m, 2H), 7.34–7.32 (m, 2H), 3.24 (tt, 1H, J = 11.7 and 3.4 Hz), 2.40 (s, 3H), 1.89–1.81 (m, 4H), 1.75–1.71 (m, 1H), 1.53–1.21 (m, 5H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 21.4 (CH₃), 25.8 (2 × CH₂), 25.9 (CH₂), 29.4 (2 × CH₂), 45.4 (CH), 125.4 (CH), 128.4 (CH), 128.7 (CH), 133.5 (CH), 136.4 (C_q), 138.3 (C_q), 204.2 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₄H₁₉O]⁺ = [M + H]⁺ 203.1430; found 203.1428.

7ha^{20h}, **7ia**^{20f}, **7ja**^{20e}, **8hc**^{20g} reported in the literature.

3-Phenylisobenzofuran-1(3H)-one (9ba).^{20f} GP-2 was carried out with keto ester **3ba** (127.1 mg, 0.50 mmol), NaBH₄ (38 mg, 1.0 mmol), CeCl₃·7H₂O (186.3 mg, 0.50 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 85:15–80:20) furnished the product **9ba** (146.0 mg, 95%) as a white colored solid (m.p.: 107–109 °C). [TLC control (petroleum ether/ethyl acetate 80:20), R_f(**3ba**) = 0.90, R_f(**9ba**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2960, 1730, 1685, 1599, 1460, 1287, 1093, 943, 716 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.97 (d, 1H, J = 7.3 Hz), 7.65 (t, 1H, J = 7.5 Hz), 7.55 (t, 1H, J = 7.5 Hz), 7.39–7.36 (m, 3H), 7.34 (d, 1H, J = 6.8 Hz), 7.27 (dd, 2H, J = 6.8 and 2.9 Hz), 6.41 (s, 1H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 83.0 (CH), 123.1 (CH), 125.8 (C_q), 125.9 (CH), 127.3 (2 × CH), 129.3 (2 × CH), 129.6 (CH), 129.6 (CH), 134.6 (CH), 136.7 (C_q), 150.0 (C_q), 170.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₄H₁₁O₂]⁺ = [M + H]⁺ 211.0754; found 211.0760.

3-Butylisobenzofuran-1(3H)-one (9bd). GP-2 was carried out with keto ester **5bd** (117.1 mg, 0.50 mmol), NaBH₄ (38 mg, 1.0 mmol), CeCl₃·7H₂O (186.3 mg, 0.50 mmol). Purification of the crude material

by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10–85:15) furnished the product **9bd** (112.5 mg, 78%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**5bd**) = 0.90, R_f(**9bd**) = 0.35, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2970, 1740, 1685, 1602, 1460, 1287, 1093, 946, 752 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.87 (d, 1H, J = 7.8 Hz), 7.65 (td, 1H, J = 7.5 and 0.9 Hz), 7.50 (t, 1H, J = 7.5 Hz), 7.43–7.41 (m, 1H), 5.46 (dd, 1H, J = 7.8 and 3.9 Hz), 2.07–1.99 (m, 1H), 1.78–1.70 (m, 1H), 1.50–1.32 (m, 4H), 0.89 (t, 1H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 13.8 (CH₃), 22.4 (CH₂), 26.8 (CH₂), 34.4 (CH₂), 81.4 (CH), 121.7 (CH), 125.7 (CH), 126.1 (C_q), 129.0 (CH), 133.9 (CH), 150.1 (C_q), 170.7 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₂H₁₅O₂]⁺ = [M + H]⁺ 191.1067; found 191.1073.

3-Cyclohexylisobenzofuran-1(3H)-one (9be). GP-2 was carried out with keto ester **5be** (130.2 mg, 0.50 mmol), NaBH₄ (38 mg, 1.0 mmol), CeCl₃·7H₂O (186.3 mg, 0.50 mmol). Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 90:10–85:15) furnished the product **9be** (134.8 mg, 86%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**5be**) = 0.90, R_f(**9be**) = 0.35, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2957, 1735, 1695, 1588, 1440, 1227, 1083, 953, 723 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 7.85 (d, 1H, J = 7.3 Hz), 7.65–7.65 (m, 1H), 7.48 (t, 1H, J = 7.5 Hz), 7.42 (d, 1H, J = 8.3 Hz), 5.30 (d, 1H, J = 3.9 Hz), 1.93–1.75 (m, 3H), 1.68–1.62 (m, 2H), 1.04–1.31 (m, 6H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 25.7 (CH₂), 25.9 (CH₂), 25.9 (CH₂), 26.1 (CH₂), 29.1 (CH₂), 42.0 (CH), 85.3 (CH), 122.1 (CH), 125.5 (CH), 126.5 (C_q), 128.9 (CH), 133.7 (CH), 148.7 (C_q), 170.7 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₁₄H₁₇O₂]⁺ = [M + H]⁺ 217.1223; found 217.1223.

Pitofener (10ap).¹³ To an oven-dried Schlenk tube, were added keto ester **3ap** (30.0 mg, 0.08 mmol), piperidine (21.1 mg, 0.24 mmol) and K₂CO₃ (33.8 mg, 0.24 mmol) in DMF. The resulting reaction mixture was stirred 50 °C for 1 h. The reaction mixture was allowed to cool to room temperature, diluted with H₂O and then extracted with ethyl acetate (3 × 10 mL). The organic layers were washed with saturated NaCl solution, dried (Na₂SO₄) and filtered. Evaporation of the solvent under reduced pressure and purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 30:70–20:80) furnished the product **10ap** (25.0 mg, 82%) as a colorless viscous liquid. [TLC control (petroleum ether/ethyl acetate 00:100), R_f(**3ap**) = 0.99, R_f(**10ap**) = 0.20, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 2960, 2912, 1722, 1685, 1566, 1455, 1255, 1060, 944, 718 cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.02 (dd, 1H, J = 7.8 and 0.9 Hz), 7.69 (d, 2H, J = 9.2 Hz), 7.60 (td, 1H, J = 7.3 and 1.4 Hz), 7.53 (td, 1H, J = 7.3 and 1.4 Hz), 7.36 (dd, 1H, J = 7.8 and 0.9 Hz), 6.89 (d, 2H, J = 9.2 Hz), 4.13 (t, 2H, J = 6.1 Hz), 3.62 (s, 1H), 2.76 (t, 2H, J = 6.1 Hz), 2.49 (brs, 4H), 1.61–1.56 (m, 4H), 1.45–1.41 (m, 2H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 24.1 (CH₂), 25.9 (2 × CH₂), 52.2 (CH₃), 55.0 (2 × CH₂), 57.7 (CH₂), 66.2 (CH₂), 114.3 (2 × CH), 127.6 (CH), 129.0 (C_q), 129.3 (CH), 130.1 (CH), 130.2 (C_q), 131.6 (2 × CH), 132.3 (CH), 142.0 (C_q), 162.8 (C_q), 166.4 (C_q), 195.8 (C_q) ppm. HR-MS (ESI⁺) *m/z* calcd for [C₂₂H₂₆NO₄]⁺ = [M + H]⁺ 368.1856; found 368.1861.

2,2,6,6-Tetramethylpiperidin-1-yl benzoate (11a).^{14b} To an oven-dried Schlenk tube, were added **1a** (100 mg, 1.0 mmol), aldehyde **2a** (169.8 mg, 1.6 mmol), TEMPO (312.5 mg, 2.0 mmol), Pd(OAc)₂ (5.0 mg, 5 mol %), Ag₂O (111.3 mg, 0.48 mmol), and TBHP (257.1 mg, 2.0 mmol). The resulting reaction mixture was stirred at 120 °C for 12 h. Progress of the reaction was monitored by TLC until the reaction was completed. The reaction mixture was quenched by the addition of aqueous NaHCO₃ solution and then extracted with ethyl acetate (3 × 15 mL). The organic layers were washed with saturated NaCl solution, dried (Na₂SO₄) and filtered. Evaporation of the solvent under reduced pressure and purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 55:45–50:50) first furnished the simple deiodinated ester (8.3 mg, 15%) as colorless viscous liquid and then gave the product **11a** (397.1 mg, 95%) as white solid. [TLC control (petroleum ether/ethyl acetate 50:50), R_f(**2a**) = 0.99, R_f(**11a**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm⁻¹): ν_{max} = 3422, 2943, 1635, 1497, 1450, 1395, 1373, 1098, 1024, 824, 748

cm⁻¹. ¹H NMR (CDCl₃, 400 MHz): δ = 8.10 (d, 2H, J = 7.3 Hz), 7.59 (d, 1H, J = 7.0 Hz), 7.48 (t, 2H, J = 7.3 Hz), 1.84–1.59 (m, 5H), 1.44–1.50 (m, 1H), 1.30 (s, 3H), 1.14 (s, 3H) ppm. ¹³C NMR (CDCl₃, 100 MHz): δ = 16.3 (CH₂), 21.0 (2 × CH₃), 31.3 (2 × CH₃), 38.4 (2 × CH₂), 59.7 (2C_q), 127.8 (2 × CH), 128.9 (2 × CH), 129.0 (C_q), 132.2 (CH), 165.7 (C_q) ppm. HR-MS (ESI⁺) m/z calcd for [C₁₆H₂₃NNaO₂]⁺ = [M + Na]⁺ 284.1621; found 284.1624.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.6b01064.

¹H NMR and ¹³C NMR spectra of all compounds and CIF file for **3ah** (PDF)

(PDF)

(XLS)

■ AUTHOR INFORMATION

Corresponding Author

*Phone: (040) 2301 6033. Fax: (040) 2301 6003/32. E-mail: gvsatya@iith.ac.in

Notes

The authors declare no competing financial interest.

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