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Supporting Information

for

# Palladium-catalyzed decarbonylative and decarboxylative cross-coupling of acyl chlorides with potassium perfluorobenzoates affording unsymmetrical biaryls

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#### 1. General

#### Instrumentation

Unless otherwise noted, all the reactions were carried out under an argon or a nitrogen atmosphere in oil bath using standard Schlenk techniques. Solvents were employed as eluents for all other routine operation, as well as dehydrated solvent were purchased from commercial suppliers and employed without any further purification. Glassware was dried in an oven (130 °C) and heated under reduced pressure before use. For thin layer chromatography (TLC) analyses throughout this work, Merck precoated TLC plates (silica gel 60 GF<sub>254</sub>, 0.25 mm) were used. Silica gel column chromatography was carried out using Silica gel 60 N (spherical, neutral, 40–100  $\mu$ m) from Kanto Chemicals Co., Ltd. NMR spectra (<sup>1</sup>H, <sup>31</sup>P{<sup>1</sup>H}, and <sup>19</sup>F{<sup>1</sup>H}) were recorded on Mercury-400 (400 MHz) spectrometer. Chemical shifts ( $\delta$ ) are in parts per million relative to CDCl<sub>3</sub> at 7.26 ppm for <sup>1</sup>H NMR measurements. The <sup>19</sup>F{<sup>1</sup>H} NMR spectra were measured by using CCl<sub>3</sub>F ( $\delta$  = 0.00 ppm) as an external standard. The <sup>31</sup>P{<sup>1</sup>H} chemical shifts were reported in ppm relative to external reference of H<sub>3</sub>PO<sub>4</sub> at  $\delta$  0.00. The GC yields were determined by GC analysis of the crude mixture, using *n*-tetradecane as an internal standard. GC analyses were performed on a Shimadzu GC-14A equipped with a flame ionization detector using Shimadzu Capillary Column (CBP1-M25-025) and Shimadzu C-R6A-Chromatopac integrator.

#### Chemicals

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Acyl chlorides **1** were purchased from Tokyo Chemical Industry Co., Ltd. and Sigma-Aldrich Co. Toluene (super dehydrated) was purchased from FUJIFILM Wako Pure Chemical Corporation. Benzoates **2** were prepared according to the literatures<sup>1</sup> and showed the identical spectra as reported.

# 2. Optimization of Reaction Conditions

# Table S1. Optimization<sup>a</sup>

		F O [ OK _ F	at. (10 mol %) P] (20 mol %) additive toluene 150 °C, 20 h		O │ C <sub>6</sub> F₅
<b>1a</b> (1	.0 equiv) 2a (	(1.5 equiv)	3aa	4	
entry	cat.	ligand	additive	<b>3aa</b> <sup>b</sup> /%	<b>4</b> <sup>b</sup> /%
1	$Pd(OAc)_2$	$PCy_3 \cdot HBF_4$	-	72	5
2	$Pd_2(dba)_3$	$PCy_3{\cdot}HBF_4$	-	73	5
3	$[Pd(C_3H_5)Cl]_2$	$PCy_3{\cdot}HBF_4$	-	63	5
4	$Pd(cod)Cl_2$	$PCy_3{\cdot}HBF_4$	-	62	3
5	Pd(dba) <sub>2</sub>	PCy <sub>3</sub> ·HBF <sub>4</sub>	-	74	4
6	Ni(cod) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	-	6	36
7	$Pd(dba)_2$	PPh <sub>3</sub>	-	10	10
8	Pd(dba) <sub>2</sub>	DCYPE <sup>c</sup>	-	15	2
9	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	1,4-dioxane (0.1 mL)	71	5
10	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	diglyme (0.1 mL)	48	4
11	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	$nBu_3N$ (1.0 equiv)	66	3
12	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	DBA (1.0 equiv)	47	6
13	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	1,5-cyclooctadiene (1.0 equiv)	67	4
14	Pd(dba) <sub>2</sub>	$PCy_3{\cdot}HBF_4$	Zn (1.0 equiv)	64	1
15	Pd(dba) <sub>2</sub>	$PCy_3 \cdot HBF_4$	K <sub>3</sub> PO <sub>4</sub> (0.2 equiv)	61	2

<sup>*a*</sup>Reactions were carried out with [Pd] or [Ni] (0.02 mmol, 10 mol %), ligand (0.04 mmol, 20 mol %), additive, **1a** (0.2 mmol, 1.0 equiv), and **2a** (0.3 mmol, 1.5 equiv) in toluene (1 mL) at 150 °C for 20 h. <sup>*b*</sup>GC yields, using *n*-tetradecane as an internal standard. <sup>*c*</sup>10 mol%. Table S2. Control experiments<sup>a</sup>

	$CI + F O F O Fd(dba)_2 (10 mol \%)$ $PCy_3 HBF_4 (20 mol \%)$ $row f F F O F Toluene$ $150 °C, 20 h$	► C <sub>6</sub> F <sub>5</sub> +	O C <sub>6</sub> F <sub>5</sub>						
1a (1.0 equiv)       2a (1.5 equiv)       3aa       4									
entry	deviation from standard conditions	<b>3aa</b> <sup>b</sup> /%	<b>4</b> <sup>b</sup> /%						
1	C <sub>6</sub> F <sub>5</sub> COONa instead of C <sub>6</sub> F <sub>5</sub> COOK	26	3						
2	C <sub>6</sub> F <sub>5</sub> COOLi instead of C <sub>6</sub> F <sub>5</sub> COOK	1	0						
3	2-chloronaphthalene instead of 2-naphthoyl chloride	9	0						
4	170 °C in mesitylene	23	12						
5	140 °C	68	7						
6	Pd(dba) <sub>2</sub> (5 mol%), PCy <sub>3</sub> ·HBF <sub>4</sub> (10 mol%)	49	5						
7	1.2 equiv C <sub>6</sub> F <sub>5</sub> COOK	70	2						
8	6 h	64	4						
9	12 h	74 (69)	4						
10	without Pd(dba) <sub>2</sub>	0	3						
11	without PCy <sub>3</sub> ·HBF <sub>4</sub>	0	9						

<sup>*a*</sup>Reactions were carried out with  $Pd(dba)_2$  (0.02 mmol, 10 mol %),  $PCy_3 \cdot HBF_4$  (0.04 mmol, 20 mol %), **1a** (0.2 mmol, 1.0 equiv), and **2a** (0.3 mmol, 1.5 equiv) in toluene (1 mL) at 150 °C for 20 h. <sup>*b*</sup>GC yields, using *n*-tetradecane as an internal standard and an isolated yield is shown in parentheses.

#### 3. Experimental Procedures and Spectroscopic Data for the Products

3.1 General procedure for palladium-catalyzed decarbonylative and decarboxylative cross-coupling of acyl chlorides 1 with potassium perfluorobenzoates 2.



In an oven-dried Schlenk tube (25 mL) containing a magnetic stirring bar,  $Pd(dba)_2$  (0.02 mmol, 10 mol %),  $PCy_3 \cdot HBF_4$  (0.04 mmol, 20 mol %), acyl chloride **1** (0.2 mmol, 1.0 equiv), and potassium perfluorobenzoate **2** (0.3 mmol, 1.5 equiv) were charged in toluene (1 mL) under argon. The mixture was heated at 150 °C in oil bath with stirring for 12 h or 20 h. After reaching room temperature, the mixture was quenched with saturated  $NH_4Cl$  and the aqueous solution was extracted with  $Et_2O$ . The combined organic extracts were dried over anhydrous  $MgSO_4$ , then filtered and evaporated under vacuum to obtain the crude product, which was purified by column chromatography on silica gel (hexane or EtOAc/hexane) to afford the desired products **3**.

## **3.2 Spectroscopic Data for the Products.**

#### 2-(Pentafluorophenyl)naphthalene (3aa)<sup>2</sup>



3aa

White solid.  $R_f = 0.37$  (hexane). Isolated yield is 69% (40.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.97-7.89 (m, 4H), 7.58-7.55 (m, 2H), 7.51-7.49 (m, 1H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.4 (m), – 155.8 (m), –162.5 (m).

#### 1-(Pentafluorophenyl)naphthalene (3ba)<sup>2</sup>





White solid.  $R_f = 0.37$  (hexane). Isolated yield is 71% (41.5 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.00 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 7.2 Hz, 1H), 7.60-7.44 (m, 5H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  – 139.8 (m), -155.0 (m), -162.2 (m).

## 2,3,4,5,6-Pentafluoro-1,1'-biphenyl (3ca)<sup>2</sup>





White solid.  $R_f = 0.52$  (hexane). Isolated yield is 49% (24.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.53-7.46 (m, 3H), 7.44-7.41 (m, 2H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.6 (m), –156.0 (m), –162.6 (m).

#### 2,3,4,5,6-Pentafluoro-3'-methyl-1,1'-biphenyl (3da)<sup>3</sup>



3da

White solid.  $R_f = 0.52$  (hexane). Isolated yield is 45% (23.0 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.39 (t, J = 7.6 Hz, 1H), 7.28 (d, J = 7.6 Hz, 1H), 7.23-7.20 (m, 2H), 2.42 (s, 3H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.5 (m), –156.2 (m), –162.7 (m).

## 2,3,4,5,6-Pentafluoro-4'-methyl-1,1'-biphenyl (3ea)<sup>3</sup>



3ea

White solid.  $R_f = 0.45$  (hexane). Isolated yield is 45% (23.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.31 (s, 4H), 2.42 (s, 3H); <sup>19</sup>F {<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  -143.7 (m), -156.5 (m), -162.8 (m).

4'-(tert-Butyl)-2,3,4,5,6-pentafluoro-1,1'-biphenyl (3fa)<sup>2</sup>



White solid.  $R_f = 0.46$  (hexane). Isolated yield is 46% (27.7 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.51 (dt, J = 8.8, 2.2 Hz, 2H), 7.38-7.35 (m, 2H), 1.37 (s, 9H); <sup>19</sup>F {<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.7 (m), –156.5 (m), –162.8 (m).

## 2,3,4,5,6-Pentafluoro-1,1':4',1''-terphenyl (3ga)<sup>2</sup>



White solid.  $R_f = 0.27$  (hexane). Isolated yield is 47% (30.4 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.72 (dt, J = 8.8, 2.0 Hz, 2H), 7.66-7.63 (m, 2H), 7.52-7.46 (m, 4H), 7.42-7.38 (m, 1H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.5 (m), –155.8 (m), –162.5 (m).

Methyl 2',3',4',5',6'-pentafluoro-[1,1'-biphenyl]-4-carboxylate (3ha)<sup>2</sup>



White solid.  $R_f = 0.24$  (EtOAc : hexane = 1:20). Isolated yield is 61% (37.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.16 (dt, J = 8.4, 2.0 Hz, 2H), 7.53-7.50 (m, 2H), 3.96 (s, 3H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.2 (m), –154.5 (m), –162.0 (m).

## 2,3,4,5,6-Pentafluoro-4'-(trifluoromethyl)-1,1'-biphenyl (3ia)<sup>2</sup>



White solid.  $R_f = 0.44$  (hexane). Isolated yield is 48% (30.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.77 (d, J = 8.0 Hz, 2H), 7.57 (d, J = 8.0 Hz, 2H); <sup>19</sup>F {<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  -63.3 (s), -143.3 (m), -154.1 (m), -161.7 (m).

#### 4'-Chloro-2,3,4,5,6-pentafluoro-1,1'-biphenyl (3ja)<sup>3</sup>



White solid.  $R_f = 0.56$  (hexane). Isolated yield is 34% (18.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.48 (dt, J = 8.8, 2.4 Hz, 2H), 7.38-7.35 (m, 2H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.5 (m), –155.1 (m), –162.1 (m).

## 2',3',4',5',6'-Pentafluoro-*N*,*N*-dipropyl-[1,1'-biphenyl]-4-sulfonamide (3ka)<sup>4</sup>



Pale yellow solid.  $R_f = 0.34$  (EtOAc:hexane = 1:20). Isolated yield is 52% (42.4 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.93 (dt, J = 8.8, 2.0 Hz, 2H), 7.59-7.55 (m, 2H), 3.15-3.11 (m, 4H), 1.64-1.55 (m, 4H), 0.89 (t, J = 7.4 Hz, 6H); <sup>19</sup>F {<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –143.1 (m), –153.8 (m), –161.6 (m).

## 2-(2,6-Difluorophenyl)naphthalene (3ab)<sup>2</sup>



3ab

White solid.  $R_f = 0.39$  (hexane). Isolated yield is 63% (30.2 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.98-7.89 (m, 4H), 7.60-7.52 (m, 3H), 7.36-7.29 (m, 1H), 7.06-7.02 (m, 2H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –114.7 (m).

# 2-(2,4,6-Trifluorophenyl)naphthalene (3ac)<sup>1c</sup>



3ac

White solid.  $R_f = 0.47$  (hexane). Isolated yield is 42% (21.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.94-7.87 (m, 4H), 7.56-7.51 (m, 3H), 6.85-6.77 (m, 2H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –109.2 (m), – 111.5 (m).

## 2-(2,3,5,6-Tetrafluoro-4-methylphenyl)naphthalene (3ad)<sup>2</sup>



3ad

White solid.  $R_f = 0.47$  (hexane). Isolated yield is 54% (31.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.96-7.89 (m, 4H), 7.58-7.52 (m, 3H), 2.35 (t, J = 2.2 Hz, 3H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –144.4 (m), –145.8 (m).

## 2-(2,3,5,6-Tetrafluorophenyl)naphthalene (3af)<sup>2</sup>





White solid.  $R_f = 0.44$  (hexane). Isolated yield is 20% (11.2 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.98-7.95 (m, 2H), 7.92-7.90 (m, 2H), 7.60-7.53 (m, 3H), 7.15-7.07 (m, 1H); <sup>19</sup>F{<sup>1</sup>H} NMR (376 MHz, CDCl<sub>3</sub>, rt):  $\delta$  –139.4 (m), –144.0 (m).

#### Negative scope (for reference only)





An oven-dried Schlenk tube (25 mL) containing a magnetic stirring bar was charged with Pd(PCy<sub>3</sub>)<sub>2</sub> (94.8 mg, 0.1 mmol, 1.0 equiv) and benzoyl chloride (**1c**) (14.1 mg, 0.1 mmol, 1.0 equiv) in toluene (0.5 mL) under nitrogen. The reaction was stirred at room temperature for 24 h. The mixture was removed by cannulation and the insoluble yellow powder **5** was then dried under vacuum in 26% isolated yield (20.7 mg, 0.026 mmol).<sup>5</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.42 (t, *J* = 7.2 Hz, 1H), 7.27-7.23 (m, 2H), 7.18-7.15 (m, 2H), 2.16-2.02 (m, 12H), 1.79-1.13 (m, 48H), 0.96-0.93 (m, 6H); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>, rt):  $\delta$  19.7.



## 3.4 Decarbonylation of Complex 5 upon Heating



An oven-dried Schlenk tube (25 mL) containing a magnetic stirring bar was charged with Pd(dba)<sub>2</sub> (23.0 mg, 0.04 mmol, 0.4 equiv), PCy<sub>3</sub> (22.4 mg, 0.08 mmol, 0.8 equiv), and benzoyl chloride (**1c**) (14.1 mg, 0.1 mmol, 1.0 equiv) in toluene (0.5 mL) under argon. The mixture was stirred at room temperature for 24 h and presented <sup>31</sup>P{<sup>1</sup>H} NMR in CDCl<sub>3</sub> proposed as Pd(PCy<sub>3</sub>)<sub>2</sub> ( $\delta$  25.2) and *trans*-Pd(PCy<sub>3</sub>)<sub>2</sub>(PhCO)Cl **5** ( $\delta$  20.0).<sup>6</sup>



The mixture was then heated in an oil bath at 140 °C for 3 h with stirring. After reaching room temperature, <sup>31</sup>P{<sup>1</sup>H} NMR of the mixture showed that the signal at  $\delta$  20.0 had completely disappeared and a new signal appeared at  $\delta$  20.4. These can be assigned to *trans*-Pd(PCy<sub>3</sub>)<sub>2</sub>(Ph)Cl ( $\delta$  20.4) and Pd(PCy<sub>3</sub>)<sub>2</sub> ( $\delta$  25.2), respectively.



#### 3.5 Stoichiometric Reaction of Complex 6 with 2a



Complex **6** was synthesized by the corresponding reports<sup>7</sup> and showed a singlet at  $\delta$  20.4 in the <sup>31</sup>P{<sup>1</sup>H} NMR spectrum in CDCl<sub>3</sub>. An oven-dried Schlenk tube (25 mL) containing a magnetic stirring bar was charged with *trans*-Pd(Ph)Cl(PCy<sub>3</sub>)<sub>2</sub> (78.0 mg, 0.1 mmol, 1.0 equiv) and potassium pentafluorobenzoate (**2a**) (37.5 mg, 0.15 mmol, 1.5 equiv) in toluene (0.5 mL) under argon. The mixture was heated at 140 °C in an oil bath with stirring for 12 h. After being at room temperature, the mixture was quenched with saturated NH<sub>4</sub>Cl and the aqueous solution was extracted with Et<sub>2</sub>O. The combined organic extracts were dried over anhydrous MgSO<sub>4</sub>, then filtered and evaporated under vacuum to detect the product **3ca** in 77% GC yield, using *n*-tetradecane as an internal standard.

4. Copies of <sup>1</sup>H and <sup>19</sup>F{<sup>1</sup>H} NMR Charts for the Products



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of 3aa (rt, CDCl\_3).



 $^{1}$ H NMR (400 MHz) and  $^{19}$ F{ $^{1}$ H} NMR (376 MHz) spectra of **3ba** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of 3ca (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3da** (rt, CDCl<sub>3</sub>).



 $^{1}$ H NMR (400 MHz) and  $^{19}$ F{ $^{1}$ H} NMR (376 MHz) spectra of **3ea** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3fa** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3ga** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3ha** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3ia** (rt, CDCl<sub>3</sub>).



 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3ja** (rt, CDCl<sub>3</sub>).



 $^{1}$ H NMR (400 MHz) and  $^{19}$ F{ $^{1}$ H} NMR (376 MHz) spectra of **3ka** (rt, CDCl<sub>3</sub>).















 $^1H$  NMR (400 MHz) and  $^{19}F\{^1H\}$  NMR (376 MHz) spectra of **3af** (rt, CDCl<sub>3</sub>).

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