# Synthesis of 2-Aryl- and 2,5-Diarylfurans and Thiophenes by Suzuki–Miyaura Reactions Using Potassium Trifluoroborate Salts and Heteroaryltellurides

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The Suzuki–Miyaura cross-coupling reaction of 2-(butyltellanyl) or 2,5-bis-(butyltellanyl)furans and thiophenes with potassium aryltrifluoroborate salts catalyzed by palladium afforded 2-aryl- or 2,5-diaryl-furans and thiophenes in moderate to good yields.

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## Introduction

Heterobiaryls have important biological properties, and the biaryl unit is present in several types of compounds of current interest including natural products, polymers, advanced materials, liquid crystals, luminescent molecular chemosensors, and molecules of medicinal interest.<sup>[1,2]</sup> The preparation of heterobiaryls using the Suzuki–Miyaura coupling reaction, in which the aryl moieties comprise either one or both of the heterocyclic rings, has attracted considerable attention, and there are examples of this type of synthesis.<sup>[1–3]</sup>

2,5-Diphenylfurans have already been prepared by Stille coupling,<sup>[4–6]</sup> Suzuki–Miyaura coupling,<sup>[6]</sup> Paal–Knoor synthesis,<sup>[7]</sup> and from phenacyl bromides<sup>[8]</sup> as well as from butane or butyndiones by microwave irradiation.<sup>[9]</sup> The structural feature of furans with conjugated aromatic substituents at the 2- and 5-positions should be associated with useful and specific fluorescent properties.<sup>[10,11]</sup>

Boronic acids and boronate esters are the most commonly used derivatives in Suzuki–Miyaura cross-coupling reactions. Recently, potassium organotrifluoroborate salts have been used as an alternative to these boron reagents in Suzuki–Miyaura cross-coupling reactions.<sup>[12–15]</sup> These salts are easily prepared by the addition of a saturated aqueous solution of inexpensive, widely available KHF<sub>2</sub> to a great variety of organoboron intermediates. Organotrifluoroborates are stable, non-hygroscopic, relatively nucleophilic compounds and their inorganic byproducts are environmentally benign and easily removed in the workup step.<sup>[16,17]</sup>

Over the past decade, organotellurium chemistry has been extensively explored, and many methods employing tellurium compounds have been developed.<sup>[18,19]</sup> Among these methods, organotellurium reagents were successfully used as the electrophilic partner<sup>[20]</sup> in several metal-catalyzed crosscoupling reactions, such as the Sonogashira,<sup>[21–24]</sup> Negishi,<sup>[25]</sup> Heck,<sup>[26–28]</sup> and Suzuki–Miyaura reactions.<sup>[29–32]</sup> Due to the important and advantageous features of potassium organotrifluoroborate salts and organotellurium compounds in Suzuki–Miyaura cross-coupling reactions, we have endeavoured to develop new methods for construction of biaryls, enynes, stilbenes, and 1,3-diene systems.<sup>[29–32]</sup> Extending our research interest in the preparation and reactivity of potassium organo-trifluoroborate salts, we report herein a new approach for the synthesis of 2-aryl- or 2,5-diarylfurans and thiophenes compounds by the palladium-catalyzed cross-coupling reaction of heteroaryl tellurides and potassium aryltrifluoroborate salts.

## **Results and Discussion**

Our initial studies focussed on the development of an optimum set of reaction conditions for the coupling reaction of 2,5-bis(butyltellanyl)furans<sup>[33]</sup> and 2,5-bis(butyltellanyl) thiophenes<sup>[34]</sup> with potassium aryltrifluoroborate salts. In preliminary experiments, we investigated the catalyzed cross-coupling reaction between 2,5-bis(butyltellanyl)furan **1** and phenyltrifluoroborate salt **2a** in methanol, using Pd(OAc)<sub>2</sub> as catalyst and 1,1'-bis-(diphenylphosphino)ferrocene (dppf) as the ligand at room temperature in one step, which produced 2,5-diphenylfuran **3a** in 73% yield (Table 1; entry 11). As we reported earlier,<sup>[29–31]</sup> the influence of silver salt

As we reported earlier,  $[2^{2-31}]$  the influence of silver salt as an additive was noteworthy in this cross-coupling reaction. When the reaction was performed in the absence of Ag<sub>2</sub>O, no cross-coupling product was isolated and the starting material **1** was recovered unchanged (Table 1, entry 12). Also tested were AgOAc and CuI (Table 1, entries 16 and 17, respectively) as additives, and no product formation was observed in either case.

The use of PdCl<sub>2</sub>, Pd(PPh<sub>3</sub>)<sub>4</sub>, and Pd(dppf)Cl<sub>2</sub> in the presence of Ag<sub>2</sub>O yielded the desired product, but in unsatisfactory amounts (Table 1, entries 2–4). Among the several catalyst systems tested, Pd(OAc)<sub>2</sub> (10 mol%), using dppf (0.1 mmol) as ligand and Ag<sub>2</sub>O as additive, was found to be ideal and the product was obtained in 73% isolated yield (Table 1, entry 11).

	$Bu^{n}Te \longrightarrow PhBF_{3}K \longrightarrow Ph O Ph$					
	1	2a	3a			
Entry	Catalyst (mol%)	Ligand (mmol)	Base (equiv.)	Yield <sup>A</sup> [%]		
1	Pd(OAc) <sub>2</sub> (10)	_		34		
2	PdCl <sub>2</sub> (10)	—	_	36		
3	Pd(PPh <sub>3</sub> ) <sub>4</sub> (10)	_	_	38		
4	Pd(dppf)Cl <sub>2</sub> (10)		_	48		
5	PdCl <sub>2</sub> (10)	PPh <sub>3</sub> (0.2)	_	48		
6	Pd(OAc) <sub>2</sub> (10)	PPh <sub>3</sub> (0.2)	_	50		
7	$Pd(OAc)_2$ (10)	BINAP (0.05)	_	45		
8	Pd(OAc) <sub>2</sub> (10)	Tri(o-tolyl)phosphine (0.1)	_	50		
9	$Pd(OAc)_2$ (10)	Tri(2-furyl)phosphine (0.1)	_	53		
10	Pd(OAc) <sub>2</sub> (10)	dppe (0.1)	_	55		
11	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	_	73		
12 <sup>B</sup>	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	_	_		
13	Pd(OAc) <sub>2</sub> (20)	dppf (0.1)	_	75		
14	$Pd(OAc)_2(5)$	dppf (0.1)	_	50		
15	$Pd(OAc)_2(1)$	dppf (0.1)	_	30		
16 <sup>C</sup>	$Pd(OAc)_{2}$ (10)	dppf (0.1)	_	_		
17 <sup>D</sup>	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	_			
18	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	Et <sub>3</sub> N (3)			
19	$Pd(OAc)_{2}$ (10)	dppf (0.1)	$K_2CO_3(3)$			
20	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	$Cs_2CO_3(3)$			
21	$Pd(OAc)_{2}$ (10)	dppf (0.1)	DIPEA (3)	45		
22	Pd(OAc) <sub>2</sub> (10)	dppf (0.1)	DMAP(3)	—		
23	$Pd(OAc)_2$ (10)	dppf (0.1)	Pyridine (3)	_		

Table 1. Screening for the best Suzuki-Miyaura cross-coupling reaction conditions

Reaction

conditions

T

<sup>A</sup>Isolated yield.

<sup>B</sup>No additive was employed.

<sup>C</sup>AgOAc was employed instead of Ag<sub>2</sub>O.

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 $^{D}CuI$  was employed instead of Ag<sub>2</sub>O.

No significant improvement on the yield occurred with either higher stoichiometry of  $Pd(OAc)_2$  (20 mol%) (Table 1, entry 13) or when lower catalyst loadings were used (Table 1, entries 14 and 15). The reactions were monitored by consumption of starting material and appearance of the desired product by TLC analysis.

In addition, other ligands such as triphenylphosphine, 2,2bis(diphenylphosphino)-1,1'-binaphthyl (BINAP), tri(*o*-tolyl) phosphine, tri(2-furyl)phosphine, and dppe (Table 1, entries 6–10 respectively) were tested and in all cases the desired product formed, but in unsatisfactory yields.

Finally, to optimize the reaction several bases such as  $Et_3N$ ,  $K_2CO_3$ ,  $Cs_2CO_3$ , dimethylaminopyridine (DMAP), *N*,*N*-diisopropylethylamine (DIPEA), and pyridine (Table 1, entries 18–21) were used. Only the use of DIPEA afforded the desired product, but in a modest yield of 45% (Table 1, entry 21).

To demonstrate the efficiency of this reaction, we explored the method by extending these conditions to other potassium aryl-trifluoroborate salts and to 2,5-bis(butyltellanyl)thiophene **4**, to afford the 2,5-diarylfurans **3** and thiophenes **5** (Table 2).

However, when other potassium aryltrifluoroborate salts were employed, only reasonable yields to 2,5-diarylfurans were achieved (35–73%). In all cases we observed a homocoupling reaction of the potassium aryltrifluoroborate salts, which led to the formation of the respective biaryl products. Even the use of an excess of potassium aryltrifluoroborate did not afford better yields.

In the same manner, the cross-coupling between 2,5bis(butyltellanyl)thiophene **4** and potassium aryltrifluoroborate salts 2a-e afforded low to medium yields (15–55%) and the homocoupling product was also observed.

The effect of substituents was also studied, as shown in Table 2. When potassium aryltrifluoroborate salts containing electron-withdrawing groups were used no reaction was observed (Table 2, entries 6 and 7) except when fluorine was attached in the *para*-position (Table 2, entry 3). Electron-donating groups were also used, but with no further benefit. When potassium heteroaryltrifluoroborate salts, such as pyridinyl or thiophenyl were employed, no reaction was observed (Table 2, entries 8 and 9).

When the same standard catalyst systems were employed with 2-(butyltellanyl)furan 6 and 2-(butyltellanyl)thiophene 7, the corresponding 2-arylfurans 8 and 2-arylthiophenes 9 were obtained in better yields as compared with the 2,5-diaryl derivatives (Table 3).

The effects of substituents with these substrates were comparable to those observed with the 2,5-bis(butyltellanyl) derivatives. When potassium aryltrifluoroborate salts containing electron-withdrawing substituents were used, no reaction was observed (Table 3, entries 6 and 7) except when fluorine was located in the *para*-position (Table 3, entry 5). However, when potassium 2-thiophenyltrifluoroborate was coupled with **6**, the corresponding product was achieved in 34% yield (Table 3, entry 9).

## Conclusions

We have presented here a new approach to the synthesis of 2,5-diarylfurans and 2,5-diarylthiophenes, as well as

Bu <sup>n</sup> Te -	X TeBu <sup>n</sup> + 2ArBF <sub>3</sub> K room MeOt	$H, Ag_2O$	ar - X Ar	$\sqrt{1}$	$TeBu^n$ + ArBF <sub>3</sub> K $HeO$	H, $Ag_2O$	X Ar
	X = O 1 2a-i X = S 4		X = O 3a–i X = S 5a–i	X = O 6 X = S 7	2a–i		X = O 8a–i X = S 9a–i
Entry	Compound	Х	Yield [%] <sup>A</sup>	Entry	Compound	Х	Yield [%] <sup>A</sup>
1		O 3a S 5a	73 <sup>B</sup> 45 <sup>C</sup>	1	x	O 8a S 9a	68 <sup>B</sup> 55 <sup>B</sup>
2		O 3b S 5b	60 <sup>C</sup> 15	2	X	O 8b S 9b	55 <sup>B</sup> 64 <sup>C</sup>
3		O 3c S 5c	55 <sup>B</sup> 32 <sup>C</sup>	3 <sup>D</sup>	x	O 8c S 9c	50 <sup>C</sup> 71 <sup>B</sup>
4	MeO OMe	O 3d S 5d	55 <sup>C</sup> 55 <sup>B</sup>	4	NH <sub>2</sub>	O 8d	65 <sup>B</sup> 60 <sup>B</sup>
5 <sup>D</sup>	H <sub>2</sub> N X NH <sub>2</sub>	O 3e S 5e	35 <sup>C</sup> 50 <sup>C</sup>	5	OMe	0 8e	30 <sup>B</sup>
6	HO <sub>2</sub> C CO <sub>2</sub> H	O 3f S 5f	n.r. <sup>E</sup> n.r.	6	F	S 9e O 8f	n.r. <sup>E</sup>
7	$F_3C$ $CF_3$ $CF_3$ $CF_3$	O 3g S 5g	n.r. n.r.	7	CO <sub>2</sub> H	S 9f O 8g	n.r. n.r.
8	X	O 3h S 5h	n.r. n.r.		CF <sub>3</sub>	S 9g	n.r.
9	C X S	O 3i S 5i	n.r. n.r.	8	"X"	O 8h S 9h	n.r. n.r.
Alsolate	- d vield			9	× s	O 8i S 9i	34 n.r.

Table 2. Synthesis of 2,5-diarylfurans and thiophenes by a Suzuki-Miyaura cross-coupling reaction Pd(OAc), dppf

Table 3. Synthesis of 2,5-diarylfurans and thiophenes by a Suzuki-Miyaura cross-coupling reaction

Pd(OAc), dppf

a palladium-catalyzed cross-coupling reaction between organo-

tellurium compounds and potassium aryltrifluoroborate salts. More than 20 examples of 2,5-diaryl and 2-arylheteroaryl species were prepared in 30-73% overall yield. Studies aimed towards the template-directed synthesis of more complex structures are in progress in our laboratory.

2-arylfurans and 2-arylthiophenes. Our methodology consists of

# **Experimental**

<sup>A</sup>Isolated yield. <sup>B</sup>Reaction time: 1.5 h. <sup>C</sup>Reaction time: 2.0 h.

En.r. = no reaction.

<sup>D</sup>Using (3-aminophenyl)boronic acid.

## General Procedure for 2,5-Diarylfurans and Thiophenes

In a two-necked round-bottomed flask (15 mL), under a nitrogen atmosphere, were added 2,5-bis(butyltellanyl)furan or thiophene A Isolated vield.

<sup>B</sup>Reaction time: 1.5 h.

<sup>C</sup>Reaction time: 1.5 h.

<sup>D</sup>Using (3-aminophenyl)boronic acid.

 $^{E}$ n.r. = no reaction.

(0.5 mmol), silver(1) oxide (1 mmol), dppf (20 mol%, 0.1 mmol), and palladium(II) acetate (10 mol%, 0.05 mmol) in dry methanol (4 mL), after which was added potassium aryltrifluoroborate (1.1 mmol). The reaction mixture was stirred at room temperature and the consumption of the starting materials was followed by TLC. Fluorescent side products that formed as the reaction proceeded were visible on the TLC plate under long-wavelength UV light. The reaction mixture was filtered through a pad of Celite and the filtrate partitioned between ethyl acetate and saturated NH<sub>4</sub>Cl. The organic layer was washed with saturated  $NH_4Cl(3 \times 10 \text{ mL})$ , dried over  $Mg_2SO_4$ , and concentrated under

vacuum. The product was purified by silica gel chromatography (100% hexane).

#### 2,5-Bis(anisole)furan 3d

 $\delta_{\rm H}$  (300 MHz, CDCl<sub>3</sub>) 3.87 (s, 6H), 6.61 (s, 2H), 6.97 (d, *J* 7.83, 4H), 7.69 (d, *J* 7.95, 4H).  $\delta_{\rm C}$  (75 MHz, CDCl<sub>3</sub>) 158.9, 152.8, 125.0, 124.0, 114.2, 105.6, 55.4. *m/z* 1H and 3C 280 (100%), 265 (93), 194 (10), 140 (31), 77 (5), 43 (4).

## 2,5-Bis(anisole)thiophene 5d

 $\delta_{\rm H}$  (300 MHz, CDCl<sub>3</sub>) 3.83 (s, 6H), 6.91 (d, *J* 8.76, 4H), 7.13 (s, 2H), 7.53 (d, *J* 8.73, 4H).  $\delta_{\rm C}$  (75 MHz, CDCl<sub>3</sub>) 159.1, 142.6, 126.8, 122.9, 114.3, 114.2, 55.4. *m/z* 296 (100%), 281 (77), 266 (8), 210 (15), 148 (40), 127 (17), 77 (2).

#### General Procedure for 2-Arylfurans and Thiophenes

In a two-necked round-bottomed flask (15 mL), under a nitrogen atmosphere, were added 2-(butyltellanyl)furan or thiophene (0.5 mmol), silver(I) oxide (1 mmol), dppf (20 mol%, 0.1 mmol), and palladium(II) acetate (10 mol%, 0.05 mmol) in dry methanol (4 mL), after which was added potassium aryltrifluoroborate (0.55 mmol). The reaction mixture was stirred at room temperature and the consumption of the starting materials was followed by TLC. Fluorescent side products, that formed as the reaction proceeded, were visible on the TLC plate under long-wavelength UV light. The reaction mixture was filtered through a pad of Celite and the filtrate partitioned between ethyl acetate and saturated NH<sub>4</sub>Cl. The organic layer was washed with saturated NH<sub>4</sub>Cl ( $3 \times 10$  mL), dried over Mg<sub>2</sub>SO<sub>4</sub>, and concentrated under vacuum. Purification was performed by silica gel chromatography (100% hexane).

#### 2-(Anisole)furan 8d

 $δ_{\rm H}$  (300 MHz, CDCl<sub>3</sub>) 3.85 (s, 3H), 6.47 (dd, *J* 3.21, 1.8, 1H), 6.54 (d, *J* 3.3, 1H), 6.94 (d, *J* 8.79, 2H), 7.45 (d, *J* 0.96, 1H), 7.63 (d, *J* 8.79).  $δ_{\rm C}$  (75 MHz, CDCl<sub>3</sub>) 159.0, 154.1, 141.4, 125.2 (2C), 124.1, 114.1 (2C), 111.5, 103.4, 55.3. *m/z* 174 (78%), 159 (67), 131 (16), 77 (14), 43 (100).

#### 2-(Anisole)thiophene 9d

 $δ_{\rm H}$  (300 MHz, CDCl<sub>3</sub>) 3.81 (s, 3H), 6.89 (d, *J* 8.73, 2H), 7.01– 7.03 (m, 1H), 7.17–7.23 (m, 2H), 7.52 (d, *J* 8.73, 2H).  $δ_{\rm C}$ (75 MHz, CDCl<sub>3</sub>) 159.2, 144.4, 127.9, 127.3, 127.2 (2C), 123.8, 122.1, 114.3 (2C), 55.4. *m/z* 190 (98%), 175 (100), 147 (48), 77 (10), 45 (17).

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