Note

# Palladium-catalyzed carbonylative cross-coupling reaction of triarylantimony dicarboxylates with arylboronic acids: Synthesis of biaryl ketones

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## ABSTRACT

A novel palladium-catalyzed three-component carbonylative coupling reaction by use of triarylantimony diacetate, arylboronic acid and carbon monoxide (1 atm) could be attained under mild reaction conditions without any base as an additive. The reaction can be applied to a wide range of triarylantimony diacetates and boronic acids to afford the corresponding unsymmetrical biaryl ketones in good yields.

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## 1. Introduction

Palladium(Pd)-catalyzed carbonylative coupling reaction of organic halides with various nucleophiles with main group elements is an effective tool for the synthesis of a wide variety of symmetrical and unsymmetrical ketones [1]. Among them, the reaction by use of organoboron reagents such as arylboronic acids and arylboronates has attracted much interest on account of its high efficiency, high chemoselectivity, and wide applicability [1,2]. However the development of the protocol by use of aryl halides and arylboronic acids is limited due to a side reaction that gives a direct coupling product without carbon monoxide (CO) insertion [3]. In order to overcome this inefficiency, aryl diazonium salts [4] and hypervalent iodines [5] were utilized as electrophiles instead of aryl halides. It has also been reported that, in these reactions, the nature of the substrate and electrophile as well as the reaction conditions such as Pd catalyst, ligand, base, temperature, solvent and pressure of CO brought about a dramatic effect on the selectivity in the formation of ketone and/or biaryl formed without CO insertion.

Much attention has been paid to the chemistry of organoantimony compounds which has widespread use for organic synthesis [6,7]. However, to the best of our knowledge, only a few examples of Pd-catalyzed carbonylative coupling reaction by use of organ-

oantimony compounds have been reported to date. For instance, triarylantimony diacetates were successfully coupled under CO atmosphere with alkynylsilanes [8] to form alkynyl ketones and organostannanes [9] to give aryl- and vinyl ketones. We have also recently demonstrated a new carbonylative cross-coupling reaction between ethynylstibanes and aryl iodides to give ethynyl ketones [10]. Meanwhile we have recently reported that triarylantimony diacetates were useful coupling partners for Pd-catalyzed cross-coupling reactions with a variety of arylboronic acids and triarylbismuthanes, and the corresponding biaryls were synthesized under mild reaction conditions without a base as a activator of boron or bismuth reagent [11]. As an extension of this work, we present here an efficient Pd(0)-catalyzed carbonylative coupling reaction of triarylantimony diacetates as electrophile with arylboronic acids under CO atmosphere to form symmetrical and unsymmetrical diaryl ketones. It should be noted that the reaction took place smoothly giving the expected coupling products without any additive such as base for activation of boron or antimony reagent.

## 2. Results and discussion

### 2.1. Optimization of the reaction conditions

In our previous work, we demonstrated the Pd-catalyzed Suzuki-type cross-coupling reaction of pentavalent triarylantimony diacetates with arylboronic acids, in that the reaction was performed

in 1,4-dioxane at 60 °C in the presence of Pd(PPh₃)₄ catalyst (5 mol%) without any base under argon atmosphere [11]. Elsewhere it has been reported that the product distribution toward the expected carbonylative cross-coupling product or direct cross-coupling product without CO insertion was largely dependent on the nature of solvents and additives [2,3]. Thus, we chose the reaction of triphenylantimony diacetate (**1a**) with p-tolylboronic acid (**2a**, 3.0 equiv.) as a model system and searched for the optimal reaction conditions for the expected carbonylative cross-coupling reaction under CO atmosphere by using a variety of solvents. The progress of the reaction was monitored by gas-liquid chromatography (GLC) while measuring the yields of the coupling product **3a** and direct coupling product **4a**, because **1a** and **2a** could not be detected on thin-layer chromatography (TLC) or GLC. The results are shown in Table 1. The reaction by use of 1,4-dioxane, which has been shown to be the best solvent for direct cross-coupling reaction between **1a** and **2a** to form **4a**, gave unsatisfactory results in terms of the selectivity of the product. The reaction afforded **3a** in 75% yield accompanied by perceptible amounts (24%) of direct coupling product **4a** (Table 1, entry 1). When the reaction was carried out in another ethereal solvent, tetrahydrofuran (THF), the selectivity was considerably lowered and gave approximately a 1:1 mixture of **3a** and **4a** (Table 1, entry 2). Similar results were obtained by use of toluene and 1,2-dichloroethane (1,2-DCE) and the expected improvement in the selectivity was not observed (Table 1, entries 3, 4). In contrast, the use of polar aprotic solvents such as dimethylacetamide (DMA) and N-methylpyrrolidone (NMP) brought about desirable selectivity and carbonylated product **3a** was formed in good yields with low yields of uncarbonylated side product **4a**, although acetonitrile gave unexpected result in the yield of **3a** (Table 1, entries 5–7). These results are in marked contrast to that, in the carbonylative coupling reaction between aryl halides and boric acids, the use of ethereal solvents such as dioxane led to the predominant formation of carbonylated coupling product in the best yield [3d]. The results in Table 1 also show that one of three phenyl groups on the antimony reagent can be coupled in the reaction. It is noteworthy that no perceptible differences in the yield of **3a** and the selectivity were observed even when the amount of boronic acid **2a** was reduced from 3.0 equiv. to 1.5 equiv. (Table 1, entry 8). A perceptible

improvement in the yield of **3a** was also observed when the pressure of CO was increased from 1 (5%) to 20 atm (42%) at room temperature in the present carbonylative cross-coupling reaction (Table 1, entries 9–11). Consequently, the best result was obtained when **1a** was treated with **2a** (1.5 equiv.) in the presence of Pd(PPh₃)₄ (5 mol%) at 60 °C phenylboronic NMP as a solvent.

## 2.2. Carbonylative cross-coupling reaction of triphenylantimony diacetate with arylboronic acids

In order to prove the generality of the present carbonylative cross-coupling reaction, the reaction of triphenylantimony diacetate (**1a**) with various arylboronic acids (**2b–i**: 1.5 equiv.) was examined in NMP under CO atmosphere (1 atm) by use of 5 mol% Pd(PPh₃)₄. The reaction was carried out at 60 °C for 6 h and the results are summarized in Table 2. As a result, all boronic acids gave the corresponding aryl ketones **3b–i** in moderate to good yields. In the case of 4-methoxyphenylboronic acid (**2b**), the reaction gave carbonylated coupling product **3b** in 83% yield accompanied by 15% yield of uncarbonylated coupling product, 4-methoxybiphenyl (**4b**) (Table 2, entry 1). Similar results have been reported in the carbonylative cross-coupling reaction between aryldiazonium tetrafluoroborate and electron-rich **2b**, in that 23% yield of **4b** was formed as a side product along with 50% yield of carbonylated coupling product **3b** [4b]. We have also recently reported that the direct cross-coupling reaction of **1a** with electron-rich 4-methoxyphenyl- (**2b**) and 3,4-(methylenedioxy)phenylboronic acid under argon atmosphere was completed in a shorter reaction time than that of electron-deficient ones [11a]. These results imply that the electron-rich boronic acids would promote transmetallation of the Pd-complex Ar²PdSbAr²₂(OAc)L₂ (**A**) to Ar²PdAr¹L₂ (**E**) shown in Scheme 1, and facilitate the pathway to give an uncarbonylated product without CO insertion. 1-Naphthylboronic acid (**2c**) gave the desired product **3c** in modest yield, which could be interpreted from unfavorable steric hindrance of the α-naphthyl group for the progress of the coupling reaction (Table 2, entry 2). In the case of the boronic acids **2d–i** bearing electron-withdrawing substituents, the reaction took place with high selectivity. Thus, the reaction of **2d–i** gave the corresponding biaryl ketones **3e–i** in moderate to good yields, and the formation of biaryls **4d–i** was conspic-

**Table 1**

Carbonylative cross-coupling reaction of triphenylantimony diacetate **1a** with 4-methylphenylboronic acid **2a**[a]
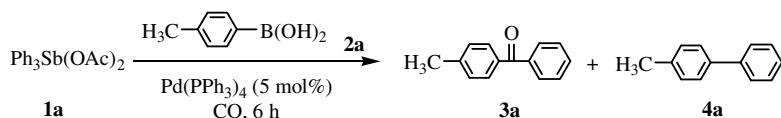


| Entry | Solvent | CO (atm) | Temperature (°C) | Yield[b] (%) | |
|---|---|---|---|---|---|
| | | | | **3a** | **4a** |
| 1 | 1,4-Dioxane | 1 | 60 | 75 | 24 |
| 2 | THF | 1 | 60 | 52 | 47 |
| 3 | Toluene | 1 | 60 | 73 | 15 |
| 4 | 1,2-DCE | 1 | 60 | 77 | 9 |
| 5 | CH₃CN | 1 | 60 | 7 | 1 |
| 6 | DMA | 1 | 60 | 91 | 3 |
| 7 | NMP | 1 | 60 | 97 (95)[c] | 2 (2)[c] |
| 8[d] | NMP | 1 | 60 | 97 | 3 |
| 9 | NMP | 1 | rt | 5 | 1 |
| 10 | NMP | 5 | rt | 38 | 1 |
| 11 | NMP | 20 | rt | 42 | 1 |

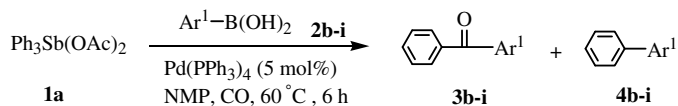[a] **1a** (0.5 mmol), **2a** (1.5 mmol), Pd(PPh₃)₄ (5 mol%), solvent (3 ml).
[b] The yields of **3a** and **4a** were determined by GLC. The yield 100% corresponds to the involvement of one phenyl group on **1a**.
[c] Isolated yields.
[d] **2a** (0.75 mmol).

**Table 2**
Carbonylative cross-coupling reaction of triphenylantimony diacetate **1a** with arylboronic acids **2b–i**[a]

$$Ph_3Sb(OAc)_2 \quad \xrightarrow[\substack{Pd(PPh_3)_4 \ (5 \ mol\%) \\ NMP, \ CO, \ 60\,°C, \ 6\ h}]{Ar^1-B(OH)_2 \ \ 2b\text{-}i} \quad \underset{3b\text{-}i}{\text{Ph-CO-}Ar^1} \ + \ \underset{4b\text{-}i}{\text{Ph-}Ar^1}$$

**1a**

| Entry | Ar¹ (**2b–i**) | Yield[b] (%) | |
|---|---|---|---|
| | | **3** | **4** |
| 1 | 4-Methoxyphenyl (**2b**) | **3b**: 83 | **4b**: 15 |
| 2 | 1-Naphthyl (**2c**) | **3c**: 52 | **4c**: 7 |
| 3 | 4-Chlorophenyl (**2d**) | **3d**: 92 | **4d**: 1 |
| 4 | 4-(Trifluoromethyl)phenyl (**2e**) | **3e**: 81 | **4e**: 4 |
| 5 | 4-Acetylphenyl (**2f**) | **3f**: 83 | **4f**: ND[c] |
| 6 | 4-(Methoxycarbonyl)phenyl (**2g**) | **3g**: 64 | **4g**: 2 |
| 7 | 4-Cyanophenyl (**2h**) | **3h**: 91 | **4h**: 3 |
| 8 | 4-Nitrophenyl (**2i**) | **3i**: 81 | **4i**: ND[c] |

[a] **1a** (0.5 mmol), **2b–i** (0.75 mmol), Pd(PPh₃)₄ (5 mol%), NMP (3 ml).
[b] Isolated yield. The yield 100% corresponds to the involvement of one phenyl group on **1a**.
[c] Not detected.

uously suppressed (Table 2, entries 3–8). The results in Table 2 also suggest that the electron-donating or -withdrawing substituents on arylboronic acid do not affect the progress and the yield of **3** in the present carbonylative cross-coupling reaction.
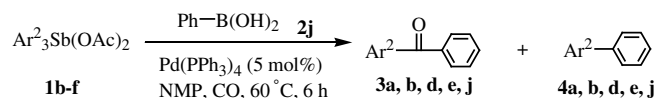
### 2.3. Carbonylative cross-coupling reaction of triarylantimony diacetates with phenylboronic acid

In order to elucidate the correlation between reactivity and electronic nature of the antimony reagent in the present coupling

reaction, we finally attempted the reaction using a variety of triarylantimony diacetates having electron-donating **1b,c** and -withdrawing **1d–f** groups with phenylboronic acid (**2j**) under the same reaction conditions. The results are collected in Table 3. When triarylantimony diacetates having 4-methoxy **1b**, 4-methyl **1c**, 4-chloro **1d**, and 4-ethoxycarbonyl **1e** substituents on the aryl group were reacted under normal reaction conditions, the corresponding carbonylative coupling products **3b**, **3a**, **3d**, and **3j** were formed in good yields (81–95%) with high selectivity (Table 3, entries 1–4). However, the antimony compound **1f** owning electron-withdrawing trifluoromethyl (CF₃) group on position 4 of the benzene ring gave the carbonylated product **3e** in unsatisfactory yield (60%) accompanied by a conspicuous amount of the uncarbonylated coupling product **4e**: 18% (Table 3, entry 5). It has been reported that, in the reaction of aryl halides with arylboric acids, the

**Table 3**
Carbonylative cross-coupling reaction of triarylantimony diacetates **1b–f** with phenylboronic acid (**2j**)[a]

$$Ar^2_3Sb(OAc)_2 \quad \xrightarrow[\substack{Pd(PPh_3)_4 \ (5 \ mol\%) \\ NMP, \ CO, \ 60\,°C, \ 6\ h}]{Ph-B(OH)_2 \ \ 2j} \quad \underset{3a,\,b,\,d,\,e,\,j}{Ar^2\text{-CO-Ph}} \ + \ \underset{4a,\,b,\,d,\,e,\,j}{Ar^2\text{-Ph}}$$

**1b-f**

| Entry | Ar²: **1b–f** | Yield[b] (%) | |
|---|---|---|---|
| | | **3** | **4** |
| 1 | **1b**: 4-Methoxyphenyl | **3b**: 91 | **4b**: 2 |
| 2 | **1c**: 4-Methylphenyl | **3a**: 95 | **4a**: 2 |
| 3 | **1d**: 4-Chlorophenyl | **3d**: 84 | **4d**: 3 |
| 4 | **1e**: 4-Ethoxycarbonylphenyl | **3j**: 81 | **4j**: 2 |
| 5 | **1f**: 4-(Trifluoromethyl)phenyl | **3e**: 60 | **4e**: 18 |

[a] **1b–f** (0.5 mmol), **2j** (0.75 mmol), Pd(PPh₃)₄ (5 mol%), NMP (3 ml).
[b] Isolated yield. The yield 100% corresponds to the involvement of one phenyl group on **1**.
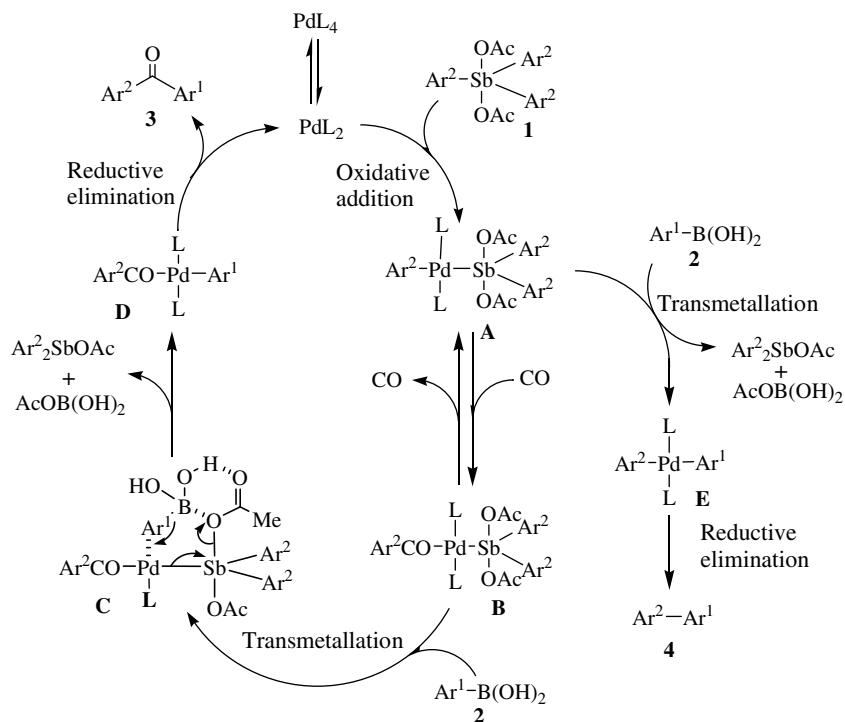


**Scheme 1.** Proposed mechanism.

presence of a strong electron-withdrawing substituent such as nitro group on the aryl halide inhibits CO insertion and promotes the direct cross-coupling reaction [12].

### 2.4. Reaction mechanism

A plausible reaction mechanism of the present carbonylative reaction is depicted in Scheme 1. It has been demonstrated in our previous report that triarylantimony diacetate acts as a pseudo-halogen in the Pd(0)-catalyzed cross-coupling reaction with boronic acids and triarylbismuthanes [11]. Taking these results into consideration, the initial step of the present carbonylative cross-coupling reaction is considered to be oxidative addition of **1** onto Pd(0) catalyst to form complex **A** [11,12]. The insertion of CO between antimony–palladium bonds in complex **A** led to the formation of complex **B** [3a,3c,12]. The formation of diaryl ketones **3** from the complex **B** should proceed along a similar pathway to that of the direct cross-coupling reaction of **1** with **2** reported in our previous paper [11]. Thus, transmetallation between the complex **B** and boronic acid **2** gave diorganopalladium complex **D** *via* intermediate **C**. In the intermediate **C**, the inter-coordination between boronic acid moiety and acetoxy group on antimony (B···O, OH···O) would construct a six-membered ring to activate boronic acid moiety and to accelerate the transmetallation in the absence of a base. Indeed, boron reagents such as 5,5-dimethyl-2-phenyl-1,3,2-dioxaborinane (**2k**), sodium tetraphenylborate (**2l**), and potassium phenyltrifluoroborate (**2m**) having no ability to form the six-membered ring were ineffective in the present reaction. The carbonylated complex **D** thus formed undergoes reductive elimination to give the carbonylative cross-coupling product **3** and reproduces the Pd(0) species.

In the reaction with electron-poor triarylantimony diacetate **1f** bearing $CF_3$ substituent on aryl group, a larger amount of the direct cross-coupling product **4e** was formed under the same reaction conditions. This result can be explained as follows. It has been well documented that the CO insertion into the transition-metal complexes $ArMXL_2$ (M = Ni, Pd, Pt) to form $ArCOMXL_2$ was sensitive to the electric nature of substituents on the aryl group; the insertion was accelerated by the presence of an electron-donating substituent on the aryl group and was inhibited with an electron-withdrawing one [13]. It has also been reported that, in the Pd-catalyzed coupling reaction of *p*-substituted aroyl chlorides (ArCOCl) in the presence of distannane for the preparation of α-diketones [(ArCO)$_2$], decarbonylated products (Ar$_2$CO and Ar–Ar) were formed as by-products, and the decarbonylation process was facilitated by electron-withdrawing substituents [14]. These results imply that insertion of CO into the complex **A** to form the complex **B** is in equilibrium with its reverse reaction from complex **B** to **A** with decarbonylation, and the presence of an electron-withdrawing substituent on the aryl ring not only retards the CO insertion but also stimulates the decarbonylation. As a result, electron-withdrawing $CF_3$ substituent on the aryl group of **1f** would promote the formation of the direct coupling product **4e** in the present reaction.

### 3. Conclusion

In summary, we have explored the Pd-catalyzed carbonylative cross-coupling reaction of arylboronic acids using pentavalent triarylantimony diacetates as an aryl donor and established a new route to prepare asymmetrical diaryl ketones. The reaction proceeds smoothly in the presence of Pd(PPh$_3$)$_4$ as a catalyst in NMP under mild reaction conditions without any base as additives in that one of the three aryl groups on triarylantimony diacetate can participate in the coupling reaction. Both electron-rich and electron-deficient boronic acids gave the expected carbonylated

coupling products in good yield, whereas electron-deficient antimony reagent is relatively ineffective. The results show that the use of an electron-poor boron reagent and an electron-rich antimony reagent is preferable for the preparation of diaryl ketones consisting of electron-rich and -poor aryl groups. This study revealed the triarylantimony dicarboxylates to be practically useful compounds as a synthetic reagent in organic synthesis.

### 4. Experimental

#### 4.1. General

All reactions were carried out in pre-dried glassware under carbon monoxide (CO) atmosphere. Melting points were taken on a Yanagimoto micro melting point hot-stage apparatus (MP-S3) and are not corrected. $^1$H NMR (TMS: $\delta$ 0.00 ppm as an internal standard) spectra were recorded on a JEOL JNM-ECA-400 (400 MHz) spectrometer in CDCl$_3$ unless otherwise stated. Mass spectra (EI-MS) were obtained on a JEOL JMS-700 instrument. GLC analyses of the products were taken using a Shimadzu GC-16A. All chromatographic separations were accomplished with Silica Gel 60N (Kanto Chemical Co., Inc.). Thin-layer chromatography (TLC) was performed with Macherey-Nagel Pre-coated TLC plates Sil G25 UV$_{254}$. GLC analyses were performed by use of the following conditions, 1.5% SE-30, 120–240 °C, 7.5 °C/min temperature increasing program using *n*-octadecane [retention time ($t_R$) = 7.76 min] as an internal standard. High CO pressure reactions were performed in a stainless steel reactor (100 ml) provided by Taiatsu Techno Corp., Japan. NMP was purchased from Wako Pure Chemical Ind. Ltd. Japan, and used without further purification. The triarylantimony diacetates **1** were prepared according to the reported procedure [11b], and boron reagents **2** were purchased from Wako Pure Chemical Industries, Ltd., Tokyo Kasei Kogyo Co., Ltd. Japan, Sigma-Aldrich, Inc. and Kanto Chemical Co., Inc.

#### 4.2. Reaction of triarylantimony diacetates **1** with boron reagents **2**

A mixture of triarylantimony diacetate **1** (0.5 mmol), boronic acids **2** (0.75 mmol), and Pd(PPh$_3$)$_4$ (0.025 mmol) in NMP (3 ml) was heated at 60 °C for 6 h under atmospheric pressure of CO. After dilution with ether (40 ml) and water (20 ml), the reaction mixture was separated and the aqueous layer was extracted with ether (20 ml). The combined organic layer was washed with water, dried over anhydrous MgSO$_4$, and concentrated under reduced pressure. The residue was purified by SiO$_2$ column chromatography using a mixture of hexane, dichloromethane, and ether as eluent to afford biaryl ketones **3a–j** and biaryls **4a–j**, and the results are collected in Tables 2 and 3. All products are known compounds and their structures were determined by comparing their melting points and/or spectral data (MS and NMR) [15–20].

4-Methylphenyl phenyl ketone (**3a**): Colorless prisms, mp 54–56 °C (lit. [15], 57–58 °C).
4-Methoxyphenyl phenyl ketone (**3b**): Colorless prisms, mp 61–62 °C (lit. [15], 61–61.5 °C).
1-Benzoylnaphthalene (**3c**): Colorless prisms, mp 76–77 °C (lit. [19a], 73 °C).
4-Chlorophenyl phenyl ketone (**3d**): Colorless prisms, mp 73–75 °C (lit. [15], 73.5–75 °C).
4-Trifluoromethylphenyl phenyl ketone (**3e**): Colorless needles, mp 116–117 °C (lit. [20], 112–114 °C).
4-Acetylphenyl phenyl ketone (**3f**): Colorless needles, mp 84–85 °C (lit. [16], 80–84 °C).
4-Methoxycarbonylphenyl phenyl ketone (**3g**): Colorless plates, mp 109–110 °C (lit. [17], 111–115 °C).

4-Cyanophenyl phenyl ketone (**3h**): Colorless needles, mp 114–116 °C (lit. [18], 113–114 °C).

4-Nitrophenyl phenyl ketone (**3i**): Pale yellow needles, mp 139.5–142 °C (lit. [15], 138–139 °C).

4-Ethoxycarbonylphenyl phenyl ketone (**3j**): Colorless prisms, mp 48–49 °C (lit. [18], 54–55 °C).

## Acknowledgements

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