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Synthesis of arylstannanes by palladium-catalyzed desulfitative coupling reaction of sodium arylsulfinates with distannanes

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ABSTRACT

A novel Pd-catalyzed desulfitative cross-coupling reaction of sodium arylsulfinates with hexaalkyl distannanes is realized, allowing the facile synthesis of functionalized arylstannanes with moderate to excellent yields. The successful implement of gram-scale synthesis and tandem Stille coupling reaction demonstrates the potential applications of this method in organic synthesis.

Introduction

Organotin reagents, especially for arylstannanes derivatives, have been found great applications in organic synthesis and other fields [1]. In particular, the Stille coupling reaction, in which the essence is the cross-coupling reaction with organotin reagents, is rapidly becoming a powerful synthetic strategy for selective C—C bond formation [2]. Particularly, compared with Suzuki-Miyaura reactions, the Stille coupling methodology has been found to be highly effective for synthesizing functional materials [1j]. Moreover, aryl C-Sn bond can further be applied in functional group transformations, including C—N [3], C—F [4], C—OCF$_3$ [5], and C–B bond [6] formation. The wide application has created considerable need for the easy access to aryl stannane derivatives. However, the synthetic methods for accessing arylstannane compounds are still limited. The most commonly used strategy to synthesize functionalized aryl stannanes is the reaction of trialkyl tin halide with aromatic metal reagents, including aryl magnesium, -lithium, or -zinc reagents (Scheme 1a) [7]. Alternative methods are the aromatic nucleophilic substitution between aryl halides or ammonium salts and trialkyl stannyl anion (Scheme 1b) [8]. In addition, Pd-catalyzed cross-coupling reaction from aryl halides/ArOTf with distannanes has been developed (Scheme 1c) [9]. Moreover, the radical type stannylation of aryl diazonium salts or aryl iodides are carried out under transition-metal-free condition (Scheme 1d) [10]. Recently, the Ni-catalyzed stannylation reactions of aryl esters were developed by Martín [11a] and Rueping [11b], using silylstannyl derivatives and distannanes as the stannylation reagents respectively (Scheme 1e).

Arylsulfinic acids or their arylsulfinate salts, which can be conveniently synthesized from the corresponding aryl sulfonyl halides, have played important role as versatile intermediates in organic synthesis because of their readily availability, air stability and low price [12]. In addition to participating in sulfonylation reactions [13], arylsulfinic acids or sodium arylsulfinates can be served as novel alternatives for transition-metal-catalyzed desulfitative C—C bond formations by releasing SO$_2$ [14]. As a new strategy of aryl sources, the importance of desulfitative coupling reactions has been adequately proved in Heck-type reactions [15], synthesis of biaryls [16], synthesis of aryl ketones [17] and C—H bond arylation [18]. However, desulfitative coupling reaction of arylsulfinates for constructing carbon-heteroatom bond still remains great challenge. Recently, Wang and Xiao have reported the novel Pd-catalyzed desulfitative C—P coupling of sodium arylsulfinates respectively [19a,b]. The Cu-promoted desulfitative *N*-arylation with sodium arylsulfinates was also explored [19c]. Based on our interests in desulfitative coupling reactions and the

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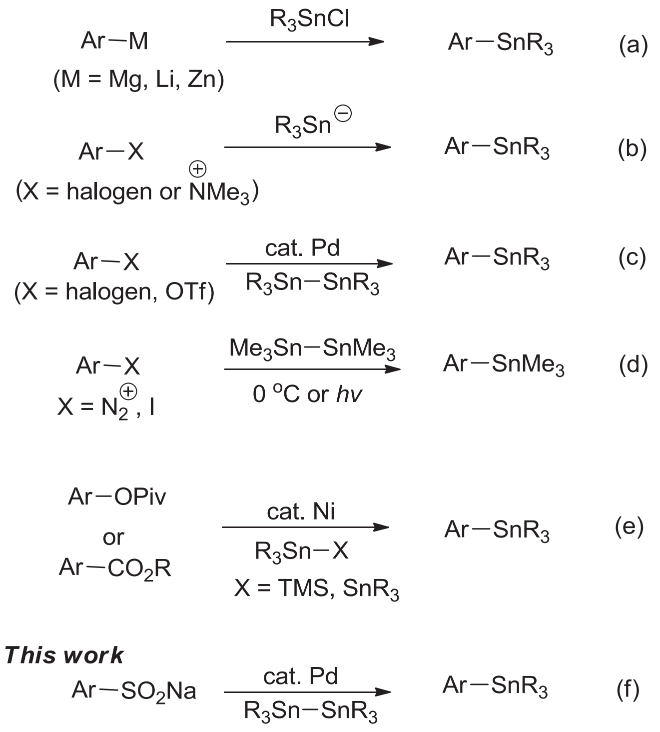
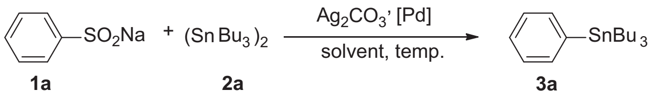


Scheme 1. Synthetic methods for arylstannanes.

Table 1
Optimization of reaction conditions.[a]



Entry	Solvent	T (°C)	[Pd] (mol%)	Yield (%)[b]
1	DMF	60	$\mathrm{Pd(PPh_3)_2Cl_2(5)}$	trace
2	DMF	80	$\mathrm{Pd(PPh_3)_2Cl_2(5)}$	20
3	DMF	110	$\mathrm{Pd(PPh_3)_2Cl_2(5)}$	44
4	DMF	110	$\mathrm{PdCl_2(dppp)}$ (5)	Trace
5	DMF	110	$\mathrm{Pd(acac)_2}$ (5)	20
6	DMF	110	$\mathrm{Pd(P^tBu_3)_2(5)}$	64
7	DMF	120	$\mathrm{Pd(P^tBu_3)_2(5)}$	71
8	DMA	120	$\mathrm{Pd(P^tBu_3)_2(5)}$	73
9	Toluene	120	$\mathrm{Pd(P^tBu_3)_2(5)}$	61
10	Dioxane	120	$\mathrm{Pd(P^tBu_3)_2(5)}$	35
11	DMA	140	$\mathrm{Pd(P^tBu_3)_2(5)}$	93
12	DMA	140	$\mathrm{Pd(P^tBu_3)_2(3)}$	97
13[c]	DMA	140	$\mathrm{Pd(P^tBu_3)_2(3)}$	83
14[d]	DMA	140	$\mathrm{Pd(P^tBu_3)_2(3)}$	63
15[e]	DMA	140	$\mathrm{Pd(P^tBu_3)_2(3)}$	trace
16	DMA	140	/	34

[a] Reaction conditions: sodium benzenesulfinate **1a** (0.3 mmol, 1.5 equiv), hexabutyldistannane **2a** (0.2 mmol, 1.0 equiv), Ag_2CO_3 (0.3 mmol, 1.5 equiv), Pd catalysis (3–5 mol%), solvent (2 mL), 1 h.
[b] Isolated yields.
[c] Ag_2CO_3 (0.24 mmol, 1.2 equiv).
[d] Sodium aryl sulfinate (0.24 mmol, 1.2 equiv).
[e] Without Ag_2CO_3. DMA = *N*,*N*-dimethylacetamide.

importance of aryl stannane reagents, herein we report the desulfitative cross-coupling reaction of sodium aryl sulfinates with hexabutyldistannane under palladium catalysis (Scheme 1f).

Results and discussion

At the outset, we started our investigation by examining Pd $(PPh_3)_2Cl_2$ catalyzed cross-coupling reaction of sodium benzenesulfinate **1a** with hexabutyldistannane **2a** in the presence of Ag_2CO_3 in DMF at 60 °C. To our disappointment, only a trace amount of product **3a** could be detected (entry 1). Subsequently, the reaction temperature was raised and the yield improved to 44% (entry 2, 3). Various palladium catalysts were examined at 110 °C, and Pd $(P^tBu_3)_2$ was found as the best catalyst in this reaction (entry 3–6). Then we continued to heat up and tried different solvents by using $Pd(P^tBu_3)_2$ as the catalyst at 120 °C, and we observed that a satisfactory yield could be obtained by using the high boiling point solvent *N*,*N*-dimethylacetamide (DMA) (entry 7–10). Considering that high temperature was favorable for this desulfitative cross-coupling reaction, we carried out this reaction under 140 °C, and the yield significantly improved to 93% (entry 11). To our delight, the palladium catalyst loading could be reduced from 5 mol% to 3 mol% while the isolated yield increased to 97% (entry 12). Reducing the equivalent of **1a** and Ag_2CO_3 resulted in slightly diminished yields respectively (entry 13, 14). Moreover, control experiments showed that Ag_2CO_3 (entry 15) and $Pd(P^tBu_3)_2$ (entry 16) were essential for this cross-coupling reaction. After evaluating other commercially available oxidants, we have found that Ag_2CO_3 plays a crucial role in this reaction owing to the oxidizing potentials and solubility (For detail, see: Supporting Information, Table S1) [18f,19a,b,20].

With the optimized condition (Table 1, entry 12) in hand, we then proceeded to expand the substrate scope to various substituted sodium arylsulfinates (Scheme 2). Initially, a variety of mono-substituted sodium arylsulfinates were examined to obtain the corresponding arylstannane derivatives. This reaction by using **2a** as the stannylation reagent has shown excellent functional group tolerance to both electron-donating and electron-withdrawing substituents on the aromatic rings, such as alkyl (**3b**, **3c**), halogen (**3d**, **3e**, **3f**), vinyl (**3g**), cyano (**3h**), trifluoromethyl (**3i**), ethoxycarbonyl (**3j**), acetyl (**3k**) and trifluoromethoxy (**3l**), acetamino (**3m**), and azo groups (**3n**). In the cases of **3f** and **3g**, the low yields are due to the Pd-catalyzed stannylation of C–Br bond to generate bis-stannylated benzene derivative, or Heck reaction with vinyl moiety respectively. In addition to the *para*-substituted arylstannanes, *meta*- and *ortho*-substituted (**3o**–**3t**) and multi-substituted aryl stannane compounds (**3u**, **3v**) could also be got in moderate to good yields. Furthermore, naphthyl derivatives (**3w**, **3x**) and heterocyclic arylsulfinates (**3y**, **3z**, **3aa**) also showed good compatibility under the standard condition. The low yield in the case of **3y** is attributed to the instability on silica gel column chromatography. Moreover, other hexaalkyl distannanes (**2b**, **2c**) were also subjected to this coupling reaction, affording corresponding trialkyl arylstannanes (**3ba**, **3ca**) with moderate yields.

To manifest the practical application of this method, the stannylation reaction has been carried out on a gram scale for five arylsulfinates. As shown in Scheme 3, the corresponding arylstannanes products (**3a**, **3b**, **3h**, **3l**, **3q**) were successfully obtained with comparable good yields.

Since purification of aryl stannane compounds is tedious and some aryl stannanes are not stable on the silica gel column chromatography, we continuously perform the consecutive Pd-catalyzed Stille cross-coupling reaction without the purification of crude stannylation products. The desulfitative stannylation and Pd-catalyzed Stille reaction is carried out in a tandem manner. After the accomplishment of the stannylation, the reaction system was filtered to remove the insoluble precipitates, followed by removing the DMA solvent under reduced pressure. Subsequently, the crude arylstannanes was then subjected to the Stille reaction to afford the diaryl products in high yields (Scheme 4).

Considering about that this reaction is carried out under 140 °C, it is presumed to proceed through radical mechanism. To gain insights into the reaction mechanism, we have carried out the stannylation reaction in the presence of radical scavenger TEMPO

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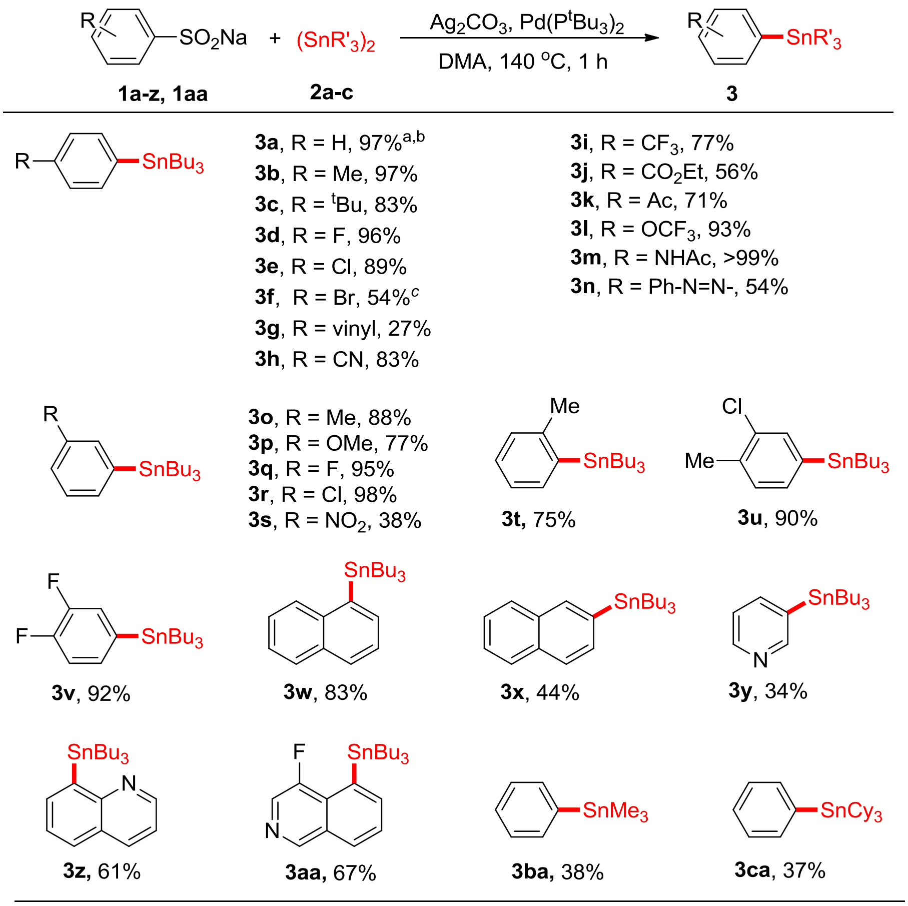
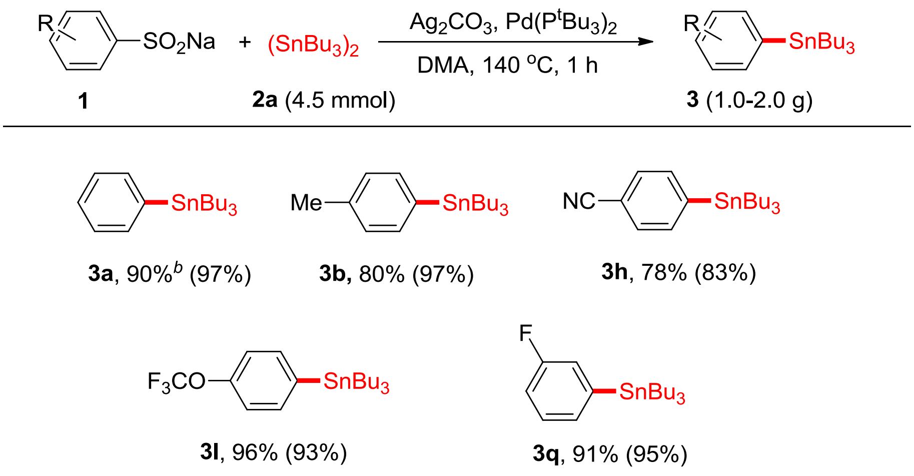


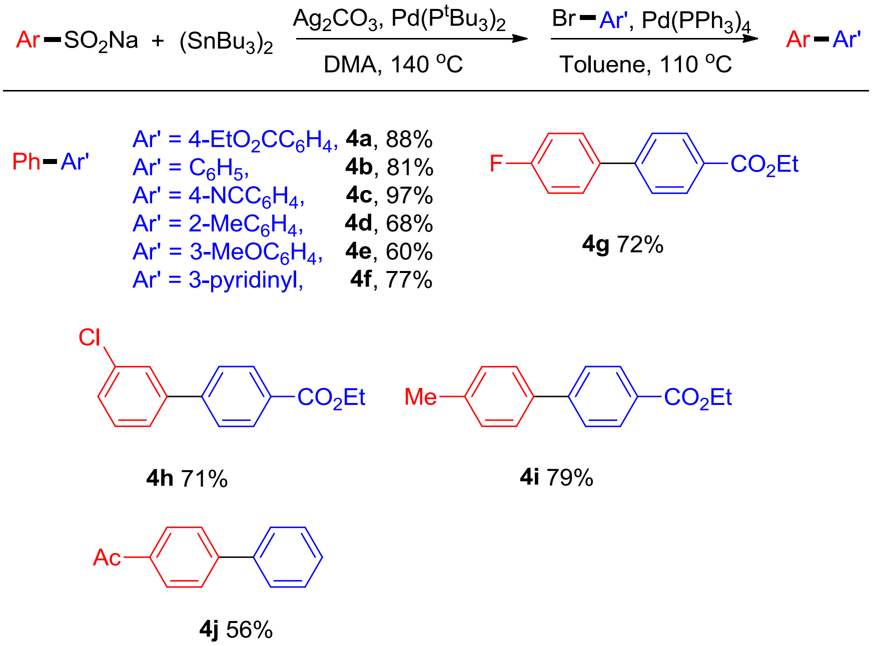
[a]Reaction conditions: sodium aryl sulfinate (0.3 mmol, 1.5 equiv), hexabutyl distannane (0.2 mmol, 1.0 equiv), Ag_2CO_3 (0.3 mmol, 1.5 equiv), $Pd(P^tBu_3)_2$ (0.006 mol, 3 mol %), DMA (2 mL), 140 °C, 1 h. [b]Isolated yields. [c]Sodium 4-bromobenzenesulfinate **1f** (0.34 mmol, 1.7 equiv).

Scheme 2. Scope of substrates.



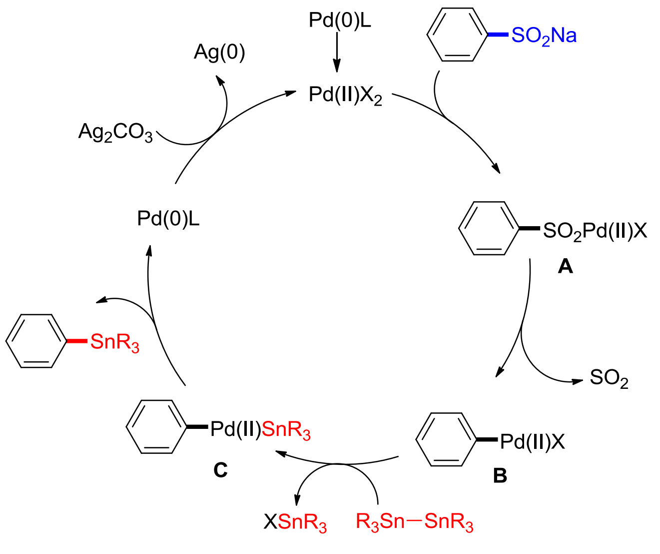
[a]Reaction conditions: sodium aryl sulfinate (6.75 mmol, 1.5 equiv), hexabutyldistannane **2a** (4.5 mmol, 1.0 equiv), Ag_2CO_3 (6.75 mmol, 1.5 equiv), $Pd(P^tBu_3)_2$ (0.135 mol, 3 mol %), DMA (30 mL), 140 °C, 1 h. solated yields. The figure in brackets refers to the yields with 0.2 mmol scale under the previous conditions.

Scheme 3. Gram-scale synthetic procedure.[a]



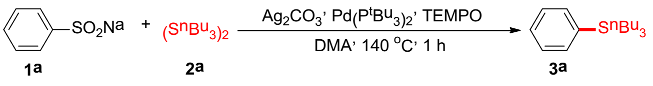
[a]Reaction conditions for the first step: sodium benzenesulfinate (0.3 mmol, 1.5 equiv), Ag_2CO_3 (0.3 mmol, 1.5 equiv), $Pd(P^tBu_3)_2$ (0.006 mol, 3 mol %), hexabutyldistannane (0.2 mmol, 1.0 equiv), DMA (2 mL), 140 °C, 1 h; second step: $Pd(PPh_3)_4$ (5 mol%), toluene (2 mL), Ar'Br (0.2 mmol, 1.0 equiv), 110 °C, 8 h. Isolated yields.

Scheme 4. Sequential stannylation and Pd-catalyzed Stille cross coupling reactions.[a]



Scheme 5. Possible reaction mechanism.

(2,2,6,6-tetramethylpiperidine-1-oxyl). As shown in Eq. (1), 10 mol %, 50 mol% and 1 equiv of TEMPO were respectively added under the standard conditions. In all the cases, the product **3a** was also obtained in good yields. This experiment indicated that this cross-coupling was not proceeding through the radical pathway.



TEMPO	0 mol %	10 mol %	50 mol %	1 equiv
3a	97%	92%	91%	86%

(1)

Based on our investigation and the literature precedents, a possible mechanism was proposed to account for this Pd-catalyzed cross-coupling reaction. As shown in Scheme 5, the catalytic cycle starts with the oxidation of Pd(0) catalyst by using Ag_2CO_3 to form Pd(II) species, which then reacts with sodium arylsulfinate, providing palladium salt **A**. Elimination of SO_2 from **A** generates aryl palladium intermediate **B**, followed by the transmetallation with ditin reagent to give rise to the intermediate **C**. Subsequently, reductive elimination from **C** produces desired arylstannane product along with the generation of Pd(0) species. Finally, Pd(0) is oxidized by Ag(I) to regenerate the catalytically active Pd(II) complex.

Conclusion

In conclusion, we have developed a new synthetic method towards trialkyl arylstannanes through Pd-catalyzed desulfitative cross-coupling reaction of sodium aryl sulfinates with distannanes. The reaction features high efficiency with low catalyst loading, good functional groups tolerance, affording stannylation products with moderate to excellent yields. Besides, the starting materials are easily prepared from the corresponding aryl sulfonyl chlorides. Because of the importance of arylstannane compounds in Stille cross-coupling reactions, we expect that this stannylation method will demonstrate potential applications in organic synthesis. Further expansion of the substrate scope as well as the detailed mechanistic study is currently underway in our laboratory.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2018.09.065.

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