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Synthesis of Benzyltributylstannanes by the Reaction of *N*-Tosylhydrazones with Bu_3SnH

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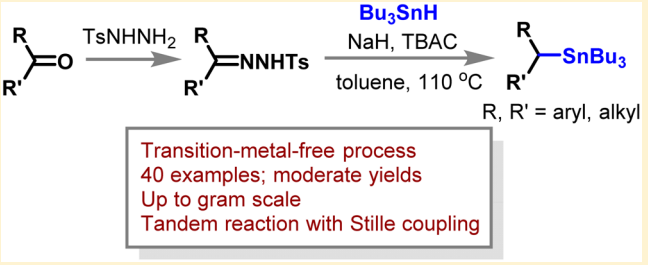
Supporting Information

ABSTRACT: An efficient stannylation process with *N*-tosylhydrazones or directly with carbonyl compounds has been developed. A series of functionalized benzyl- and alkyltributylstannanes can be synthesized in moderate to good yields under transition-metal-free conditions. Tandem transformations involving stannylation/Stille cross-coupling reaction have been carried out without purification of the benzyltributylstannane intermediates to afford a series of diarylmethane derivatives.



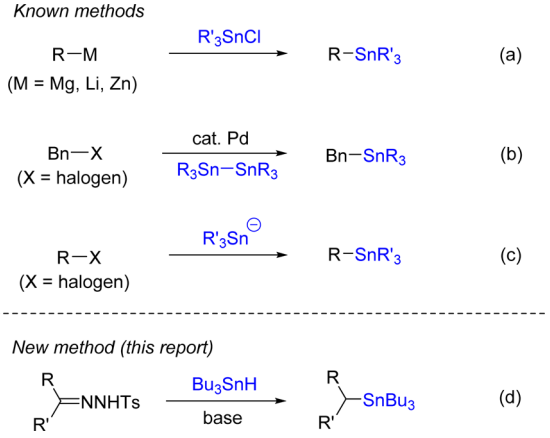
INTRODUCTION

Organotin reagents have found many applications in various fields.[1] In particular, organotin reagents have been widely applied in cross-coupling reactions (Stille coupling).[2] In addition to the commonly used arylstannane reagents, the corresponding alkylstannane reagents (in particular, the benzylstannane reagents) have also been extensively explored as important building blocks for C−C bond formation.[2b,3] Because of their importance, the development of synthetic methods for alkylstannane reagents has attracted attention over the years; however, the methods for accessing these reagents are still limited.

The general method for synthesizing alkylstannanes involves the reaction between alkyl Grignard reagents and lithium or zinc reagents with trialkyltin chloride (Scheme 1a).[4] Although widely applied, this traditional strategy requires rigorous reaction conditions, and thus, functional group tolerance is poor. More recently, palladium-catalyzed stannylation of benzyl or alkyl halides with distannanes (R_3SnSnR_3) has been developed (Scheme 1b).[5] The direct substitution reaction of alkyl halides with trialkylstannyl anion represents a straightforward approach (Scheme 1c).[6] In addition, it was reported that the reaction of diazo compounds with Bu_3SnH gave alkylstannane products through carbene insertion, but the studies were mainly focused on mechanistic aspects of the reaction.[7] Tributyltin radical addition to alkenes has also been reported.[8] Although various stannylation methods have been established, it is still highly desirable to develop efficient alternative methods for the synthesis of alkylstannane reagents.

N-Tosylhydrazones, which can be conveniently prepared from the corresponding ketones and aldehydes, have played an important role as versatile intermediates in organic synthesis. In particular, nonstabilized diazo compounds, which do not bear electron-withdrawing substituents on the diazo carbon, can be generated in situ from *N*-tosylhydrazones in the presence of base (Bamford−Stevens reaction).[9] The in situ generated diazo compounds can undergo various transformations, particularly the transition-metal-catalyzed carbene-transfer reactions and cross-coupling reactions.[10] In addition, transition-metal-free transformations of *N*-tosylhydrazones have also been shown in recent studies to form C−C,[11] C−O,[12] C−N[13] and C−S[14] bonds. We have previously reported the transition-metal-free formation of C−B bond from *N*-tosylhydrazones.[15] In connection to our interests on the transformations of *N*-tosylhydrazones, we report herein the conversion of *N*-

Scheme 1. Synthetic Methods for Alkylstannanes



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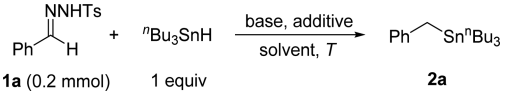
tosylhydrazones into benzylstannanes. The reaction represents a new general access to alkylstannane reagents (Scheme 1d).

RESULTS AND DISCUSSION

At the outset, the commercially available tributyltin hydride was chosen as the stannylating reagent and *N*-tosylhydrazone **1a** as the substrate. The initial reaction in the presence of ^{t}BuOLi in toluene at 90 °C afforded the desired benzyltributylstannane **2a** in 29% yield (Table 1, entry 1). We noted that the major

Table 1. Optimization of Reaction Conditions[a]



entry	solvent	T (°C)	base (equiv)	additive (mol %)	2a, yield[b] (%)
1	toluene	90	^{t}BuOLi (1.1)		29
2	MeCN	90	^{t}BuOLi (1.1)		trace
3	toluene	60	^{t}BuOLi (1.1)		trace
4	toluene	90	MeONa (1.1)		17
5	toluene	90	K_2CO_3 (1.1)		<10
6	toluene	90	NaH (1.1)		38
7	toluene	90	NaH (1.1)	TBAB (10)	53
8	toluene	90	NaH (1.1)	BTBAC (10)	46
9	toluene	90	NaH (1.5)	TBAB (10)	37
10	toluene	90	NaH (2.0)	TBAB (10)	20
11	toluene	90	NaH (1.1)	TBAC (10)	68
12	toluene	90	NaH (1.1)	TBAC (20)	74
13	toluene	110	NaH (1.1)	TBAC (20)	79

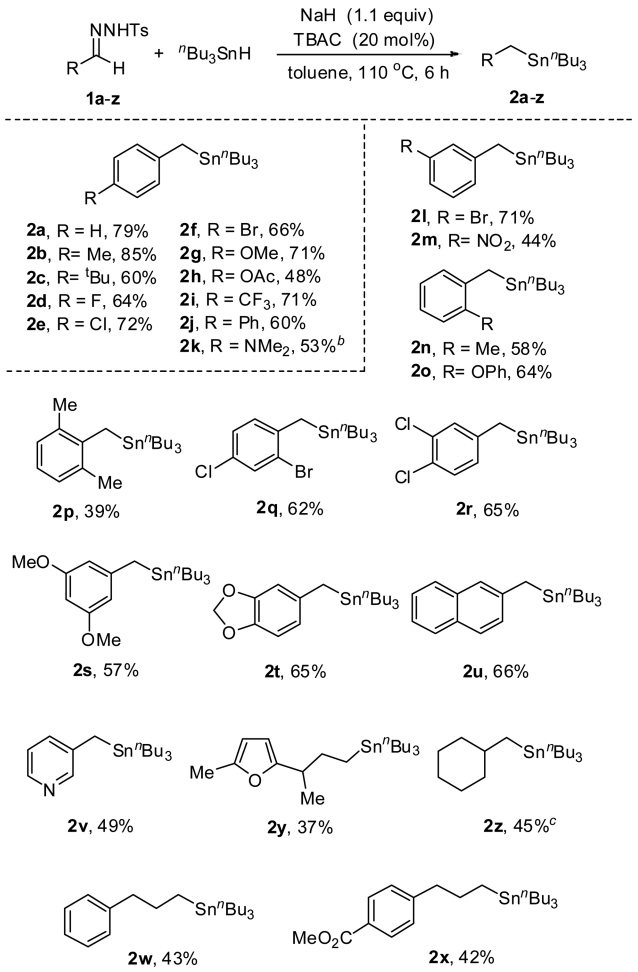
[a]Reaction conditions: *N*-tosylhydrazone (0.2 mmol), ^{n}Bu$_3$SnH (0.2 mmol), solvent (3 mL), 6 h. [b]Yields refer to the purified products by silica gel column chromatography. TBAB: tetrabutylammonium bromide. BTBAC: benzyltributylammonium chloride. TBAC: tetrabutylammonium chloride.

byproducts were $Bu_3Sn-SnBu_3$ and diphenylethene. When the reaction was carried out at 60 °C, the reaction gave only a trace amount of the product **2a** (entry 3). Switching the solvent to MeCN also resulted in only a trace amount of the product (entry 2). Subsequently, we proceeded to screen the effect of base. With MeONa or K_2CO_3 as the base, the reaction gave diminished yields (entries 4 and 5). Gratifyingly, with NaH as the base, the reaction afforded an improved result (entry 6).

In our previous studies, we noted that the phase-transfer catalyst (PTC) could enhance the solubility and thus significantly accelerate the transformation of *N*-tosylhydrazones to the diazo intermediates. We have thus examined a series of cationic PTCs in the current reaction system (entries 7−13). Indeed, the yield of **2a** was enhanced significantly when 10% tetrabutylammonium bromide (TBAB) or benzyltributylammonium chloride (BTBAC) was added (entries 7 and 8). With TBAB as the additive, increasing the loading of NaH resulted in diminished yields (entries 9 and 10). After subsequently examining the reaction conditions, we found that tetrabutylammonium chloride (TBAC) could provide the optimal result (entry 11). The yield could be further improved by increasing the reaction temperature and the TBAC loading (entries 12 and 13).

With the optimized reaction conditions, we proceeded to examine the scope of this transformation. First, a series of *N*-tosylhydrazones derived from the corresponding aldehydes were examined (Scheme 2). The reaction proceeded smoothly,

Scheme 2. Scope of *N*-Tosylhydrazones Derived from Aldehydes[a]



[a]Reaction conditions: *N*-tosylhydrazone **1a−z** (0.2 mmol), ^{n}Bu$_3$SnH (0.2 mmol), NaH (50 wt %, 0.22 mmol), TBAC (0.04 mmol), toluene (3 mL), 110 °C, 6 h. Yield of product with silica gel column chromatography. [b]The product was decomposed on silica gel column chromatography. The yield was determined by ^{1}H NMR spectroscopy with mesitylene as the internal standard. [c]The product was contaminated by $Bu_3SnSnBu_3$, which could not be removed by silica gel column chromatography. The yield was determined by ^{1}H NMR spectroscopy.

and a series of benzyl- and alkyltributylstannanes could be obtained in moderate yields. For the *N*-tosylhydrazones derived from the benzaldehydes bearing various substituents on the aromatic ring, this strategy has shown good functional tolerance for both electron-withdrawing and electron-donating substituents, which include alkyl (**2b,c**), phenyl (**2j**), halogen (**2d−f,l,q,r**), methoxy (**2g,s**), acetoxy (**2h**), trifluoromethyl (**2i**), nitro (**2m**), and *N,N*-dimethylamino groups (**2k**).

For *ortho*-substituted *N*-tosylhydrazones, the reaction afforded diminished yields (**2n** and **2p**), presumably due to steric hindrance. With the *N*-tosylhydrazones derived from piperonal (**2t**), naphthyl (**2u**), or pyridinyl (**2v**) aldehydes, the corresponding stannylation products were isolated in moderate yields. Furthermore, the reactions with *N*-tosylhydrazones derived from alkyl aldehydes were examined. The corresponding alkyltributylstannanes could also be obtained under the same reaction conditions, but the yields were diminished (**2w−z**).

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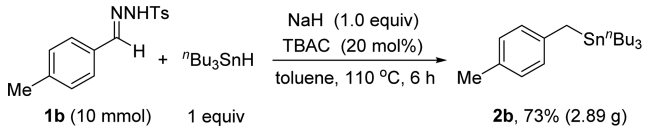
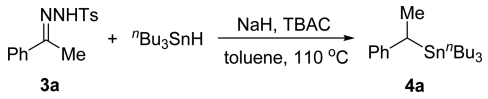


(1)

To demonstrate the practical usefulness of this transformation, the reaction has been carried out with 10 mmol of *N*-tosylhydrazone **1b**. As shown in eq 1, 2.89 g of the corresponding tolyltributylstannane **2b** could be isolated (73% yield).

Subsequently, we studied the reaction with *N*-tosylhydrazone derived from acetophenone (Table 2). To our disappointment,

Table 2. Optimization of the Reaction with *N*-Tosylhydrazone Derived from Acetophenone[a,b]



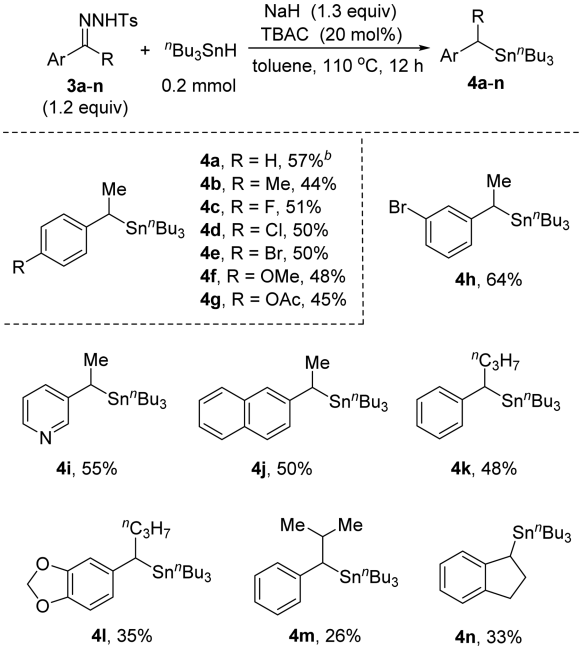
entry	3a/Bu_3SnH/NaH	TBAC (mol %)	4a, yield (%)
1	1:1:1.1	20	41
2	1:1:1.1	10	35
3	1:1:1.1	40	27
4	1.2:1:1.3	20	57
5	1.5:1:1.6	20	46
6	1:1.5:1.1	20	47

[a]Reaction conditions: substrates on 0.2 mmol scale, toluene (3 mL), 110 °C, 12 h. [b]Yields refer to the products purified by silica gel column chromatography. TBAC: tetrabutylammonium chloride.

we could only obtain a low yield of the product **4a** (entry 1). The byproducts formed from *N*-tosylhydrazones via [1,2]-H shift and dimerization were observed. It becomes apparent that the *N*-tosylhydrazones of ketones have low reactivity toward Bu_3SnH. To improve the reaction, modification of the previous conditions was then carried out. It was found that changing the loading of PTC could not improve the reaction (entries 2 and 3). Then the ratio of the two reactants and base was adjusted (entries 4–6). We found that with increased loading of *N*-tosylhydrazone the reaction could afford a slightly improved result (entry 4).

With the adjusted reaction conditions, a series of *N*-tosylhydrazones derived from the corresponding ketones were then examined (Scheme 3). The *N*-tosylhydrazones bearing various substituents on the aromatic ring, such as methyl (**3b**), halogen (**3c–e,h**), methoxy and acetoxy (**3f,g**), pyridinyl (**3i**), and naphthyl (**3j**) ketone derivatives were subjected to this transformation. The reaction afforded the stannylation products in low to moderate yields. When the R group was methyl, this reaction gave the secondary alkyl tributylstannanes in moderate yields in most cases. However, when *N*-tosylhydrazones bearing

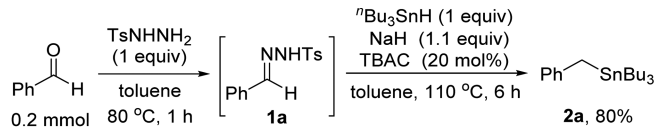
Scheme 3. Scope of *N*-Tosylhydrazones Derived from Ketones[a]



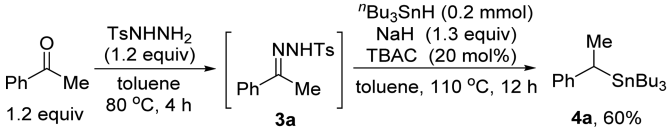
[a]Reaction conditions: *N*-tosylhydrazone (0.24 mmol), $^nBu_3SnH$ (0.2 mmol), NaH (50 wt %, 0.26 mmol), TBAC (0.04 mmol), toluene (3 mL), 110 °C, 12 h. [b]All of the yields refer to the products purified by silica gel column chromatography.

steric bulky groups were tested, the corresponding reaction gave diminished yields (**4k–m**). In the case of **4n**, the lower yield was attributed to the formation of indene as the side product. In general, the low yields are partially attributed to the fact that the stannylation products have low stability upon silica gel column chromatography due to the easy protonation. However, it is worth mentioning that most of these products are difficult to synthesize with traditional methods.

Since *N*-tosylhydrazones are generated with high efficiency from the reaction of *N*-tosylhydrazine ($TsNHNH_2$) with the corresponding aldehydes or ketones, we then proceeded to carry out the one-pot synthesis of benzyltributylstannanes from the carbonyl compounds without purification of the *N*-tosylhydrazones intermediates. To our delight, the one-pot transformation of both aldehydes and ketones afforded the stannylation products in good yields (eqs 2 and 3). The small amount of water generated from the reaction between carbonyl



(2)



(3)

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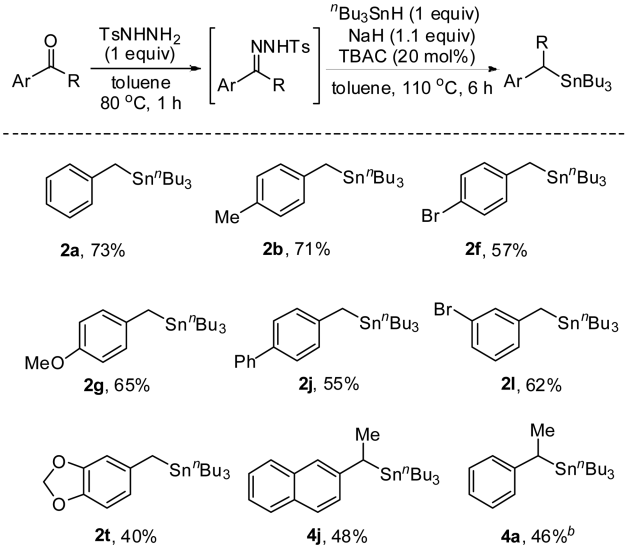
J. Org. Chem. 2017, 82, 624–632

compounds and $TsNHNH_2$ did not have a negative effect on this transformation.

Encouraged by the results, a series of carbonyl substrates were then subjected to the one-pot transformation in 0.5 g scale (Scheme 4). In most cases, the carbonyl substrates could be

Scheme 4. One-Pot Synthesis of Benzyltributylstannanes from Aldehydes or Ketones[a]



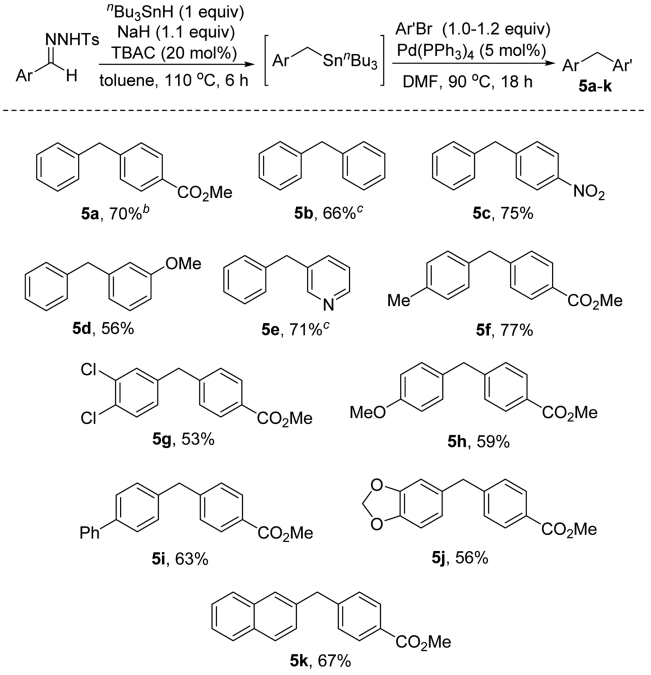
[a]Reaction conditions for **2a–t**: (1) aldehyde (2 mmol), $TsNHNH_2$ (2 mmol), toluene (10 mL), 80 °C, 1 h; (2) $^nBu_3SnH$ (2 mmol), NaH (50 wt %, 2.2 mmol), TBAC (0.4 mmol), toluene (15 mL), 110 °C, 6 h. Reaction conditions for **4a,j**: (1) ketones (2.4 mmol), $TsNHNH_2$ (2.4 mmol), toluene (10 mL), 80 °C, 4 h; (2) $^nBu_3SnH$ (2 mmol), NaH (50 wt %, 2.6 mmol), TBAC (0.4 mmol), toluene (15 mL), 110 °C, 12 h. Yields refer to the products purified by silica gel column chromatography. [b]TBAC (0.6 mmol).

transformed to the corresponding benzylstannanes in moderate yields. For the reaction with ketone substrates, longer reaction time is required both for the formation of *N*-tosylhydrazones and the stannylation process.

Since the purification of tin reagents is tedious and the stannylation products are generally not stable on silica gel, we then explored the tandem transformation of direct Pd-catalyzed cross-coupling of the benzyltributylstannane products without purification. Namely, the stannylation and Pd-catalyzed Stille reaction with aryl bromides is carried out in a tandem manner. As shown in Scheme 5, upon stannylation of *N*-tosylhydrazone, the reaction mixture was filtered to remove the precipitates followed by the removal of solvents under reduced pressure. Subsequently, the crude benzyltributylstannane product was subjected to a Pd-catalyzed Stille reaction to afford the diarylmethane product. A series of diarylmethanes can be accessed in moderately high yields with this two-step tandem transformation.

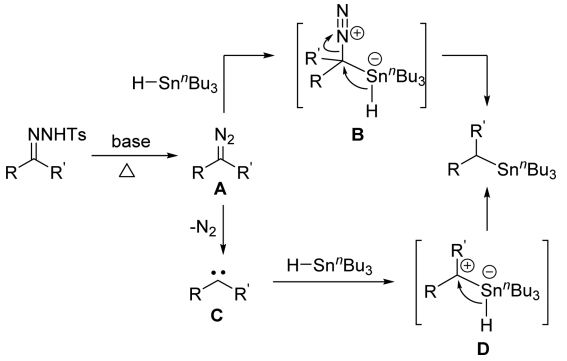
The reaction mechanism is proposed in the Scheme 6. The diazo intermediate **A** is first generated in situ from *N*-tosylhydrazone in the presence of base. The nucleophilic diazo carbon atom interacts with tributyltin hydride to form the tin anion complex **B**, followed by [1,2]-H migration to afford the product with the extrusion of nitrogen gas. An alternative pathway involves formation of carbene intermediate **C** followed by the formation of zwitterionic intermediate **D** and [1,2]-H migration to complete the formal carbene insertion into the

Scheme 5. Tandem Stannylation and Pd-Catalyzed Stille Cross-Coupling Reactions for the Synthesis of Diarylmethanes[a]



[a]Reaction conditions: (1) *N*-tosylhydrazone (0.2 mmol), $^nBu_3SnH$ (0.2 mmol), NaH (50 wt %, 0.22 mmol), TBAC (0.04 mmol), toluene (3 mL), 110 °C, 6 h; (2) Ar'Br (0.2 mmol), $Pd(PPh_3)_4$ (0.01 mmol), DMF (3 mL), 90 °C, 18 h. [b]All the yields refer to the products purified by silica gel column chromatography. [c]Ar'Br (0.22 mmol).

Scheme 6. Proposed Reaction Mechanism



Sn–H bond.[7,16] With the currently available experimental evidence it is not possible to distinguish these two mechanisms. To gain insights into the mechanistic details of this stannylation process, the reaction of **1b** was carried out in the presence of 10 equiv of 1-methoxy-4-vinylbenzene in order to test whether a free carbene intermediate was involved. Under such conditions, the stannylation process was almost blocked completely, and the corresponding cyclopropane product was obtained in 75% yield as a mixture of *cis*- and *trans*-isomers (Scheme 7). The result indicates that an in situ generated free carbene intermediate is most likely to be involved in the stannylation transformation.

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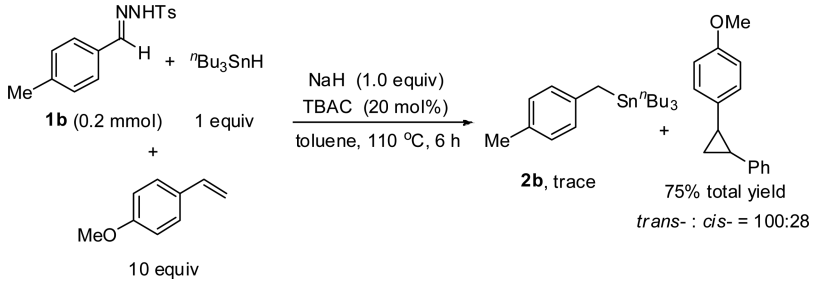
J. Org. Chem. 2017, 82, 624–632

Scheme 7. Stannylation Reaction in the Presence of Styrene



CONCLUSION

In summary, we have developed a transition-metal-free conversion of *N*-tosylhydrazones to benzyl- or alkyltributylstannanes, which are an important type of organometallic reagent widely utilized in cross-coupling reactions. We have also demonstrated a tandem transformation to convert the *N*-tosylhydrazones to diarylmethanes without purification of the stannylation products. The reaction further expands the synthetic applications of *N*-tosylhydrazones, which are easily available from the corresponding aldehydes or ketones.

EXPERIMENTAL SECTION

General Methods. Toluene was distilled from calcium hydride. For chromatography, 200−300 mesh silica gel was employed. Chemical shifts for ^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (100 MHz) are reported relative to the chemical shift of tetramethylsilane (TMS): chemical shifts (δ) were reported in ppm and coupling constants (J) in hertz (Hz). IR spectra are reported in wave numbers, cm^{-1}. For HRMS measurements, the mass analyzer is FT-ICR. Starting materials were obtained from commercial suppliers and were used without further purification.

Typical Procedure for the Stannylation of *N*-Tosylhydrazones Derived from Aldehydes (Scheme 2). Under nitrogen atmosphere, *N*-tosylhydrazone of benzaldehyde (0.2 mmol, 55 mg), NaH (1.1 equiv, 0.22 mmol, 50 wt %, 11 mg), and TBAC (20% equiv, 0.04 mmol, 11 mg) were weighed in a 10 mL Schlenk tube. Toluene (3 mL) was then added. The solution was stirred at room temperature for 10 min. Then ^{n}Bu$_3$SnH (1 equiv, 0.2 mmol, 58 mg, 54 μL) was added to the reaction system through a syringe. The resulting reaction solution was stirred at 110 °C for 6 h. The reaction mixture was then filtered through silica gel with petroleum ether as eluent. The combined filtrate was concentrated on a rotary evaporator under reduced pressure to leave a crude residue, which was purified by flash chromatography with silica gel (eluted with petroleum ether). Benzyltributylstannane **2a** was obtained as colorless liquid (60 mg, 79%).

Benzyltributylstannane ***2a***.[17] Flash chromatography (silica gel, petroleum ether) afforded **1a** (70 mg, 79%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.16 (t, J = 7.6 Hz, 2H), 6.99−6.95 (m, 3H), 2.30 (s, 2H), 1.44−1.39 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.4 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 143.7, 128.2, 126.9, 122.8, 29.0, 27.3, 18.1, 13.7, 9.3.

Tributyl(4-methylbenzyl)stannane ***2b***.[17] Flash chromatography (silica gel, petroleum ether) afforded **2b** (67 mg, 85%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 6.97 (d, J = 7.8 Hz, 2H), 6.87 (d, J = 7.8 Hz, 2H), 2.26 (s, 3H), 2.25 (s, 2H), 1.46−1.38 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.79 (t, J = 7.4 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 140.3, 132.0, 128.9, 126.9, 29.0, 27.3, 20.8, 17.5, 13.7, 9.2.

Tributyl(4-tert-butylbenzyl)stannane ***2c***. Flash chromatography (silica gel, petroleum ether) afforded **2c** (52 mg, 60%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.17 (d, J = 8.1 Hz, 2H), 6.91 (d, J = 8.1 Hz, 2H), 2.27 (s, 2H), 1.41−1.36 (m, 6H), 1.31−1.21 (m, 15H), 0.85 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 145.6, 140.3, 126.6, 125.1, 34.1, 31.4, 29.0, 27.3, 17.4, 13.7, 9.3; IR (film) 2957, 2929, 2872, 1506, 1464, 1215, 1191, 907,829, 734 cm^{-1}; EI-MS (m/z, relative intensity): 438 (M$^+$, 5), 381 (16), 291 (89), 267 (57), 235 (100), 179 (95), 147 (60); HRMS (EI) calcd for $C_{19}H_{33}{}^{120}Sn$ [M − C_4H_9]$^+$ 381.1599, found 381.1632.

Tributyl(4-fluorobenzyl)stannane ***2d***.[18] Flash chromatography (silica gel, petroleum ether) afforded **2d** (51 mg, 64%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 6.93−6.83 (m, 4H), 2.26 (s, 2H), 1.43−1.37 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 159.5 (d, J = 240 Hz), 139.2 (d, J = 2.9 Hz), 127.9 (d, J = 7.4 Hz), 114.9 (d, J = 20.9 Hz), 29.0, 27.3, 17.1, 13.7, 9.2.

Tributyl(4-chlorobenzyl)stannane ***2e***.[18] Flash chromatography (silica gel, petroleum ether) afforded **2e** (60 mg, 72%): colorless liquid. ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.12 (d, J = 8.4 Hz, 2H), 6.90 (d, J = 8.4 Hz, 2H), 2.26 (s, 2H), 1.43−1.38 (m, 6H), 1.28−1.22 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 142.4, 128.2, 128.1, 29.0, 27.3, 17.6, 13.6, 9.3.

(4-Bromobenzyl)tributylstannanes ***2f***.[17] Flash chromatography (silica gel, petroleum ether) afforded **2f** (61 mg, 66%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.26 (d, J = 8.3 Hz, 2H), 6.85 (d, J = 8.4 Hz, 2H), 2.24 (s, 2H), 1.43−1.39 (m, 6H), 1.29−1.22 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 142.9, 131.1, 128.6, 115.9, 29.0, 27.3, 17.7, 13.7, 9.3.

Tributyl(4-methoxybenzyl)stannane ***2g***.[19] Flash chromatography (silica gel, petroleum ether) afforded **2g** (58 mg, 71%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 6.90 (d, J = 8.5 Hz, 2H), 6.73 (d, J = 8.5 Hz, 2H), 3.76 (s, 3H), 2.24 (s, 2H), 1.44−1.38 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.79 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 155.8, 135.5, 127.8, 113.8, 55.3, 29.0, 27.3, 16.8, 13.7, 9.2.

4-((Tributylstannyl)methyl)phenyl Acetate ***2h***. Flash chromatography (silica gel, petroleum ether:ethyl acetate = 100:1, then 50:1) afforded **2h** (42 mg, 48%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 6.96 (d, J = 8.2 Hz, 2H), 6.88 (d, J = 8.3 Hz, 2H), 2.28 (s, 2H), 2.26 (s, 3H), 1.44−1.38 (m, 6H), 1.28−1.23 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.81 (t, J = 7.3 Hz, 6H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 169.7, 146.6, 141.3, 127.6, 121.1, 29.0, 27.3, 21.1, 17.5, 13.6, 9.3; IR (film) 2957, 2924, 1763, 1503, 1369, 1219, 1195, 908, 733 cm^{-1}. EI-MS (m/z, relative intensity):440 (M$^+$, 7), 383 (6), 291 (40), 235 (59), 179 (89), 107 (100); HRMS (EI) calcd for $C_{21}H_{36}O_2{}^{120}Sn$ [M]$^+$ 440.1732, found 440.1751.

Tributyl(4-(trifluoromethyl)benzyl)stannane ***2i***. Flash chromatography (silica gel, petroleum ether) afforded **2i** (64 mg, 71%): colorless liquid. ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.40 (d, J = 8.1 Hz, 2H), 7.05 (d, J = 8.1 Hz, 2H), 2.35 (s, 2H), 1.44−1.38 (m, 6H), 1.28−1.22 (m, 6H), 0.88−0.80 (m, 15H); ^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 148.6, 126.8, 125.2 (q, J = 3.7 Hz), 124.7 (q, J = 270 Hz), 29.0, 27.3, 18.7, 13.6, 9.4; IR (film) 2957, 2925, 2854, 1612, 1324, 1162, 1121, 1067 cm^{-1}; EI-MS (m/z, relative intensity) 393 ([M-57]$^+$, 11), 337 (6), 291 (74), 235 (75), 177 (100), 140 (58); HRMS (EI) calcd for $C_{16}H_{24}F_3{}^{120}Sn$ [M − C_4H_9]$^+$ 393.0847, found 393.0825.

(Biphenyl-4-ylmethyl)tributylstannanes ***2j***. Flash chromatography (silica gel, petroleum ether) afforded **2j** (55 mg, 60%): colorless liquid; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.56 (d, J = 7.8 Hz, 2H), 7.42−7.38

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J. Org. Chem. 2017, 82, 624−632

(m, 4H), 7.30−7.26 (m, 1H), 7.05 (d, J = 7.8 Hz, 2H), 2.34 (s, 2H), 1.47−1.40 (m, 6H), 1.29−1.24 (m, 6H), 0.88−0.81 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 143.0, 141.3, 135.7, 128.6, 127.3, 126.9, 126.7, 126.5, 29.0, 27.3, 17.9, 13.7, 9.4; IR (film) 2956, 2921, 2854, 1609, 1486, 1212, 1075, 907, 732 cm^{-1}; EI-MS (m/z, relative intensity): 458 (M^+, 2), 401 (9), 291 (51), 235 (43), 179 (48), 167 (100), 121 (15); HRMS (EI) calcd for $C_{25}H_{38}{}^{120}Sn$ $[M]^+$ 458.1990, found 458.1991.

N,N-Dimethyl-4-((tributylstannyl)methyl)aniline **2k**. Decomposed on silica gel chromatography. The yield was estimated by ^1H NMR spectroscopy with mesitylene (0.2 mmol) as internal standard (mixture with toluene and $Bu_3SnSnBu_3$): ^1H NMR (400 MHz, $CDCl_3$) δ 6.87 (d, J = 8.5 Hz, 2H), 6.63 (d, J = 8.5 Hz, 2H), 2.84 (s, 6H), 2.22 (s, 2H), 1.44−1.38 (m, 6H), 1.29−1.24 (m, 6H), 0.88−0.81 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 147.3, 131.9, 127.6, 113.8, 41.3, 29.0, 27.3, 16.4, 13.6, 9.2; EI-MS (m/z, relative intensity) 425 (M^+, 8), 291 (3), 135 (6), 179 (8), 134 (100), 118 (9), 91 (3); HRMS (EI) calcd for $C_{21}H_{39}N^{120}Sn$ $[M]^+$ 425.2099, found 425.2120.

(3-Bromobenzyl)tributylstannanes **2l**. Flash chromatography (silica gel, petroleum ether) afforded **2l** (65 mg, 71%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.13 (s, 1H), 7.09 (d, J = 7.9 Hz, 1H), 7.01 (t, J = 7.7 Hz, 1H), 6.88 (d, J = 7.7 Hz, 1H), 2.26 (s, 2H), 1.45−1.38 (m, 6H), 1.29−1.23 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.82 (t, J = 7.3 Hz, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 146.5, 129.7, 125.8, 125.4, 122.4, 29.0, 27.3, 18.1, 13.7, 9.4; IR (film) 2957, 2922, 2849, 1593, 1414, 1208, 1070, 734 cm^{-1}. EI-MS (m/z, relative intensity) 403 $[(M - 57)^+, 10]$, 347 (5), 291 (100), 235 (89), 177 (93), 121 (26); HRMS (EI) calcd for $C_{15}H_{24}{}^{79}Br^{120}Sn$ $[M - C_4H_9]^+$ 403.0078, found 403.0079.

Tributyl(3-nitrobenzyl)stannane **2m**.[19] Flash chromatography (silica gel, petroleum ether/ethyl acetate =100:1) afforded **2m** (37 mg, 44%): yellow liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83−7.81 (m, 2H), 7.34−7.26 (m, 2H), 2.40 (s, 2H), 1.44−1.38 (m, 6H), 1.28−1.23 (m, 6H), 0.88−0.82 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 148.4, 146.5, 132.9, 129.0, 121.2, 117.9, 28.9, 27.2, 18.5, 13.6, 9.5.

Tributyl(2-methylbenzyl)stannane **2n**.[17] Flash chromatography (silica gel, petroleum ether) afforded **2n** (46 mg, 58%): colorless liquid: ^1H NMR (400 MHz, $CDCl_3$) δ 7.06−7.00 (m, 2H), 6.96−6.95 (m, 1H), 6.89 (dt, J = 1.2, 7.3 Hz, 1H), 2.27 (s, 2H),2.19 (s, 3H), 1.44−1.37 (m, 6H), 1.29−1.22 (m, 6H), 0.85 (t, J = 7.3 Hz, 9H), 0.81−0.77 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 142.1, 133.2, 129.8, 127.4, 125.9, 123.1, 29.0, 27.3, 20.2, 16.1, 13.6, 9.7.

Tributyl(2-phenoxybenzyl)stannane **2o**. Flash chromatography (silica gel, petroleum ether, then petroleum ether:ethyl acetate = 200:1) afforded **2o** (61 mg, 64%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, J = 7.9 Hz, 2H), 7.10−7.08 (m, 1H), 7.03 (t, J = 7.3 Hz, 1H), 6.98−6.94 (m, 2H), 6.91 (d, J = 8.3 Hz, 2H), 6.82−6.80 (m, 1H), 2.23 (s, 2H), 1.44−1.38 (m, 6H), 1.26−1.20 (m, 6H), 0.85−0.78 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 157.7, 152.4, 135.8, 129.6, 129.2, 124.2, 123.8, 122.2, 119.1, 117.5, 29.0, 27.3, 13.7, 12.7, 9.8; IR (film) 2955, 2924, 2871, 1589, 1485, 1237, 907, 879, 734 cm^{-1}; EI-MS (m/z, relative intensity): 417 ($[M - 57]^+$, 100), 361 (6), 303 (11), 235 (14), 179 (23), 155 (9); HRMS (EI) calcd for $C_{21}H_{29}O^{120}Sn$ $[M - C_4H_9]^+$4 17.1235, found 417.1245.

Tributyl(2,6-dimethylbenzyl)stannane **2p**. Flash chromatography (silica gel, petroleum ether) afforded **2p** (32 mg, 39%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 6.93 (d, J = 7.4 Hz, 2H), 6.82 (t, J = 7.4 Hz, 1H), 2.25 (s, 2H),2.20 (s, 6H), 1.42−1.34 (m, 6H), 1.28−1.22 (m, 6H), 0.84 (t, J = 7.1 Hz, 9H), 0.80−0.76 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 140.5, 133.2, 127.6, 122.7, 29.0, 27.3, 20.9, 13.6, 13.4, 10.4; IR (film) 2957, 2927, 2871, 1464, 1477, 1096, 907, 733 cm^{-1}; EI-MS (m/z, relative intensity) 353 $[(M - 57)^+, 4]$, 291 (32), 235 (48), 177 (52), 119 (100), 91 (19); HRMS (EI) calcd for $C_{17}H_{29}{}^{120}Sn$ $[M - C_4H_9]^+$ 353.1286, found 353.1306.

(2-Bromo-4-chlorobenzyl)tributylstannanes **2q**. Flash chromatography (silica gel, petroleum ether) afforded **2q** (61 mg, 62%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 2.1 Hz, 1H), 7.09 (dd, J = 2.1, 8.3 Hz, 1H), 6.98 (d, J = 8.3 Hz, 1H), 2.41 (s, 2H), 1.46−1.38 (m, 6H), 1.30−1.23 (m, 6H), 0.88−0.82 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 142.8, 131.7, 128.9, 128.6, 127.4, 122.4, 28.9, 27.3, 19.7, 13.6, 10.2; IR (film) 2956, 2928, 2853, 1466, 1376, 1260, 1108, 1033, 907, 733 cm^{-1}; EI-MS (m/z, relative intensity) 437 $[(M - 57)^+, 72]$, 381 (21), 323 (17), 291 (86), 235 (93), 177 (100); HRMS (EI) calcd for $C_{15}H_{23}{}^{35}Cl^{79}Br^{120}Sn$ $[M - C_4H_9]^+$ 436.9688, found 436.9656.

Tributyl(3,4-dichlorobenzyl)stannane **2r**.[20] Flash chromatography (silica gel, petroleum ether) afforded **2r** (59 mg, 65%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, J = 8.3 Hz, 1H), 7.06 (d, J = 2.0 Hz, 1H), 6.79 (dd, J = 2.0, 8.3 Hz, 1H), 2.23 (s, 2H), 1.44−1.38 (m, 6H), 1.29−1.23 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.84−0.80 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 144.6, 131.9, 130.0, 128.4, 126.3, 29.0, 27.3, 17.7, 13.6, 9.4.

Tributyl(3,5-dimethoxybenzyl)stannane **2s**.[21] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 100:1) afforded **2s** (50 mg, 57%): pale yellow liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 6.16 (d, J = 2.0 Hz, 2H), 6.12 (d, J = 2.0 Hz, 1H), 3.74 (s, 6H), 2.25 (s, 2H), 1.46−1.38 (m, 6H), 1.29−1.24 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.84−0.80 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 160.8, 146.2, 105.0, 95.4, 55.1, 29.1, 27.4, 18.8, 13.7, 9.5.

(Benzo[d][1,3]dioxol-5-ylmethyl)tributylstannanes **2t**.[22] Flash chromatography (silica gel, petroleum ether, then petroleum ether/ethyl acetate = 100:1) afforded **2t** (55 mg, 65%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 6.64 (d, J = 7.9 Hz, 1H), 6.49 (d, J = 1.5 Hz, 1H), 6.43 (dd, J = 1.5, 7.9 Hz, 1H), 5.87 (s, 2H), 2.23 (s, 2H), 1.46−1.38 (m, 6H), 1.29−1.24 (m, 6H), 0.87 (t, J = 7.3 Hz, 9H), 0.83−0.79 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 147.4, 143.4, 137.4, 119.1, 108.1, 107.6, 100.4, 29.0, 27.3, 17.8, 13.7, 9.2.

Tributyl(naphthalen-2-ylmethyl)stannane **2u**.[17] Flash chromatography (silica gel, petroleum ether) afforded **2u** (57 mg, 66%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 8.0 Hz, 1H), 7.65 (d, J = 8.2 Hz, 2H), 7.38−7.37 (m, 2H), 7.31 (d, J = 7.9 Hz, 1H), 7.14 (d, J = 8.4 Hz, 1H), 2.46 (s, 2H), 1.44−1.39 (m, 6H), 1.28−1.22 (m, 6H), 0.86−0.79 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 141.5, 134.0, 130.5, 127.6, 127.5, 127.2, 126.7, 125.7, 123.8, 123.5, 29.0, 27.3, 18.7, 13.6, 9.4.

3-((Tributylstannyl)methyl)pyridine **2v**.[23] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 50:1, then 20:1) afforded **2v** (37 mg, 49%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.30 (s, 1H), 8.23 (s, 1H), 7.28−7.26 (m, 1H), 7.09−7.06 (m, 1H), 2.24 (s, 2H), 1.44−1.38 (m, 6H), 1.29−1.23 (m, 6H), 0.89−0.81 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 148.4, 144.4, 139.6, 133.8, 123.0, 28.9, 27.2, 14.8, 13.6, 9.4.

Tributyl(3-phenylpropyl)stannane **2w**.[24] Flash chromatography (silica gel, petroleum ether) afforded **2w** (35 mg, 43%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.29−7.25 (m, 2H), 7.19−7.16 (m, 3H), 2.60 (t, J = 7.6 Hz, 2H), 1.83- 1.77 (m, 2H), 1.48−1.44 (m, 6H), 1.32−1.26 (m, 6H), 0.90−0.80 (m, 17H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 142.6, 128.4, 128.2, 125.6, 40.8, 29.25, 29.20, 27.4, 13.7, 8.84, 8.76.

Methyl 4-(3-(Tributylstannyl)propyl)benzoate **2x**. Flash chromatography (silica gel, petroleum ether/ethyl acetate = 100:1) afforded **2x** (39 mg, 42%): pale yellow liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.1 Hz, 2H), 7.23 (d, J = 8.1 Hz, 2H), 3.90 (s, 3H), 2.66 (t, J = 7.6 Hz, 2H), 1.83- 1.79 (m, 2H), 1.49−1.42 (m, 6H), 1.32−1.26 (m, 6H), 0.90−0.80 (m, 17H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 167.2, 148.1, 129.6, 128.5, 127.6, 51.9, 40.8, 29.2, 28.9, 27.4, 13.7, 8.8, 8.7; IR (film) 2956, 2926, 2853, 1724, 1610, 1437, 1279, 1111, 908 cm^{-1}; EI-MS (m/z, relative intensity) 411 ($[M - 57]^+$, 100), 355 (14), 295 (12), 235 (11), 177 (24), 145 (32), 118 (16); HRMS (ESI) calcd for $C_{23}H_{40}NaO_2{}^{120}Sn$ $[M + Na]^+$ 491.1946, found 491.1956.

Tributyl(3-(5-methylfuran-2-yl)butyl)stannane **2y**. Flash chromatography (silica gel, petroleum ether) afforded **2y** (31 mg, 37%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 5.84−5.82 (m, 2H), 2.66−2.60 (m, 1H), 2.25 (s, 3H), 1.84−1.80 (m, 1H), 1.65−1.60 (m, 1H), 1.48−1.42 (m, 6H), 1.32−1.26 (m, 6H), 1.20 (d, J = 6.9 Hz, 3H), 0.90−0.78 (m, 17H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 159.0, 149.9, 105.5, 104.0, 37.0, 33.0, 29.2, 27.4, 18.4, 13.7, 13.5, 8.6, 5.9; IR (film) 2956, 2924, 2870, 1463, 1222, 1019, 908, 735 cm^{-1}; EI-MS (m/z, relative intensity) 371 ($[M - 57]^+$, 100), 291 (17), 235

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(16), 177 (35), 121 (17), 109 (50); HRMS (EI) calcd for $C_{17}H_{31}O^{120}Sn$ $[M - C_4H_9]^+$ 371.1391, found 371.1411.

Tributyl(cyclohexylmethyl)stannane ***2z***. Flash chromatography (silica gel, petroleum ether) afforded **2z** (50 mg, **2z**/$Bu_3SnSnBu_3$ = 1:0.3; 45% yield estimated by ^1H NMR): colorless liquid; representative ^1H NMR chemicals shifts ^1H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.3 Hz, 9H), 0.80 (t, J = 7.4 Hz, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 37.6, 29.7, 29.3, 27.4, 26.7, 26.2, 18.9, 13.7, 9.4; $Bu_3SnSnBu_3$ ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.6, 27.5, 13.7, 10.0; HRMS (EI) calcd for $C_{15}H_{31}{}^{120}Sn$ $[M - C_4H_9]^+$ 331.1442, found 331.1453.

Typical Procedure for the Stannylation of *N*-Tosylhydrazones Derived from Ketones (Scheme 3). The reaction was carried out with $^nBu_3SnH$ (0.2 mmol), *N*-tosylhydrazones (1.2 equiv), and NaH (1.3 equiv). The reaction solution was stirred at 110 °C for 12 h. The workup procedures were the same as the typical procedure described previously for Scheme 2.

Tributyl(1-phenylethyl)stannane ***4a***.[25] Flash chromatography (silica gel, petroleum ether) afforded **4a** (45 mg, 57%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.6 Hz, 2H), 7.04−6.97 (m, 3H), 2.70 (q, J = 7.5 Hz, 1H), 1.57 (d, J = 7.5 Hz, 3H), 1.42−1.34 (m, 6H), 1.28−1.21 (m, 6H), 0.85 (t, J = 7.2 Hz, 9H), 0.79−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 149.0, 128.2, 125.5, 123.2, 29.0, 27.4, 26.8, 17.3, 13.6, 8.7.

Tributyl(1-p-tolylethyl)stannane ***4b***. Flash chromatography (silica gel, petroleum ether) afforded **4b** (36 mg, 44%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.02 (d, J = 7.9 Hz, 2H), 6.92(d, J = 7.9 Hz, 2H), 2.65 (q, J = 7.6 Hz, 1H), 2.28 (s, 3H), 1.55 (d, J = 7.6 Hz, 3H), 1.40−1.34 (m, 6H), 1.29−1.22 (m, 6H), 0.85 (t, J = 7.3 Hz, 9H), 0.78−0.74 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 145.8, 132.4, 128.8, 125.5, 29.1, 27.5, 26.3, 20.8, 17.6, 13.7, 8.7; IR (film) 2956, 2924, 2855, 1510, 1463, 1376, 907, 734 cm^{-1}; EI-MS (m/z, relative intensity) 410 (M^+, 1), 353 ($[M - 57]^+$, 5), 291 (46), 235 (55), 179 (67), 119 (100), 91 (22); HRMS (EI) calcd for $C_{17}H_{29}{}^{120}Sn$ $[M - C_4H_9]^+$ 353.1286, found 353.1294.

Tributyl(1-(4-fluorophenyl)ethyl)stannane ***4c***. Flash chromatography (silica gel, petroleum ether) afforded **4c** (42 mg, 51%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 6.98−6.94 (m, 2H), 6.92−6.88 (m, 2H), 2.66 (q, J = 7.6 Hz, 1H), 1.54 (d, J = 7.6 Hz, 3H), 1.41−1.35 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.79−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 159.7 (d, J = 241 Hz), 144.6 (d, J = 2.9 Hz), 126.5 (d, J = 7.5 Hz), 114.8 (d, J = 20.8 Hz), 29.0, 27.4, 25.8, 17.7, 13.6, 8.7; IR (film) 2956, 2927, 2871, 1505, 1463, 1228, 907, 831, 733 cm^{-1}; EI-MS (m/z, relative intensity): 357 $[(M - 57)^+, 7]$, 291 (53), 235 (66), 179 (100), 123 (89), 103 (30); HRMS (EI) calcd for $C_{16}H_{26}F^{120}Sn$ $[M - C_4H_9]^+$ 357.1035, found 357.1057.

Tributyl(1-(4-chlorophenyl)ethyl)stannane ***4d***. Flash chromatography (silica gel, petroleum ether) afforded **4d** (43 mg, 50%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 8.4 Hz, 2H), 6.95(d, J = 8.4 Hz, 2H), 2.66 (q, J = 7.5 Hz, 1H), 1.54 (d, J = 7.5 Hz, 3H), 1.40−1.34 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.79−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 147.6, 128.2, 126.7, 29.0, 27.4, 26.2, 17.3, 13.6, 8.7; IR (film) 2955, 2922, 2872, 1488, 1459, 1091, 1010, 907, 826, 734 cm^{-1}; EI-MS (m/z, relative intensity) 373 ($[M - 57]^+$, 9), 291 (65), 235 (76), 179 (100), 139 (43), 103 (35); HRMS (EI) calcd for $C_{16}H_{26}{}^{35}Cl^{120}Sn$ $[M-C_4H_9]^+$ 373.0740, found 373.0760.

(1-(4-Bromophenyl)ethyl)tributylstannanes ***4e***. Flash chromatography (silica gel, petroleum ether) afforded **4e** (48 mg, 50%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 8.4 Hz, 2H), 6.89 (d, J = 8.4 Hz, 2H), 2.65 (q, J = 7.6 Hz, 1H), 1.53 (d, J = 7.6 Hz, 3H), 1.40−1.34 (m, 6H), 1.27−1.21 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.80−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 148.2, 131.1, 127.2, 116.3, 29.0, 27.4, 26.3, 17.2, 13.6, 8.8; IR (film) 2956, 2930, 2853, 1488, 1463, 1007, 908, 822, 734 cm^{-1}; EI-MS (m/z, relative intensity) 417 $[(M - 57)^+,8]$, 291 (88), 235 (86), 179 (100), 121 (26), 104 (54); HRMS (EI) calcd for $C_{16}H_{26}{}^{79}Br^{120}Sn$ $[M - C_4H_9]^+$ 417.0234, found 417.0247.

Tributyl(1-(4-methoxyphenyl)ethyl)stannane ***4f***. Flash chromatography (silica gel, petroleum ether) afforded **4f** (41 mg, 48%): colorless liquid; ^1H NMR (500 MHz, $CDCl_3$) δ 6.95 (d, J = 8.6 Hz, 2H), 6.78(d, J = 8.6 Hz, 2H), 3.78 (s, 3H), 2.63 (q, J = 7.6 Hz, 1H), 1.54 (d, J = 7.6 Hz, 3H), 1.41−1.37 (m, 6H), 1.27−1.23 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.78−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ 156.1, 141.1, 126.4, 113.8, 55.3, 29.1, 27.4, 25.7, 17.9, 13.6, 8.7; IR (film) 2954, 2927, 2852, 1508, 1464, 1244, 1042, 908, 734 cm^{-1}; EI-MS (m/z, relative intensity) 426 (M^+, 6), 369 $[(M - 57)^+, 5]$, 291 (44), 235 (51), 179 (52), 135 (100); HRMS (EI) calcd for $C_{17}H_{29}O^{120}Sn$ $[M - C_4H_9]^+$ 369.1235, found 369.1236.

4-(1-(Tributylstannyl)ethyl)phenyl Acetate ***4g***. Flash chromatography (silica gel, petroleum ether:ethyl acetate = 100:1, then 50:1) afforded **4g** (41 mg, 45%): pale yellow liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.01 (d, J = 8.5 Hz, 2H), 6.92 (d, J = 8.5 Hz, 2H), 2.69 (q, J = 7.5 Hz, 1H), 2.27 (s, 3H), 1.55 (d, J = 7.5 Hz, 3H), 1.41−1.34 (m, 6H), 1.27−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.80−0.75 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 169.7, 146.8, 146.6, 126.1, 121.0, 29.0, 27.4, 26.1, 21.1, 17.5, 13.6, 8.7; IR (film)2957, 2917, 2850, 1766, 1506, 1191, 908, 733 cm^{-1}; EI-MS (m/z, relative intensity) 454 (M^+, 22), 397 $[(M - 57)^+, 9]$, 291 (43), 235 (64),179 (64), 121 (100); HRMS (EI) calcd for $C_{22}H_{38}O_2{}^{120}Sn$ $[M]^+$ 454.1888, found 454.1887.

(1-(3-Bromophenyl)ethyl)tributylstannanes ***4h***. Flash chromatography (silica gel, petroleum ether) afforded **4h** (61 mg, 64%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.17 (s, 1H), 7.11 (d, J = 8.1 Hz, 1H), 7.06 (t, J = 7.7 Hz, 1H), 6.93 (d, J = 7.5 Hz, 1H), 2.66 (q, J = 7.5 Hz, 1H), 1.54 (d, J = 7.5 Hz,3H), 1.40−1.34 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.81−0.76 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 151.7, 129.6, 128.3, 126.1, 124.1, 122.5, 29.0, 27.4, 26.7, 17.1, 13.6, 8.9; IR (film) 2956, 2926, 2870, 1589, 1464, 1072, 907, 733 cm^{-1}; EI-MS (m/z, relative intensity) 417 $[(M - 57)^+, 6]$, 291 (82), 235 (86), 179 (100), 120 (29), 104 (45); HRMS (EI) calcd for $C_{16}H_{26}{}^{79}Br^{120}Sn$ $[M - C_4H_9]^+$ 417.0234, found 417.0257.

3-(1-(Tributylstannyl)ethyl)pyridine ***4i***. Flash chromatography (silica gel, petroleum ether/ethyl acetate = 50:1, then 20:1) afforded **4i** (43 mg, 55%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, J = 1.6 Hz, 1H), 8.25 (d, J = 4.4 Hz, 1H), 7.32 (d, J = 7.6 Hz, 1H), 7.13 (dd, J = 4.8, 7.6 Hz, 1H), 2.67 (q, J = 7.5 Hz, 1H), 1.58 (d, J = 7.5 Hz, 3H), 1.41−1.35 (m, 6H), 1.28−1.22 (m, 6H), 0.86 (t, J = 7.3 Hz, 9H), 0.83−0.79 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 147.7, 144.7, 144.5, 132.2, 123.0, 29.0, 27.4, 23.7, 16.9, 13.6, 8.9; IR (film) 2956, 2925, 2871, 1463, 1418, 1377, 998, 908, 733 cm^{-1}; EI-MS (m/z, relative intensity) 397 (M^+, 41), 340 $[(M - 57)^+, 9]$, 291 (22), 235 (74), 179 (100), 121 (26); HRMS (EI) calcd for $C_{19}H_{35}N^{120}Sn$ $[M]^+$ 397.1786, found 397.1809.

Tributyl(1-(naphthalen-2-yl)ethyl)stannane ***4j***. Flash chromatography (silica gel, petroleum ether) afforded **4j** (44 mg, 50%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.75−7.66 (m, 3H), 7.41−7.39 (m, 2H), 7.34−7.32 (m, 1H), 7.20−7.18 (m, 1H), 2.86 (q, J = 7.5 Hz, 1H), 1.67(d, J = 7.5 Hz, 3H), 1.41−1.33 (m, 6H), 1.26−1.20 (m, 6H), 0.84−0.77 (m, 15H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 146.7, 134.0, 130.8, 127.5, 127.4, 127.0, 126.3, 125.7, 124.0, 121.7, 29.1, 27.4, 27.3, 17.3, 13.6, 8.9; IR (film) 2956, 2923, 2856, 1628, 1598, 1463, 1170, 907, 733 cm^{-1}. EI-MS (m/z, relative intensity): 446 (M^+, 1), 389 $[(M-57)^+, 2]$, 291 (33), 235 (38), 179 (49), 155 (100), 121 (17); HRMS (EI) calcd for $C_{24}H_{38}{}^{120}Sn$ $[M]^+$ 446.1990, found 446.2018.

Tributyl(1-phenylbutyl)stannane ***4k***. Flash chromatography (silica gel, petroleum ether) afforded **4k** (41 mg, 48%): colorless liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.18 (t, J = 7.6 Hz, 2H), 7.01−6.95 (m, 3H), 2.60 (dd, J = 6.2, 10.1 Hz, 1H), 2.09−2.00 (m, 1H), 1.87−1.78 (m, 1H), 1.38−1.32 (m, 6H), 1.28−1.21 (m, 8H), 0.89−0.83 (m, 12H), 0.77−0.73 (m, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 147.1, 128.2, 126.3, 123.2, 34.6, 34.1, 29.0, 27.4, 23.0, 13.9, 13.6, 9.0; IR (film) 2956, 2925, 2871, 1599, 1491, 1463, 1377, 908, 734 cm^{-1}; EI-MS (m/z, relative intensity) 424 (M^+, 1), 367 $[(M - 57)^+, 17]$, 291 (100), 235 (97), 179 (76), 91 (75); HRMS (EI) calcd for $C_{18}H_{31}{}^{120}Sn$ $[M - C_4H_9]^+$ 367.1442, found 367.1466.

(1-(Benzo[d][1,3]dioxol-5-yl)butyl)tributylstannanes ***4l***. Flash chromatography (silica gel, petroleum ether, then petroleum ether to ethyl acetate = 100:1) afforded **4l** (33 mg, 35%): colorless liquid;

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J. Org. Chem. 2017, 82, 624−632

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.66 (d, J = 7.9 Hz, 1H), 6.53 (d, J = 1.0 Hz, 1H), 6.45 (dd, J = 1.0, 7.9 Hz, 1H), 5.88 (s, 2H), 2.52 (dd, J = 6.1, 10.1 Hz, 1H), 2.00−1.90 (m, 1H), 1.81−1.72 (m, 1H), 1.40−1.36 (m, 6H), 1.28−1.22 (m, 8H), 0.90−0.84 (m, 12H), 0.78−0.74 (m, 6H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 147.5, 143.6, 141.3, 118.8, 108.0, 106.9, 100.4, 35.1, 33.8, 29.1, 27.4, 22.9, 13.9, 13.6, 9.0; IR (film) 2956, 2926, 2870, 1485, 1243, 1181, 1042, 942, 908, 733 cm^{-1}; EI-MS (m/z, relative intensity) 468 (M^+, 9), 411 [$(M - 57)^+$, 12], 291 (62), 235 (71), 177 (78), 135 (100); HRMS (EI) calcd for $C_{23}H_{40}O_2{}^{120}Sn$ $[M]^+$ 468.2045, found 468.2065.

Tributyl(2-methyl-1-phenylpropyl)stannane ***4m***. Flash chromatography (silica gel, petroleum ether) afforded **4m** (22 mg, 26%): colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (t, J = 7.6 Hz, 2H), 7.00−6.96 (m, 3H), 2.37−2.28 (m, 1H), 2.24−2.15 (m, 1H), 1.37−1.31 (m, 6H), 1.25−1.20 (m, 6H), 1.06 (d, J = 6.4 Hz, 3H), 0.86−0.82 (m, 12H), 0.74−0.70 (m, 6H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 146.9, 128.2, 127.2, 123.4, 45.8, 31.4, 29.1, 27.5, 24.8, 23.3, 13.6, 10.0; IR (film) 2955, 2925, 2871, 1593, 1493, 1463, 908, 734 cm^{-1}; EI-MS (m/z, relative intensity) 367 [$(M - 57)^+$, 17], 291 (79), 235 (94), 179 (96), 120 (31), 91 (100); HRMS (EI) calcd for $C_{18}H_{31}{}^{120}Sn$ $[M - C_4H_9]^+$ 367.1442, found 367.1435.

Tributyl(2,3-dihydro-1H-inden-1-yl)stannane ***4n***. Flash chromatography (silica gel, petroleum ether) afforded **4n** (28 mg, 33%): colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, J = 7.3 Hz, 1H), 7.07−7.03 (m, 1H), 7.01−6.99 (m, 1H), 6.97−6.93 (m, 1H), 3.01−2.99 (m, 1H), 2.93−2.90 (m, 1H), 2.81−2.72 (m, 1H), 2.40−2.32 (m, 1H), 2.18−2.16 (m, 1H), 1.44−1.38 (m, 6H), 1.28−1.23 (m, 6H), 0.85 (t, J = 7.3 Hz, 9H), 0.82−0.78 (m, 6H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 149.7, 141.7, 126.1, 124.1, 123.6, 121.9, 33.1, 32.1, 30.4, 29.1, 27.4, 13.6, 9.3; IR (film) 2955, 2925, 2852, 1473, 1457, 1079, 908, 734 cm^{-1}; EI-MS (m/z, relative intensity) 408 (M^+, 1), 351 [$(M - 57)^+$, 5], 291 (49), 235 (70), 179 (79), 117 (100), 91 (16); HRMS (EI) calcd for $C_{17}H_{27}{}^{120}Sn$ $[M - C_4H_9]^+$ 351.1129, found 351.1151.

Typical Procedure for the Stannylation of Aldehydes or Ketones (Scheme 4). Under nitrogen atmosphere, $TsNHNH_2$ (2 mmol, 372 mg), toluene (10 mL), and benzaldehyde (2 mmol, 212 mg) were weighed in a 100 mL Schlenk tube. The solution was sealed and heated at 80 °C for 1 h. After the reaction was cooled to room temperature, NaH (1.1 equiv, 2.2 mmol, 50 wt %, 106 mg), TBAC (20% equiv, 0.4 mmol, 111 mg), and toluene (15 mL) were added. The reaction mixture was stirred at room temperature for 10 min. Then ^{n}Bu$_3$SnH (1 equiv, 2 mmol, 582 mg) was added to the reaction system through a syringe. The resulting reaction solution was stirred at 110 °C for 6 h. The reaction mixture was then filtered through silica gel with petroleum ether as eluent. The combined filtrate was then concentrated on a rotary evaporator under reduced pressure to leave a crude residue, which was purified by flash chromatography with silica gel (eluted with petroleum ether). Benzyltributylstannane **2a** was obtained as colorless liquid (556 mg, 73%).

Typical Procedure of Tandem Stannylation/Stille Cross-Coupling Reactions (Scheme 5). Under nitrogen atmosphere, *N*-tosylhydrazone **1a** (0.2 mmol, 55 mg), NaH (1.1 equiv, 0.22 mmol, 50 wt %, 11 mg), and TBAC (20% equiv, 0.04 mmol, 11 mg) were weighed in a 10 mL Schlenk tube. Toluene (3 mL) was then added in succession. The solution was stirred at room temperature for 10 min. ^{n}Bu$_3$SnH (1 equiv, 0.2 mmol, 58 mg, 54 μL) was added into this system through syringe. The resulting reaction solution was stirred at 110 °C for 6 h. The reaction mixture was filtered through silica gel to remove the insoluble precipitate. The combined filtrate was then concentrated on a rotary evaporator under reduced pressure to leave a crude product. Then under nitrogen atmosphere, $Pd(PPh_3)_4$ (5 mol %, 12 mg), methyl 4-bromobenzoate (1 equiv, 43 mg), and DMF (3 mL) were added, and the solution was stirred at 90 °C for 18 h. The solution was concentrated on a rotary evaporator under reduced pressure to leave a crude residue, which was purified by silica gel column chromatography (eluted first with petroleum ether, then with petroleum ether/EtOAc = 100:1) to afford methyl 4-benzylbenzoate **3a** as pale yellow solid (32 mg, 70%).

Methyl 4-Benzylbenzoate ***5a***.[26] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 200:1, then 100:1) afforded **5a** (32 mg, 70%): yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.3 Hz, 2H), 7.31−7.21 (m, 5H), 7.17 (d, J = 8.3 Hz, 2H), 4.02 (s, 2H), 3.88 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 167.0, 146.5, 140.1, 129.8, 128.9, 128.6, 128.1, 126.3, 51.9, 41.9.

Diphenylmethane ***5b***.[26] Flash chromatography (silica gel, petroleum ether) afforded **5b** (22 mg, 66%): colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29−7.26 (m, 4H), 7.20−7.17 (m, 6H), 3.98 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 141.1, 128.9, 128.4, 126.0, 41.9.

1-Benzyl-4-nitrobenzene ***5c***.[27] Flash chromatography (silica gel, petroleum ether to ethyl acetate = 100:1) afforded **5c** (32 mg, 75%): yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 8.5 Hz, 2H), 7.34−7.30 (m, 4H), 7.26−7.24 (m, 1H), 7.17 (d, J = 7.4 Hz, 2H), 4.07 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.8, 146.5, 139.2, 129.6, 128.9, 128.8, 126.7, 123.7, 41.7.

1-Benzyl-3-methoxybenzene ***5d***.[26] Flash chromatography (silica gel, petroleum ether, then petroleum ether:ethyl acetate = 100:1) afforded **5d** (22 mg, 56%): colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30−7.26 (m, 2H), 7.22−7.18 (m, 4H), 6.98 (d, J = 7.5 Hz, 1H), 6.75−6.73 (m, 2H), 3.95 (s, 2H), 3.77 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 159.7, 142.7, 140.9, 129.4, 128.9, 128.4, 126.1, 121.4, 114.8, 111.3, 55.1, 42.0.

3-Benzylpyridine ***5e***.[28] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 20:1, then 5:1) afforded **5e** (24 mg, 71%): yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (s, 1H), 8.45 (d, J = 4.1 Hz, 1H), 7.46 (d, J = 7.6 Hz, 1H), 7.32−7.28 (m, 2H), 7.24−7.16 (m, 4H), 3.97 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 150.1, 147.6, 139.7, 136.3, 128.8, 128.7, 126.4, 123.4, 39.0.

Methyl 4-(4-methylbenzyl)benzoate ***5f***.[29] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 200:1) afforded **5f** (37 mg, 77%); yellow liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, J = 8.5 Hz, 2H), 7.24 (d, J = 8.5 Hz, 2H), 7.10 (d, J = 8.0 Hz, 2H), 7.05 (d, J = 8.0 Hz, 2H), 3.98 (s, 2H), 3.88 (s, 3H), 2.31 (s, 3H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 167.0, 146.8, 137.0, 135.8, 129.8, 129.2, 128.8, 128.8, 128.0, 51.9, 41.5, 21.0.

Methyl 4-(3,4-Dichlorobenzyl)benzoate ***5g***. Flash chromatography (silica gel, petroleum ether/ethyl acetate = 200:1) afforded **5g** (31 mg, 53%): yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, J = 8.1 Hz, 2H), 7.35 (d, J = 8.2 Hz, 1H), 7.26−7.22 (m, 3H), 7.00 (dd, J = 1.2, 8.0 Hz, 1H), 3.97 (s, 2H), 3.91 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 166.8, 145.0, 140.3, 132.5, 130.8, 130.5, 130.0, 128.9, 128.6, 128.3, 52.1, 40.9; IR (film) 1721, 1611, 1471, 1435, 1281, 1071, 1032, 907 cm^{-1}; EI-MS (m/z, relative intensity) 296 (55), 294 (M^+,77), 263 (63), 235 (63), 207 (46), 165 (100); HRMS (ESI) calcd for $C_{15}H_{13}{}^{35}Cl_2O_2$ $[M + H]^+$ 295.02871, found 295.02894.

Methyl 4-(4-Methoxybenzyl)benzoate ***5h***.[30] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 200:1, then 100:1) afforded **5h** (30 mg, 59%): yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.2 Hz, 2H), 7.08 (d, J = 8.6 Hz, 2H), 6.83 (d, J = 8.6 Hz, 2H), 3.96 (s, 2H), 3.89 (s, 3H), 3.78 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 167.0, 158.1, 147.0, 132.2, 129.9, 129.8, 128.8, 128.0, 114.0, 55.2, 52.0, 41.0.

Methyl 4-(Biphenyl-4-ylmethyl)benzoate ***5i***. Flash chromatography (silica gel, petroleum ether/ethyl acetate = 300:1, then 200:1) afforded **5i** (38 mg, 63%): yellow solid; mp = 96−97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.2 Hz, 2H), 7.56 (d, J = 7.4 Hz, 2H), 7.52 (d, J = 8.1 Hz, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.34−7.32 (m, 1H), 7.28 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 4.06 (s, 2H), 3.89 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 167.0, 146.4, 140.8, 139.3, 139.2, 129.8, 129.3, 128.9, 128.7, 128.1, 127.3, 127.1, 127.0, 52.0, 41.5; IR (film) 1721, 1609, 1487, 1435, 1280, 1110, 909, 732 cm^{-1}; EI-MS (m/z, relative intensity) 302 (M^+, 100), 271 (16), 243 (51), 228 (10), 207 (10), 165 (46); HRMS (ESI) calcd for $C_{21}H_{19}O_2$ $[M + H]^+$ 303.1380, found 303.1381.

Methyl 4-(Benzo[d][1,3]dioxol-5-ylmethyl)benzoate ***5j***. Flash chromatography (silica gel, petroleum ether:ethyl acetate = 200:1) afforded **5j** (30 mg, 56%): white solid; mp = 70−71 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.1 Hz, 2H), 7.23 (d, J = 8.1 Hz, 2H), 6.74 (d, J = 7.6 Hz, 1H), 6.65−6.63 (m, 2H), 5.91 (s, 2H), 3.93

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J. Org. Chem. 2017, 82, 624−632

(s, 2H), 3.89 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 167.0, 147.8, 146.6, 146.1, 133.9, 129.8, 128.8, 128.1, 121.8, 109.3, 108.2, 100.9, 52.0, 41.5; IR (film) 2922, 1719, 1489, 1442, 1280, 1245, 1111, 1040, 910, 732 cm^{-1}; EI-MS (*m/z*, relative intensity) 270 (M^+, 100), 239 (18), 211 (33), 181 (32), 152 (31), 135 (21); HRMS (ESI) calcd for $C_{16}H_{15}O_4[M + H]^+$ 271.0965, found 271.0965.

Methyl 4-(Naphthalen-2-ylmethyl)benzoate **5k**.[31] Flash chromatography (silica gel, petroleum ether/ethyl acetate = 200:1, then 50:1) afforded **5k** (37 mg, 67%): yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.80−7.75 (m, 3H), 7.61 (s, 1H), 7.47−7.41 (m, 2H), 7.29−7.26 (m, 3H), 4.17 (s, 2H), 3.88 (s, 3H); $^{13}C\{^1H\}$ NMR(100 MHz, $CDCl_3$) δ 167.0, 146.3, 137.6, 133.6, 132.1, 129.8, 129.0, 128.2, 128.2, 127.6, 127.5, 127.4, 127.2, 126.1, 125.5, 52.0, 42.0.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.6b02639.

^1H and ^{13}C spectra for all products (PDF)

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Notes

The authors declare no competing financial interest.

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J. Org. Chem. 2017, 82, 624−632