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Disilylation of N-(2-Halophenyl)-2-phenylacrylamides with Hexamethyldisilane via Trapping the Spirocyclic Palladacycles

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ABSTRACT

The palladium-catalyzed disilylation of the spirocyclic palladacycles with hexamethylaisilane has been realized. The key spirocyclic palladacycles are generated from N-(2-haloaryl)-2-arylacrylamide *via* intramolecular Heck reaction and followed remote C-H activation. A range of 3-((trimethylsilyl)methyl)-3-(2-(trimethylsilyl)phenyl)indolin-2-ones are obtained in good to excellent yields from readily available starting material under mild conditions.

Organosilicon compounds are a very important class of compounds due to their application in the synthetic chemistry,[1] medicinal chemistry[2] and material science.[3] As a result, substantial efforts have been directed toward the development of efficient method for the synthesis of silicon-containing compounds. The conventional methods for the construction of C-Si bonds focus on straightforward silylation of either organomagnesium or organolithium reagents with chlorosilanes[4]. However, these methods suffer from poor tolerance of functional groups and harsh reaction conditions. Recently, significant progress for the synthesis of organosilanes has been made in transition metal-catalyzed silylation reactions of hexamethyldisilane.[5-7] So far, two main types of silylation reactions have been developed by using hexamethyldisilane as silyl reagent, including C-X silylation *via* classic cross-coupling reaction[6] (Scheme 1, eq 1), and C-H silylation *via* oxidative coupling reaction (Scheme 1, eq 2).[7] Mechanistically, these coupling reactions are initiated by the formation of C-palladium intermediate, then trapped by hexamethyldisilane, and finally afford trimethylsilyl-containing compounds *via* reductive elimination of C-Si bond. Importantly, the trimethylsilyl can conveniently convert into other functional groups (such as halogen, hydroxyl, amino)[8]. Therefore, development of new method for introducing the trimethylsilyl in organic molecular is very significant.

Palladacycle, as an important intermediate, has been extensively studied and found wide applications in organic synthesis.[9,10] With two C-Pd bonds, palladacycle is conveniently used to construct a variety of cyclic compounds.[9] However, ring-opening difunctionalization products are quite rare.[10] Therefore, utilizing the two C-Pd bonds of palladacycle for the synthesis of difunctionalization products via ring-opening coupling reaction is a big challenge. Recently, Wang reported that the palladium carbine species (containing carbon-palladium double bond) could be inserted by disilane to access germinal disilanes.[11] In this reaction, two C-Si bonds were formed, and two silyls were introduced. Based on our previous studies on palladacycle[12] and silicon-containing compounds,[13] we envision that the palladacycles could be trapped by hexamethyldisilane for the synthesis of disilylation products (Scheme 1, eq 3). (the similar work was reported online by Zhang[14] when we prepared to submit our manuscript.)

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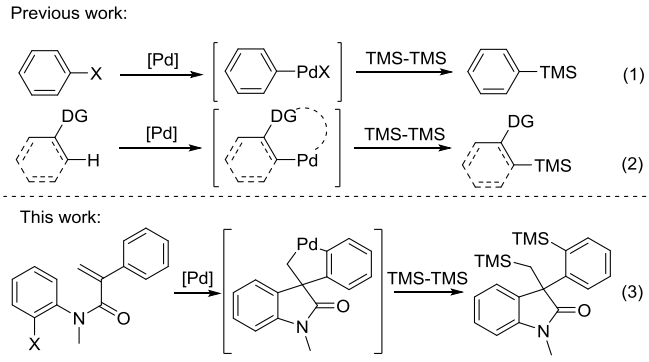
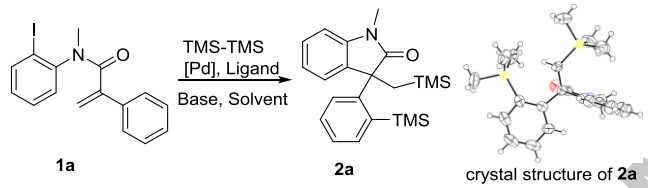


Scheme 1. Methods for Synthesis of Trimethylsilyl Compounds

The initial investigation focused on the reaction of hexamethyldisilane with N-(2-iodophenyl)-N-methyl-2-phenylacrylamide **1a** for exploring the reaction conditions

(Table 1). Fortunately, the disilylation product **2a** was obtained in 70% yield by employing $Pd(OAc)_2$ as catalyst, $P(^tBu)_3$ as ligand and Cs_2CO_3 as base in CH_3CN at 90 °C under nitrogen atmosphere, and the structure of **2a** was proved by a single-crystal X-ray diffraction (CCDC: 1822233). Encouraged by this result, the palladium catalysts were tested firstly, and the results indicated that the catalyst of $Pd(OAc)_2$ performed the best catalytic efficiency. P-ligand free condition and the other P-ligands including PPh_3, $P(o\text{-tol})_3$, X-Phos, and S-Phos were examined, and PPh_3 was found to give the best result. Subsequently, a variety of bases such as KF, K_3PO_4, K_2CO_3 and Na_2CO_3 were investigated. The results shown that the base K_3PO_4 was the best for this disilylation of C,C-palladacyclices. To enhance the reaction yield, several solvents such as THF, Toluene, DMAc and Dioxane were screened, but the yield of the disilylation product all gave rise in lower yields than CH_3CN. Finally, the temperature screening indicated that 90 °C was the most suitable temperature for this protocol. Therefore, the optimized reaction conditions were as follows: **1a** (0.2 mmol), hexamethyldisilane (1.6 mmol), K_3PO_4 (1.0 mmol), $Pd(OAc)_2$ (10 mol%), PPh_3 (20 mol%) in CH_3CN (1 mL), at 90 °C.

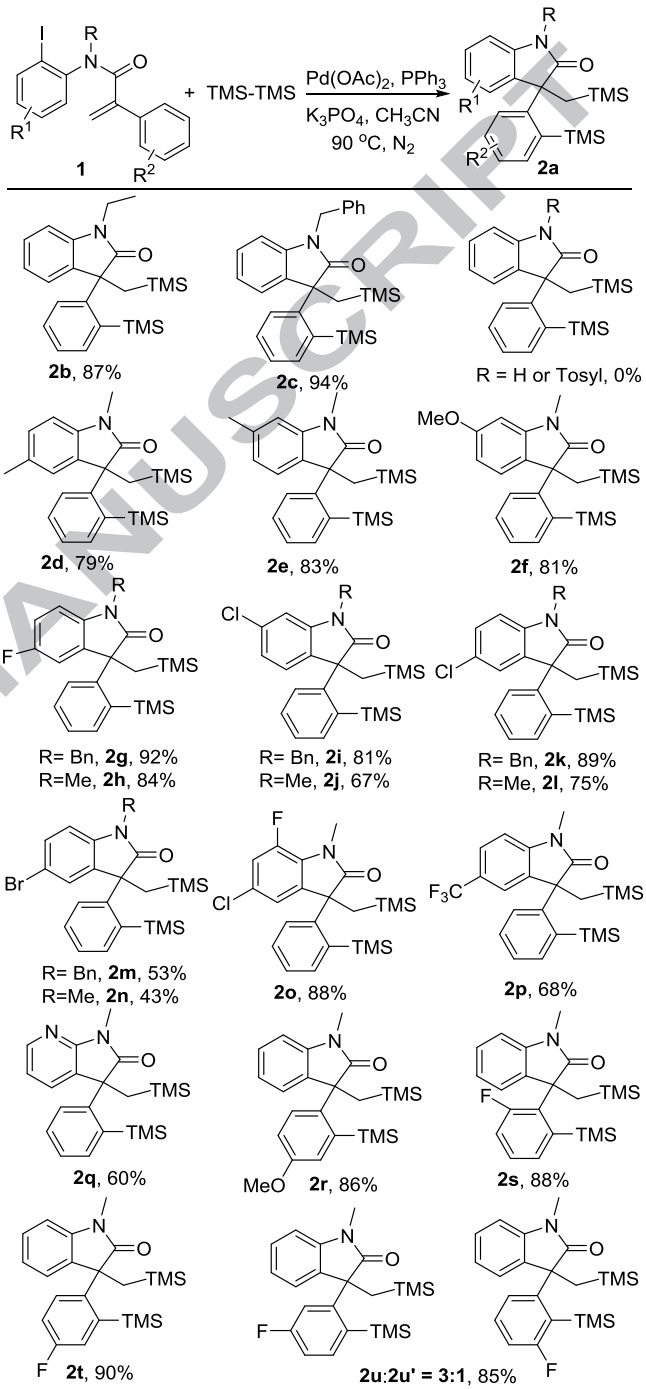
Table 1. Optimization of Reaction Conditions[a]



entry	catalyst	ligand	base	solvent	yield
1	$Pd(OAc)_2$	$P(^tBu)_3$	Cs_2CO_3	CH_3CN	70%
2	$Pd(dba)_2$	$P(^tBu)_3$	Cs_2CO_3	CH_3CN	69%
3	$Pd_2(dba)_3$	$P(^tBu)_3$	Cs_2CO_3	CH_3CN	68%
4	$[Pd(\pi\text{-ally})Cl]_2$	$P(^tBu)_3$	Cs_2CO_3	CH_3CN	65%
5	$Pd(PPh_3)_4$	$P(^tBu)_3$	Cs_2CO_3	CH_3CN	69%
6	$Pd(OAc)_2$	-	Cs_2CO_3	CH_3CN	59%
7	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	CH_3CN	72%
8	$Pd(OAc)_2$	X-Phos	Cs_2CO_3	CH_3CN	68%
9	$Pd(OAc)_2$	$P(o\text{-tol})_3$	Cs_2CO_3	CH_3CN	68%
10	$Pd(OAc)_2$	S-Phos	Cs_2CO_3	CH_3CN	69%
11	$Pd(OAc)_2$	PPh_3	KF	CH_3CN	trace
12	$Pd(OAc)_2$	PPh_3	K_3PO_4	CH_3CN	78%
13	$Pd(OAc)_2$	PPh_3	K_2CO_3	CH_3CN	71%
14	$Pd(OAc)_2$	PPh_3	Na_2CO_3	CH_3CN	11%
15	$Pd(OAc)_2$	PPh_3	K_3PO_4	THF	54%
16	$Pd(OAc)_2$	PPh_3	K_3PO_4	Toluene	trace
17	$Pd(OAc)_2$	PPh_3	K_3PO_4	DMAc	61%
18	$Pd(OAc)_2$	PPh_3	K_3PO_4	Dioxane	56%
19[b]	$Pd(OAc)_2$	PPh_3	K_3PO_4	CH_3CN	68%
20[c]	$Pd(OAc)_2$	PPh_3	K_3PO_4	CH_3CN	67%

[a] Reaction conditions: **1a** (0.2 mmol), Hexamethyldisilane (8 equiv), Palladium catalyst (10 mol%), Ligand (20 mol%), Base (5 equiv), Solvent (1 mL), 90 °C, 12 h. Yields of isolated are given.
[b] 110 °C.
[c] 70 °C.

Table 2. Disilylation of N-(2-Iodophenyl)-2-phenylylacrylamides[a,b]



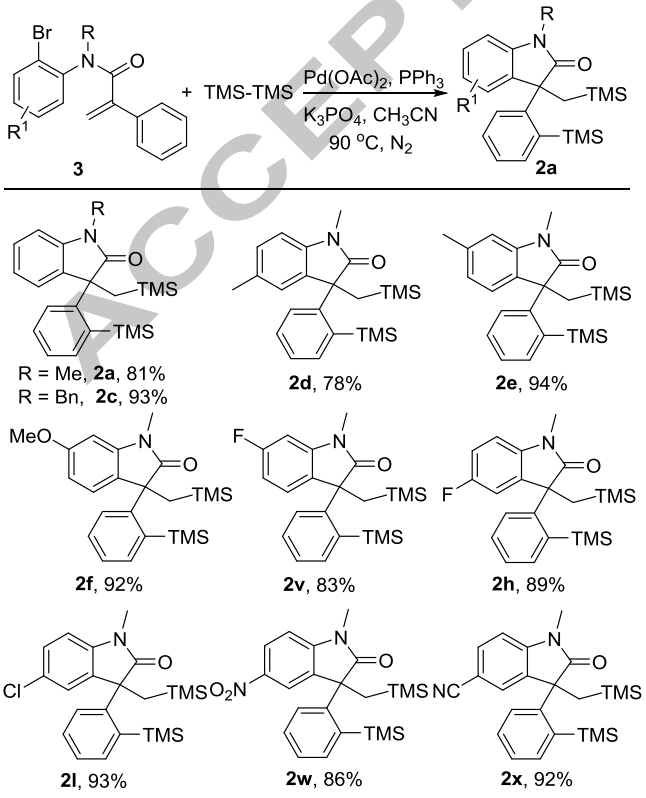
[a] Reaction conditions: **1a** (0.2 mmol), Hexamethyldisilane (8 equiv), $Pd(OAc)_2$ (10 mol%), PPh_3 (20 mol%), K_3PO_4 (5 equiv), CH_3CN (1 mL), 90 °C, 12 h.
[b] Isolated yields.

With the optimized conditions in hand, we turned our attention to survey the substrate scope of disilylation by using a variety of acrylamides and hexamethyldisilane, and the results were summarized in Table 2. First, phenylacrylamides with mono-substitution and di-substitution on the nitrogen atom were investigated. Satisfactorily, electron-donating substituents such as ethyl (**1b**) and benzyl (**1c**) substrates reacted well, and delivered

the target disilylation products in 87% and 94% yield respectively. However, the N-free and N-tosyl substituted substrates were unfavourable to this disilylation. Subsequently, our study focused on screening the effect of substitutents on the 2-iodophenyl of acrylamides. It was observed that the substrates containing electron-rich groups such as methyl and methoxyl could smoothly transform into the correspond products in high yields. The substrates containing electron-poor groups such as trifluoromethyl, fluoro, chloro, and bromo groups survived under the standard conditions, and the corresponding products were formed in yields ranging from 43% to 93%. Specifically, the benzyl protecting group on the nitrogen moiety improved the overall yield. Interesting, heterocyclic N-(3-iodopyridin-2-yl)-N-methyl-2-phenylacrylamide gave a disilylation product **2q** in 60% yield. Furthermore, the compatibility of functional groups on the phenyl linked to the double bonds was checked. The substrates containing the electron-donating group such as methoxyl and the electron-withdrawing group such as fluoro (including *orthor-*, *para-* and *meta*-fluoro groups) were well-tolerated, and the corresponding products were given in perfect yields. However, two regioisomers were generated from substrate **1u** in a ratio of 3 : 1. Notably, the electronic effect and steric effect did not affect the yields obviously.

In order to widen the scope of substrates, aryl bromides were screened. As shown in Table 3, the reaction results indicated the aryl bromides were also reactive enough to undergo the disilylation reaction. Under the standard reaction conditions, the N-methyl and N-benzyl substituted N-(2-iodophenyl)-2-phenylylacrylamides gave the corresponding products in 81% and 93% yields respectively. To our delight, the substrates bearing the electron-donating groups such as methyl and methoxyl or the electron-withdrawing groups such as fluorine, chlorine, nitryl and cyano all could tolerate, and the desired disilylation products were obtained in good to excellent yields.

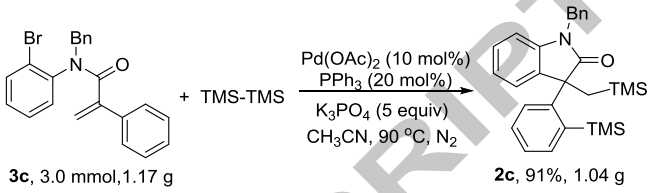
Table 3. Disilylation of N-(2-Bromophenyl)-2-phenylylacrylamide [a,b]



[a] Reaction conditions: **1a** (0.2 mmol), Hexmethyldisilane (8 equiv), $Pd(OAc)_2$ (10 mol%), PPh_3 (20 mol%), K_3PO_4 (5 equiv), CH_3CN (1 mL), 90 °C, 12 h.

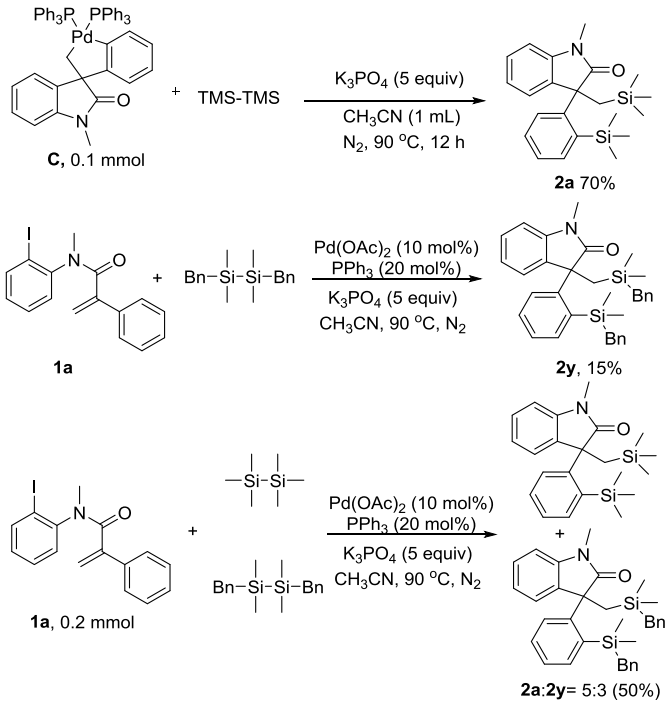
[b] Isolated yields.

It is notable that the reaction can also be performed on a gram scale, with no impact on the yield. As shown in Scheme 2, treatment of 1.17 g (3.0 mmol) of **3c** with hexmethyldisilane (8 equiv), afforded the product **2c** in 91% yield (1.04 g).

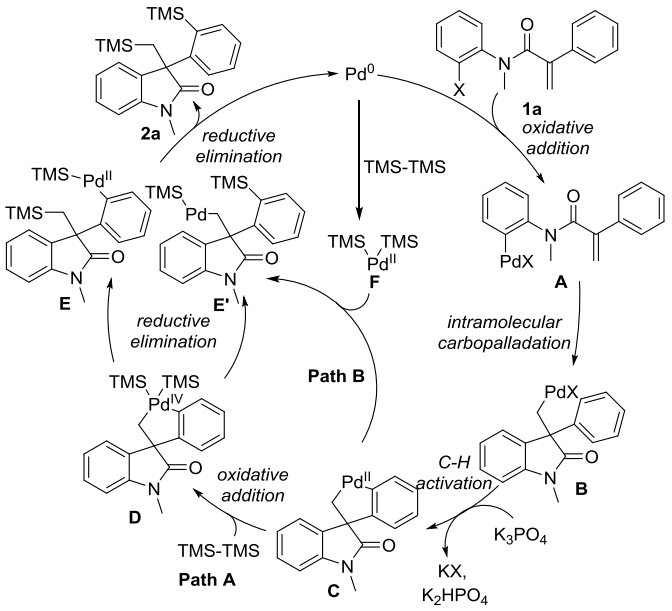


Scheme 2. Gram-Scale Double Silylation of **3c**

To further gain insight into the mechanism, the palladacycle **C** was prepared following the literature procedure.[15] It is noted that **C** was disilylated to give **2a** in 70% yield. The experimental result indicate that the disilylation reaction should proceed through palladacycle intermediates. Furthermore, a crossover experiment was carried out (Scheme 3). First, 15% of disilylation product **2y** was afforded when hexamethyldisilane was instead of 1,2-dibenzyl-tetramethyldisilane under standard conditions. Then, the reaction of **1a** with the mixture of hexamethyldisilane and 1,2-dibenzyl-tetramethyldisilane was performed, and only the corresponding products **2a** and **2y** were obtained in 50% yield (**2a**:**2y** = 5:3; determined by ^{1}H NMR). This result indicated that the disilylation of the palladacycle underwent a coordinate process.



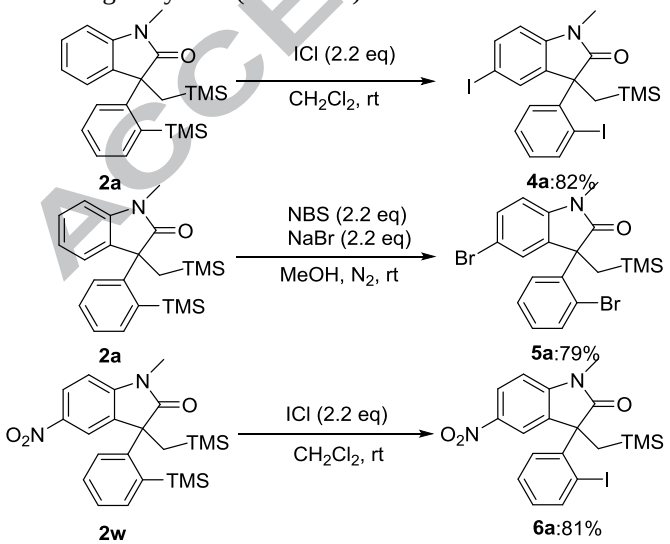
Scheme 3. Control Experiments



Scheme 4. Possible Mechanism for the Synthesis of **2a**

Based on the present experimental results, as well as previous reported mechanisms,[5-14] the hypothetical catalytic cycle is shown in Scheme 4. The reaction starts with oxidative addition of Pd(0) to C-X bond, followed by a 5-exo trig carbopalladation to form alkylpalladium(II) intermediate **B**. Then a C-H activation step would give rise to the five-membered palladacycle **C**. In general, two reaction pathways [A: Pd(IV) versus B: Pd(II)-Pd(II)] for the disilylation of hexamethyldisilane are considerable. Path A: intermediate **C** occurs an oxidative addition to generate the Pd(IV) intermediate **D**. Finally, the intermediate **D** undergoes double reductive elimination and affords the product **2a**. Path B: the reaction starts with oxidative addition of Si-Si bond to Pd(0) , followed by transmetalation-type exchange, and finally reductive elimination of C-Si bond to afford the product **2a**.

The strategic utility of the trimethylsilyl was demonstrated through selective iodination and bromination,[8a-8c] and the corresponding products were afforded in good yields (Scheme 5).



Scheme 5. Synthetic Utility of the Silylation Products

In conclusion, we have developed the palladium-catalyzed disilylation of the spirocyclic palladacycles with hexamethylsilane. Furthermore, the crossover experiments have proved that the disilylation reaction underwent a cooperative process. Successive studies on the detailed mechanism and applications of the new method are underway in our laboratory.

Acknowledgments

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Supplmentary Material

Supplementary data (experimental procedures and characterization data for all new compounds and copies of NMR spectra) associated with this article can be found, in the online version.

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Highlights:

1). Double silylation of spirocyclic palladacycles with hexamethylsilane
2). Initiated by intramolecular Heck reaction and followed remote C-H activation
3). Obtained disilylation compounds of indolinone in good to excellent yields

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Disilylation of N-(2-Halopenyl)-2-penylacrylamides with Hexamethyldisilane *via* Trapping the Spirocyclic Palladacycles

Genhua Xiao, Liang Chen, Guobo Deng, Jianbing, Liu* Yun Liang*

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