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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 hexamethylsilane 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.

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Organosilicon compounds are a very important class of compounds due to their application in the synthetic chemistry,¹ medicinal chemistry² 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 reaction⁶ (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.

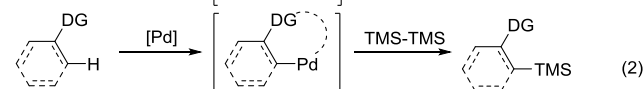
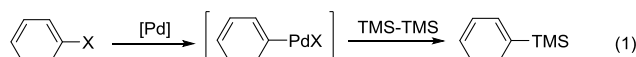
Palladacycle, as an important intermediate, has been extensively studied and found wide applications in organic synthesis.^{9,10} With two C-Pd bonds, palladacycle is conveniently used to construct a variety of cyclic compounds.⁹ However, ring-opening difunctionalization products are quite rare.¹⁰ Therefore, utilizing the two C-Pd bonds of palladacycle for the synthesis of difunctionalization products via ring-opening coupling

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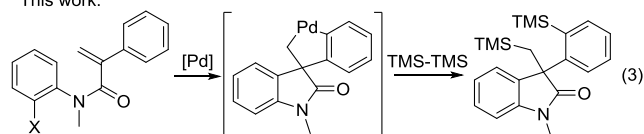
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reaction is a big challenge. Recently, Wang reported that the palladium carbene species (containing carbon-palladium double bond) could be inserted by disilane to access germinal disilanes.¹¹ In this reaction, two C-Si bonds were formed, and two silyls were introduced. Based on our previous studies on palladacycle¹² 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 Zhang¹⁴ when we prepared to submit our manuscript.)

Previous work:



This work:



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

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